STUDY OF TRANSPORTATION OF GTL PRODUCTS FROM ALASKAN NORTH SLOPE (ANS) TO MARKETS

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DISCLAIMER

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ABSTRACT

The Alaskan North Slope is one of the largest hydrocarbon reserves in the United States where Gas-to-Liquids (GTL) technology can be successfully implemented. The proven and recoverable reserves of conventional natural gas in the developed and undeveloped fields in the Alaskan North Slope (ANS) are estimated to be 38 trillion standard cubic feet (TCF) and estimates of additional undiscovered gas reserves in the Arctic field range from 64 TCF to 142 TCF. Transportation of the natural gas from the remote ANS is the key issue in effective utilization of this valuable and abundant resource. The throughput of oil through the Trans Alaska Pipeline System (TAPS) has been on decline and is expected to continue to decline in future. It is projected that by the year 2015, ANS crude oil production will decline to such a level that there will be a critical need for pumping additional liquid from GTL process to provide an adequate volume for economic operation of TAPS. The pumping of GTL products through TAPS will significantly increase its economic life. Transporting GTL products from the North Slope of Alaska down to the Marine terminal at Valdez is no doubt the greatest challenge facing the Gas to Liquids options of utilizing the abundant natural gas resource of the North Slope.

The primary purpose of this study was to evaluate and assess the economic feasibility of transporting GTL products through the Trans Alaska Pipeline System (TAPS). Material testing program for GTL and GTL/Crude oil blends was designed and implemented for measurement of physical properties of GTL products. The measurement and evaluation of the properties of these materials were necessary so as to access the feasibility of transporting such materials through TAPS under cold arctic conditions. Results of the tests indicated a trend of increasing yield strength with increasing wax content. GTL samples exhibited high gel strengths at temperatures as high as 20°F, which makes it difficult for cold restart following winter shutdowns.

Simplified analytical models were developed to study the flow of GTL and GTL/crude oil blends through TAPS in both commingled and batch flow models. The economics of GTL transportations by either commingled or batching mode were evaluated. The choice of mode of transportation of GTL products through TAPS would depend on the expected purity of the product and a trade-off between loss in product value due to contamination and cost of keeping the product pure at the discharge terminal.

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STUDY OF TRANSPORTATION OF GTL PRODUCTS FROM ALASKAN NORTH SLOPE (ANS) TO MARKETS

EXECUTIVE SUMMARY

The Alaskan North Slope is one of the largest hydrocarbon reserves in the United States where Gas-to-Liquids (GTL) technology can be successfully implemented. Gas-to-liquids (GTL) conversion technology, where natural gas is chemically converted to transportable liquid products, is an emerging technology that is expected to reach commercialization within the next decade. The proven and recoverable reserves of conventional natural gas in the developed and undeveloped fields in the Alaskan North Slope (ANS) are estimated to be 38 trillion standard cubic feet (TCF) and estimates of additional undiscovered gas reserves in the Arctic field range from 64 TCF to 142 TCF. Currently, only a small portion of the produced natural gas of the North Slope of Alaska is used in the oil-field operation, such as gas lift and power generation, and in local sales. The unused portion is injected back into the reservoir for pressure maintenance and oil production. It is expected that as crude oil production on the North Slope continues to decline, approximately 26 TCF of ANS natural gas will become available for gas sales, transportation and/ or conversion to GTL products. This equates to over 4 billion barrels of oil equivalent.

Transportation of the natural gas from the remote ANS is the key issue in effective utilization of this valuable and abundant resource. The throughput of oil through the Trans Alaska Pipeline System (TAPS) has been on decline and is expected to continue to decline in future. Currently, 4 of the 12 pump stations have been shut down due to decline in the TAPS throughput. It is projected that by the year 2015, ANS crude oil production will decline to such a level (200,000 to 400,000 bbl/day) that there will be a critical need for pumping additional liquid from GTL process to provide an adequate volume for economic operation of TAPS. The pumping of GTL products through TAPS will significantly increase its economic life. Transporting Gas to Liquids products from the North Slope of Alaska down to the Marine terminal at Valdez is no doubt the greatest challenge facing the Gas to Liquids options of utilizing the abundant natural gas resource of the North Slope.

The primary purpose of this study was to evaluate and assess the economic feasibility of different products through the Trans Alaska Pipeline System (TAPS). Material testing program for GTL and GTL/Crude oil blends was designed following discussions with John Hackworth (UAF consultant on GTL studies) and Alyeska Pipeline Service Company.

The measurement and evaluation of the properties of these materials were necessary so as to access the feasibility of transporting such materials through TAPS under cold arctic conditions. Crude oil samples were supplied by Alyeska Pipeline Service Company and two GTL samples designated as GTL1, which is a solid wax sample from LaPorte, and Fischer-Tropsch (FT) diesel or light hydrocarbon GTL (designated as GTL2) were

supplied by USDOE for this study. The 20% cut of the wax distillate from GTL1 was mixed with samples of GTL2 in different proportions, which were then blended with crude oil samples in three different blend ratios. The density, viscosity and gel strength of these samples were measured. Results of the tests indicate:

- Trend of increasing yield strength with increasing wax content.
- High gel strength of GTL samples at very low temperatures as low as 20°F, which makes it difficult for cold restart following winter shutdowns.

Simplified analytical models were developed to study the flow of GTL and GTL/crude oil blends through TAPS in both commingled and batch flow models. Commingled flow involves the blending of the GTL product and the crude oil to form a commingled homogenous liquid mixture. Batch flow involves pumping alternate slugs of GTL products and crude oil. It can be achieved by three different techniques, namely:

- Uncontrolled or traditional batching of products, termed batch mode A
- Controlled batching using physical barrier such as pigs and spacers, termed batch mode B

Controlled batching using modern batching technique, which entails pumping alternate slugs of GTL and crude oil while fluid movement is monitored by interface detection devices to minimize the loss of product value. The pressure gradients and related hydraulic flow parameters for each transportation mode were determined and compared.

The economics of GTL transportations by either commingled or batching mode were evaluated. The choice of mode of transportation of GTL products through TAPS would depend on the expected purity of the product and a trade-off between loss in product value due to contamination and cost of keeping the product pure at the discharge terminal. Tables 1 and 2 show the basic economic assumptions and the parameters used for rate of return analysis for the different transportation modes

TABLE I   Economic Assumptions

Conversion @ 60% efficiency	9.67 MScf / bbl
Plant Uptime Efficiency	95%
Project Life	20 years
Plant Capacity	100 MBPD
Taxes:	
State Income	9.4%
Federal CIT	35.0%
Property Tax	2%
Depreciation	Modified Accelerated Capital Recovery Scheme

TABLE II   Model Parameters for ROR

Cost Estimates
- Plant Cost ranging from $ 20,000/BPD to $ 35,000
- Gas cost based on net back of 20%
- Annual Operating and Maintenance cost of 5.6% of Plant Cost
- Transportation and storage estimated with Tariff estimates. Capital investments are amortized over the project life and worked out per barrel of product.

Revenue Estimates
ROR calculation based on $21.00 per barrel crude price.
GTL products given a premium of 1.4 times Spot Oil price
Batch Transportation efficiency of 95%

SENSITIVITY ANALYSIS

Key Parameters in the rate of return analysis were modified to identify those with the greatest influence on the results. The parameters include:
- Capital Expenditure was varied between $20,000 per daily barrel and $35,000 per daily barrel to accommodate speculated range of plant costs and possible North Slope scale up factor.
- The crude oil price was varied between $21.00 per barrel and $35.00 per barrel
- For the batching operation, installing new storage and relief tanks at the terminal and pump stations respectively versus refurbishing some old tanks to accommodate production of storage.

CONCLUSION

The modern batching operation consistently gave the highest return in investment and it is recommended for transportation of the Gas-to-Liquid products from the North Slope of Alaska to Valdez. The major concern with batching is the length of mixing zone or interface and the purity of GTL products as they arrive the marine terminal in Valdez. Since experience shows that the length of this interface is independent on volume pumped, it becomes an optimization issue to find the optimum holding capacity on the North Slope that can give the minimum number of batches at any given production period. The optimum fluid velocity in pipeline should be determined with reasonable accuracy based on the density and viscosity difference of the two products to be transported to ensure minimum interface.

RECOMMENDATIONS

This study indicates that even the light GTL (LaPorte type) or FT diesel can pose problems for transportation through TAPS from the cold restart point of view. Rigorous studies are needed to identify the upper limit on the quantity and nature of the paraffins in GTL that can be accepted for transportation through TAPS.

Although modern batching technique appears to be the transportation mode of choice at this time, batching GTL products through the same pipeline that carries crude oil is likely to create significant problems of GTL product contamination. Wax, sulfur, asphaltene and other assorted solid deposits on the inside walls of the pipeline can potentially re-dissolve in the slug of pure GTL. Since GTL is a clean, zero sulfur fuel, this type of contamination could defeat the very purpose of gas to liquid conversion. Further studies are necessary to investigate the effect of GTL contamination from the pipe-wall residue.

After studying the operational issues, it will be necessary to re-visit the economics of GTL transportation. For example, the economics of batching mode could potentially include an additional cost of purifying contaminated GTL products. The blending mode, on the other hand, may make it feasible to have a cheap GTL plant producing low grade GTL, thus reducing capital expenditure.

CHAPTER 1

INTRODUCTION

Gas-to-liquids (GTL) conversion technology, where natural gas is chemically converted to transportable hydrocarbon liquid products, is an emerging technology that is expected to reach commercialization within the next decade. One of the first areas in the United States to exploit this technology will be the Alaskan North Slope (ANS). The proven and recoverable reserves of conventional natural gas in the developed and undeveloped fields in the Alaskan North Slope (ANS) are estimated to be 38 trillion standard cubic feet (TCF). In addition to the known reserves, estimates of undiscovered gas reserves in the Arctic fields range from 64 TCF upwards to 142 TCF. Currently, only a small portion of the produced natural gas on the North Slope of Alaska is used in the oil-field operation, such as gas lift and power generation, and in local sales. The unused portion is injected back into the reservoir for pressure maintenance and oil production. It is expected that as crude oil production on the North Slope continues to decline, approximately 26 TCF of ANS natural gas will become available for gas sales, transportation and/or conversion to GTL products.

Several options exist for ANS gas utilization/transportation. The two most promising options are: (i) transportation of the gas via a new gas pipeline, called as the Trans-Alaska-Gas-System (TAGS) followed by Liquefaction to LNG and then to Pacific-Rim markets via LNG tankers, and (ii)conversion of the gas to GTL products followed by transportation via the existing Trans-Alaska-Pipeline-System (TAPS). In a recent study by Robertson et al. (1996), it was concluded that the TAGS/LNG and the GTL options appear economically promising and warrant consideration in the decision-making process. Moreover, the future market for ANS-LNG is less certain than a potential market for GTL products. The throughput of oil through the TAPS has been on decline and is expected to continue to decline in the future. Currently, 4 of the 12 pump stations have been shut down due to decline in TAPS throughput. It is projected that by the year 2015, ANS crude oil production will decline to such a level (200,000 to 400,000 bbl/day) that there will be critical need for pumping additional liquid from GTL process to provide an adequate volume for economic operation of the TAPS. The pumping of GTL products through TAPS will significantly increase the economic life of TAPS. Some of the reasons for considering transporting GTL products through TAPS are: 1) monetize ANS gas resources; 2) use existing oil pipeline and other transportation infrastructure for GTL transport; 3) declining ANS oil production; 4) declining ANS oil production increases the cost of oil transportation through TAPS; 5) GTL products are refined products and will receive premium compared to crude oil; 6) GTL transport will increase pipeline throughput.

This project addresses the study of GTL product transportation through the existing Trans Alaska Pipeline System (TAPS). Alyeska Pipeline Service Company (APSC), the TAPS operator, will work closely with UAF in all aspects of this project. Such a study is necessary for successful future commercialization of GTL technology in Alaska. The technical and economic factors, and transportation issues that affect the feasibility of moving GTL products through the TAPS are identified. Various commercial and pilot GTL conversion technology are reviewed and compared. A program for testing GTL materials and GTL/crude oil blends is designed and implemented. Hydraulic models, including thermodynamic considerations, are

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developed to study flow behavior of GTL products through TAPS. Finally, a preliminary economic analysis of GTL transportation through TAPS is performed.

1.1 PROJECT OBJECTIVES

The objective of this project is to evaluate the transportation of Alaskan North Slope (ANS) Gas-To-Liquids (GTL) products through the existing Trans Alaska Pipeline System (TAPS). The main purpose of this proposed project is to evaluate technical and economic feasibility of TAPS as the transportation method for the movement of GTL products. This will require an in-depth understanding of the GTL product characteristics and the relative behavior (i.e. fluid properties, mixing phenomena, and downstream separation characteristics) of mixtures or blends of the GTL products with the North Slope crude oil.

The main objectives of this project are as follows:

- To identify various transportation issues related to GTL products transport through TAPS and gather relevant information and data.
- To identify types of GTL material that could be moved through TAPS either as individual batches (slugs) or commingled with crude oil and to design a program for testing properties of these materials.
- To implement the material testing program for various GTL and GTL/crude oil blends.
- To determine the flow behavior of GTL and GTL/crude oil blends through TAPS for various GTL transportation modes.
- To provide an overall technical and economic assessment of various options of GTL transportation through TAPS.

1.2 PROJECT TASKS

The tasks envisioned in the original proposal are described below.

Task 1 – Information Required for the National Environmental Policy Act (NEPA)
The Department of Energy (DOE) shall prepare the appropriate level of NEPA documentation for the project. The University of Alaska Fairbanks (UAF) shall provide all the necessary information for the completion of this documentation.

Task 2 – Determination of TAPS Transportation Issues
The objective of this task is to identify various transportation issues related to transport of GTL products through TAPS and to gather relevant information and data. To accomplish this goal, meetings will be arranged at the beginning of this project with Alyeska Pipeline Service Company (APSC), which operates TAPS, in order to explore the issues of possible movement of GTL material through TAPS. The issues to be discussed with the TAPS operator will include (but not limited to) the following:

1. What would be the volume availability in future years for the possible movement of GTL materials through TAPS?

2. What are the potential ways of moving GTL product through TAPS? Would GTL product have to be commingled with crude oil or would it be possible to batch GTL product through the line?
3. Determine the temperature and flow conditions in TAPS and any physical property requirements for liquids moved through the system. Gather information on the range of crude oils that have been moved through TAPS and if any problems have been encountered in the past.
4. If GTL batch movement appears to be a practical possibility, gather preliminary information on issues for movements: length of time of batch cycles, degree of mixing at interfaces, other operational issues.
5. Discuss any other flow issues with the TAPS operator such as line heating capabilities, corrosion issues, pumping, cost, etc.

Task 3 – GTL TAPS Movement Options and GTL Material Testing Program Design
The objective of this task is to identify types of GTL material that could be moved through TAPS either as individual batches or commingled with crude oil and to design a program for testing properties of these materials. The primary focus will be on a range of products from Fischer-Tropsch (FT) type processes. The range will include lighter (higher gasoline yield) to heavier GTL products, with and without upgrading (such as hydrocracking to deal with conversion of heavier wax components), oxygenated liquids such as alcohols or ether mixtures produced by catalytic reforming processes. A program for testing of the properties of materials before pipeline movement and after movement will be designed. In case of commingled material, the test program will analyze what the quality attributes of the individual boiling range fractions would be when the material is distilled at a refinery after it has been moved through the pipeline and delivered to a refinery at a distant market location. Designing a GTL material testing plan will entail the following.

- Gather product quality data for GTL products for various types of FT process schemes. These will include raw products with different possible carbon number ranges and upgraded products assuming possible levels of upgrading. Also gather assay data for North Slope crude oils and crude blends with which GTL product could be blended for transport through TAPS.
- Develop an outline of the physical and chemical property tests that are needed to assess how GTL and GTL/crude blends will behave in the TAPS environment. If questions concerning stability of products during storage exist, these will also be addressed.
- Design tests to provide information on schemes involving commingling GTL product and crude oil on the transportability, costs, and value of GTL/crude blends in refinery market locations. This will include some level of assay for delivered commingled blends with detailed quality data testing for gasoline and distillate boiling range fractions. The commingled blends will span a reasonable range of percents of GTL in the blends.
- Arrange for acquisition of samples of GTL and North Slope crude oils to carry out test program

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Task 4 – Test Program for GTL and GTL/Crude Blends

This task will consist of carrying out the test program developed in Task 3, analyzing the results and studying their impact on GTL transportation issues.

Task 5 – Study of TAPS Flow Behavior

Two modes of transportation of GTL products through TAPS oil pipeline to Valdez Terminal will be considered. These are: 1) sending a certain size of slug of GTL products intermittently between the crude oil transport (batch movement or slugging); and 2) blending the GTL products with crude oil prior to transportation via TAPS (commingling). The objective of this task is to develop information needed to comparatively analyze batch movement of GTL product through TAPS with movement of a commingled blend of crude and GTL product.

For batch movement options, estimate the impact on overall throughput and costs of batch operation. Will there be an increased likelihood of operational problems with downtime and cost implications? Estimates on factors determining and limiting batch cycle lengths will be determined. Pipeline fluid flow simulation study will be conducted to determine the degree of intermixing at the interfaces during GTL batch transport and the volume fraction of GTL material downgraded will be estimated. A minimum slug size of GTL product needed to be transported via 800-mile long, 48-inch diameter TAPS without full disintegration of the slug due to mixing will be determined. The impact of batch cycle lengths (or slug size) on the costs of batch operation will be estimated. Other operational problems that may result from batch flow operations will be described.

The costs and operational problems for shipping commingled GTL/crude blended will be analyzed for comparing with GTL batch shipment options. The fluid flow simulations will also be done for blending cases. The compositions of the GTL-crude oil mixtures at the end of TAPS will be determined and compared for the two modes of transportation.

Task 6 – Overall Evaluation of GTL Transport Modes

In this task, the results of the previous tasks will be integrated to provide an overall assessment of the options for transporting GTL on Alaska's North Slope. For the slugging mode, storage requirements for GTL and crude oil at both ends and additional capital investments, pump-station load factors, blending and separation costs for different GTL slug sizes will be determined. Optimal size of GTL liquid slug will be determined from the economic analysis. For the blending mode, storage requirements for GTL and crude oil at both ends and additional capital investments, pump-station load factors, blending and separation costs for different crude oil – GTL blends will be determined. Optimal blend ratio will be determined from the economic analysis. The two modes of GTL transportation will be evaluated on the basis of cost to benefit ratio. While economic evaluation of GTL process options will have been developed by DOE contractors and process development firms, it would be useful in the course of this project to provide information on cost impacts of building and operating process facilities on the North Slope. The contacts with North Slope operators could be helpful in achieving this objective.

Task 7 – GTL/TAPS Transport Assessment and Final Report
A comprehensive evaluation of all data collected in Tasks 2-6 will be conducted and a final report assessing the feasibility of utilizing TAPS as a transportation vehicle for GTL produced on the Alaskan North Slope will be submitted to the Department of Energy for review and approval.

1.3 GTL TRANSPORTATION ISSUES

Since the Trans Alaska Pipeline System (TAPS) was designed to carry a specific type of crude oil with no provision for batching, the possibility of flowing GTL products through TAPS gives rise to numerous questions. These questions or the transportation issues are dynamic and continue to evolve. Addressing all of these issues is not feasible within the scope of the present study. The following is a summary of the transportation issues that were identified in collaboration with Alyeska Pipeline Service Company (APSC).

- Effect of solids (wax and/or asphaltene) precipitation within the pipeline. Metering of a fluid stream containing solids may pose problems.
- Effect of cold temperature on the GTL material.
- Impact of GTL transport on internal monitoring of corrosion and pigging. Ultrasonic/magnetic properties of the transported fluids will affect monitoring programs. Additionally, GTL may interact with corrosion inhibitors.
- Impact of GTL batching on local refinery (Petrostar, MAPCO) operations. Vapor recovery and volatility of the downstream end product will have to be estimated. The need for adding another berth for vapor recovery will have to be considered.
- Impact on existing metering systems. The TAPS was originally designed for a single grade crude oil system with no provision for batching different fluids.
- Interaction of Drag Reducing Agents (DRA) with GTL.
- Fluid deliverance i.e. ability to transport crude oil and GTL batches or blends. The TAPS was specifically designed for Prudhoe Bay crude, which has a gravity of 24°API to 32°API. On the other hand, GTL has much higher API gravity than Prudhoe Bay crude. In future, as crude oil volume decreases, GTL to crude oil ratio will increase. This is to be considered in the analysis.
- Gelling and cold restart issues. Temperatures at which GTL gelling occur, and gel strengths will need to be determined to address cold restart problems.
- Contamination of GTL product by wax. Wax buildup on the pipeline interior may redissolve in GTL causing contamination and change in GTL properties.
- Mixing at the oil-GTL interface in case of batch mode of transportation.
- To take elevation into account in all Batching/Blending calculations. Here, Austin and Palfrey correlation or derivations by Levenspiel may be used.
- Comparison of simulated mixing behavior with the real mixing behavior by actually testing the samples in slackline. Red Dye Test is used by Alyeska for this purpose.
- GTL compatibility issues which include stripping of asphaltenes, resins, and compatibility for valves, seats.

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- Gel strength and vapor pressure are the critical parameters, which will govern the limits on crude oil and GTL blending ratios. Therefore, gel strength and vapor pressure measurements are of utmost importance.
- Effect of solids buildup (wax and asphaltenes) continues to be of great concern. There is need to study the composition and source of wax, and the effect of asphaltene on wax.

It is obvious from these transportation issues that using the TAPS to move GTL products is far from being a simple matter of switching fluids in the pipeline. Fluid properties and the hydraulics of flowing GTL products and GTL-crude oil blends through TAPS will have to be thoroughly studied before making any decision regarding feasibility of such processes. This project will, therefore, focus primarily on fluid property measurement and development of hydraulic models for studying GTL flow through the TAPS. Finally, an economic analysis will be incorporated to examine the economic feasibility of GTL transportation.

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CHAPTER 2

GTL Process Review

The conversion of natural gas into liquid products (GTL) is a two-step process. The first step involves the conversion of natural gas into Synthesis Gas (syngas), a mixture of Carbon Monoxide (CO) and Hydrogen (H_2). In the second step Synthesis gas is converted into long chain hydrocarbons predominantly paraffins using the Fischer Tropsch (FT) synthesis. Synthesis Gas can be produced by steam reforming, partial oxidation, or autothermal reforming of natural gas. In steam reforming, natural gas and steam are catalytically and endothermically converted to syngas that consists of carbon monoxide and hydrogen. The steam reforming is operated at low temperature. Partial oxidation combines natural gas and oxygen via the exothermic noncatalytic reaction, it is operated at high temperature as opposed to steam reforming. Autothermal reforming combines steam reforming and partial oxidation in one reactor to produce syngas, its temperature is higher than steam reforming and lower than partial oxidation. Autothermal reforming has high thermal efficiency and the desired CO/H_2 ratio for FT synthesis. The FT synthesis is used to convert synthesis gas into higher hydrocarbons, it is conducted in FT reactors, which includes fixed bed, fluidized bed and slurry bed reactors. Fluidized bed is mainly used to produce gasoline, fixed bed or slurry bed is used to produce diesel. The FT synthesis for diesel production produces substantial amount of heavy waxes and lower molecular weight olefins. These products can be upgraded into usable paraffinic fuels that fall in the gasoline and diesel range by upgrading them.

An upgrading step is needed to convert olefins and waxes produced in the FT synthesis into high value products that fall in the boiling range of gasoline and diesel. It is an essential process to improve liquid fuel selectivity and quality of GTL products. The upgrading process includes oligomerization of C_3 to C_6 olefins and hydrocracking of the waxes into valuable paraffins.

In 1992 the first commercial GTL plant using FT technologies was commissioned in South Africa. In 1993, the second commercial natural gas based FT plant was commissioned in Malaysia to convert natural gas into middle distillates. These two commercial GTL plants using FT technologies show the technological viability of FT synthesis process. As the cost of syngas production decreases, the GTL products will be able to compete with the conventional crude oil refined products, and the GTL option via the FT synthesis becomes more and more attractive. The following sections provide a review of syngas production, FT synthesis, and GTL product upgrading processes.

2.1 SYNTHESIS GAS PRODUCTION

The three major methods available to produce synthesis gas from natural gas are as follows:
1) Noncatalytic Partial Oxidation of Natural Gas
2) Steam Reforming
3) Autothermal reforming

2.1.1 Non-Catalytic Partial Oxidation of Natural Gas

Non-Catalytic partial oxidation of natural gas with pure oxygen results in a H_2/CO ratio of synthetic gas close to 2 as shown below.

$$2CH_4 + O_2 \rightarrow 2CO + 4H_2$$
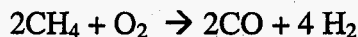
The process operates at 1300 to 1500°C and pressures up to 70 bar. The carbon efficiency exceeds 95%(Eilers et al., 1990). The synthetic gas is composed of H_2 and CO (up to 95% by vol), the rest being N_2, CO_2, H_2O and traces of CH_4 and solids. This process needs a little adjustment to become suitable for the production of the middle distillates.

2.1.2 Steam Reforming

Steam reforming is another important technique for producing synthesis gas from lighter Hydrocarbons and free carbon like coke. In this process a steam reformer is used for manufacturing synthesis gas, the CO/H_2 ratio from a steam reformer using Natural gas is 3 as shown below.

$$CH_4 + H_2O \rightarrow CO + 3H_2$$
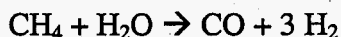
This is a catalytic process operating at 850°C temperature and a pressure of 30 bar over a nickel catalyst. Due to the water gas shift reaction, in which CO reacts with H_2, more H_2 is produced and the actual H_2/CO ratio occurs in the range of 5-7.

$$CO + H_2O \rightarrow CO_2 + H_2$$
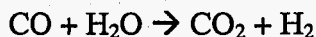
The excess hydrogen produced in this process can be used in the hydrocracking process for the conversion of heavier hydrocarbon to usable products and the Synthesis gas can be used in the Fischer Tropsch synthesis of long chain hydrocarbons.Natural gas required for steam reforming should be free from sulphur, in order to achieve this objective the natural gas is compressed to reformer pressure, commingled with CO_2, heated and passed through a zinc oxide drum where all the sulpur compounds are removed. The steam reformer converts the natural gas into synthesis gas that is cooled, compressed and fed to an amine scrubber where CO_2 is removed and recycled. The scrubbed gas is passed through a membrane separation system to adjust the H_2/CO ratio. The adjusted synthesis gas is ready to be fed into the FT reactor for heavy Paraffin synthesis.

2.1.3 Autothermal reforming

Autothermal reforming is an attractive solution to the production of synthesis gas required for FT synthesis. The process combines steam reforming and partial oxidation in one reactor. Reformed methane and ethane are cryogenically fractionated with steam and water over nickel catalysts. The H_2 rich stream that is extracted from the cryogenic unit can be recycled to the FT synthesis unit. This process provides the desired H_2/CO ratio of 2:1 and it has a high thermal efficiency.

Partial oxidation and steam reforming of natural gas are carried out simultaneously in a fluidized bed syngas generator which provides for good temperature control at significantly

lower temperature than thermal partial oxidation (Chang et al, 1996). This process provides an inherent thermal efficiency and economy of scale advantages as compared to partial oxidation reactors or the multitude of parallel tubes used in the steam reformers. There is a wide range of operating temperatures to produce a H_2/CO ratio of 2/1.

2.1.4 Process evaluation

The steam reforming of natural gas is strongly endothermic. The steam reformer is larger in size. Because of the water gas shift reaction in the steam reforming, the H_2/CO ratio of syngas is in the range of 5-7. Compared with partial oxidation, the steam reforming requires higher capital cost. Partial oxidation of natural gas is exothermic and generates the H_2/CO ratio that is less than 2. In contrast to steam reformer, the partial oxidation unit is very small, conversion of natural gas to synthesis gas is high. Partial oxidation requires an air separation plant and operates at a very high temperature. Autothermal reforming combines steam reforming and partial oxidation in one reactor, which provides the desired H_2/CO ratio of 2:1 and high thermal efficiency. Compared with the steam reforming and partial oxidation, the Autothermal reforming has a lower capital investment and greater potential for economics of scale. Autothermal reforming operates at a lower temperature than partial oxidation. F-T synthesis requires the H_2/CO ratio of 2:1, the H_2/CO ratio of Autothermal reforming of natural gas satisfy this requirement. Autothermal reforming is an advanced technology. The H_2/CO ratio obtained from steam reforming and partial oxidation needs to be adjusted.

Steam reforming operates at a maximum pressure of 30 bar where as the FT synthesis requires a much higher pressure and thus partial oxidation and Autothermal reforming which operates at pressure close to FT synthesis scores over steam reforming by reducing the costs incurred in compressing the gas. The Synthesis Gas production accounts for one half to two third of the total capital cost incurred in the FT synthesis process plant.

Because of the nature of catalysts used in Fischer Tropsch process the synthesis gas should be free from sulphur and sulphur compounds. Natural gas must be desulpherized prior to synthesis gas production. In practice, zinc oxide beds are used to remove traces of sulphur in the last step. After the synthesis gas is ready and is available at the appropriate ratio of H_2/CO of 2, it is subject to Fischer Tropsch synthesis of long chain Hydrocarbons to form GTL.

2.2 FISCHER TROPSCH SYNTHESIS

Fischer Tropsch synthesis of synthesis gas produced from the above-mentioned processes is the second stage for producing GTL from natural gas or coal. The primary reaction that takes place for the formation of hydrocarbons from synthesis gas is as given below.

$$CO + 2H_2 \rightarrow H_2O + -(CH_2)-$$

Synthesis gas reacts over a catalyst in a reactor to form paraffins ranging from light gases to long chain, heavy paraffin. The formation and distribution of FT products is very sensitive to the types of catalysts used and the reactor type. Other factors that govern the product distribution of FT synthesis are pressure, temperature, H_2/CO ratio, recycle ratio and space

velocity. The FT synthesis is composed of five plant sections, which are F-T synthesis, CO_2 removal, recycle gas compression, hydrocarbon recovery, and hydrogen recovery.

Fischer Tropsch synthesis can be conducted in fixed bed, fluidized bed, or slurry bed Reactors. Heat removal from the exothermic reaction, effective contact of feed and catalyst, CO conversion per pass, chain growth probability (α), separation of products from catalyst, the desired end products, initial capital cost, and the economics to scale up are the various factors that drives the choice of reactor type, catalyst, and operating conditions to be used

2.2.1 FT Synthesis in Fixed Bed Reactors
The tubular fixed bed reactors (TFBR) are ideally suited for the production of long chain, heavy paraffin, mainly waxes which are upgraded to produce transportable and usable end products that falls in the diesel range and these have the capability of operating with reactants in the liquid phase.

The purified syngas is converted into a broad range of products in the FT reactors. The tubular fixed bed reactors are used to produce high molecular weight hydrocarbons in the diesel range, catalyst used in tubular fixed bed reactor is a precipitated and promoted Fe-catalyst. Cobalt based catalysts are also used in the Multi-Tubular fixed bed reactors, these have a high probability for formation of long chain ($\alpha > 0.90$) hence maximizing wax production and minimizing the formation of undesired light hydrocarbons.

The tubular fixed bed reactor (TFBR) with Fe-based catalyst operates at a pressure of 25 bar and at 220-250°C, hence can be coupled with syngas formed by steam reforming, which is at a lower temperature and pressure close to the tubular fixed bed reactor. Pressure drop across the TFBR is considerable. Since the fixed bed reactors produce higher molecular weight paraffins the carbon build up probability on the catalysts are much higher and this may cause catalyst breakdown and catalyst regeneration becomes difficult, which in turn causes blockage and need the replacement of catalyst. Therefore there is a maximum allowable peak for temperature to avoid carbon formation on the catalyst and hence the range of products produced using TFBR is limited. The fixed bed route diesel fuel is completely free from sulfur, aromatics, naphthenes and nitrogen compounds (Dry, 1983)

2.2.2 Fluidized Bed FT reactors
Fluidized bed FT reactors are ideally suited for production of lower molecular weight long chain Hydrocarbons from synthesis gas. These reactors can operate only when all the reactants are in gaseous phase. Circulating fluidized bed reactor (CFBR) runs at 330-350°C and at 25 bar. Fixed Fluidized bed reactors (FFBR) operate at a pressure of 24 bar and a temperature of 340°C. Two types of reactors are used to produce a light syncrude for production of gasoline. The catalyst used for the CFBR and FFBR is a fused and promoted Fe-Catalyst. The FFBR has the following advantages over CFBR: its simplicity, elimination of catalyst recycling which requires a complex support system to handle the circulating catalysts load, and low operating and maintenance costs. Catalyst consumption of FFBR is about 40% of that of CFBR, the cost of FFBR is about 40% of that of an equivalent CFBR. The FFBR can operate at lower temperature and somewhat higher pressure and flow rate than CFBR. The selectivity of heavier syncrude and higher conversion are higher as obtained in the FFBR than that in CFBR.

In the FFBR increase of the catalyst bed due to increase in the carbon deposition does not cause an increase in conversion considerably. When increasingly fluidized bed height interferes with operations of the cyclones in the CFBR, carbon formation on the iron F-T catalyst reduces the density and the amount of iron catalyst and requires higher catalysts circulating rates to maintain the reaction rates hence a higher space velocity is involved which reduces the conversion per pass. The catalyst activity declines with time in the CFBR due to carbon deposition, which negatively affects normal operations of the CFBR. The FFBR has a larger Capacity than the CFBR. Compared with CFBR, FFBR eliminates recycling of catalyst, it is simpler and has a lower operating and maintenance costs (Jager et al, 1990, Table 2.1).

The chain growth probability (α) in fluidized bed reactors is less than 0.71 for the stable fluidized bed process, which eliminates production of heavy wax. For very high α, removal of hydrocarbon from the catalyst becomes a serious problem. Catalyst regeneration is frequent due to the high proportion of condensable products in the fluidized bed process.

TABLE 2.1

Comparison of capital cost and energy efficiency for Circulating Fluidized bed (CFB) and fixed Fluidized bed reactors (FFB)

Type		CFB (base)	FFB	FFB
Number of reactors		3	2	2
Operating pressure		25 bar	25 bar	High
Relative capital cost	Reactors	1.00	0.46	0.49
	Gasloop	1.00	0.78	0.71
	Total plant	1.00	0.87	0.82
Energy efficiency (%)		61.9	63.6	64.7
Relative power import		1.00	0.44	0.41

Source: Jager et al, 1990

2.2.3 Slurry Bed Reactors

The slurry bed reactors operate in series, with a number of reactors operating together. Fischer Tropsch synthesis involving slurry bed reactors is composed of five stages; FT syntheses, CO_2 removal, recycle gas compression and dehydration, hydrocarbon recovery and hydrogen recovery. Total of 24 slurry bed reactors are arranged in series in eight parallel trains, each train have two first stage slurry bed reactors, which feed a single second stage slurry bed reactor. The synthesis gas is fed in the first-stage slurry bed reactors operating at 220^0C and 21.4 bar over a cobalt based catalyst. The CO conversion in the first stage slurry bed reactor is 56%. Before the unconverted synthesis gas leaving the first-stage slurry bed reactors is reheated and fed to the single second-stage reactor, it is cooled and flashed to recover liquids. The single second stage reactor operates at a temperature of 220^0C and a pressure of 21 bar, over the cobalt based catalyst with the CO conversion of about 56%. An overall CO conversion per pass is about 82% (Choi et al, 1996). The Slurry Bed Reactor (SBR) can operate at higher

temperature without carbon formation, higher temperature results in wide product selectivities. Hydrogen is recovered from the unconverted synthesis gas and is used in the downstream hydroprocessing units. The excess hydrogen is recycled to the slurry bed reactors. Excess heat is removed by regenerating 11.4 bar steam from tubes within the slurry bed reactors.

The SBR provides on-line removal and addition of catalyst that is not possible with the TFBR, it is simpler and more easily scaled up then the TFBR. The SBR has higher productivity than TFBR. The capital cost required for a large scale SBR plant is less than 40% of that of an equivalent TFBR plant (Jager et al., 1994). High temperature FT synthesis in the FFBR and CFBR results in carbon deposition as opposed to the SBR, which affects the normal operations. The FFBR and SBR have larger capacities and Potential, are suitable to economy of scale.

Synthesis gas can be converted into higher molecular weight linear paraffin in a bubble column slurry bed reactor over a cobalt-based catalyst. The bubble column slurry bed reactor removes substantial heat, released by the FT synthesis reaction via steam generation and provides significant advantages over conventional fixed bed tubular reactor and fluidized bed reactors, this results in high productivity and selectivity and significant economy of scale advantages. 63% straight run liquid fuel can be obtained from a bubble column slurry bed reactor, with a selectivity of 89% conversion of (H_2 + CO) per pass at 514°C. The product upgrading process provides considerable flexibility of product option that includes high quality diesel and jet fuel, chemicals for manufacturing solvents, alcohols, polymers and specialty such as lube oils and waxes. Table 2.2 compares the product slate of cat-feed option and diesel/jet option in the bubble column slurry reactor. The product produced by the bubble column slurry reactor is free from sulphur, nitrogen, nickel, vanadium, asphaltenes, and multi-ring aromatics.

TABLE 2.2
Typical yield from a slurry bubble column reactor

Product	Cat feed option	Diesel / Jet Option
Naphtha	15	30
Diesel/Jet	50	70
Cat Feed	35	0
Total	100	100

Exxon Research and Engineering Company
Source: Chang et al, 1996

Fixed Bed and Slurry bed reactors yield heavier linear chain paraffins that falls in the diesel range as opposed by the fluidized bed reactors that yields comparatively lighter paraffins falling in the gasoline boiling range. The selectivities from different reactors are given in Table 2.3.

TABLE 2.3
Product selectivity for the FT process

	High temp CFB/FFB	Low temperature Fixed Bed Reactor	Low Temperature Slurry Bed Reactor
CH_4	7.0	2.0	3.3
C_2H_4	4.0	0.2	1.9
C_2H_6	3.0	0.6	1.2
C_3H_6	10.7	0.9	3.2
C_3H_8	1.7	0.9	0.8
C_4H_8	9.4	1.0	3.1
C_4H_{10}	1.1	1.4	1.9
C_5+C_6	16.5	4.8	6.1
C_7-160^{0}C (C7-320^{0}F)	20.0	6.2	7.6
160 - 350^{0}C (320-662^{0}F)	15.5	18.3	17.0
+350^{0}C (+662^{0}F)	6.0	62.3	49.5
Oxygenates	5.1	1.4	4.4

CFB: Circulating Fluidized Bed Reactor
FFB: Fixed Fluidized Bed Reactor
High Temperature. CFB/FFB: 330-350^{0}C, 25 bar, H2/CO: >2.0
Low Temperature Fixed Bed: 220-250^{0}C, 25(or 45 bar), 500 hr^{-1}

Source: Dry, 1990

2.3 RANGE OF PRODUCTS

In order to identify the types of GTL products that could flow through the TAPS, GTL product composition and distribution in different process are identified and compared. The FT product selectivity is very important to economics of FT process. Lower C_4 and higher hydrocarbon liquid selectivities are preferred.

The range of products produced by the FT synthesis of natural gas depends on many factors, predominantly,
1) Type of reactors
2) Catalysts Used
3) Operating Condition (Pressure and Temperature)
4) H_2/CO ratio
5) Fresh feed gas composition and recycle ratio

2.3.1 The Fischer Tropsch Process – Reactors, Catalysts and Operating Conditions

All the currently competing GTL processes use the Fischer- Tropsch process to convert synthesis gas to liquid hydrocarbons. Commercial scale Fischer-Tropsch plants were built in Germany during World War II, and during the 1980s and early 1990s in South Africa by the

South African Coal, Oil and Gas Corporation Ltd. (SASOL). In both of these situations the types of feedstock, choice of process operations and products produced were mainly the result of the unique political situations existing in Germany and South Africa.

SASOL has built several plants using the FT process to convert synthesis gas derived from coal with the major focus on gasoline production. Over the years SASOL has explored several reactors, catalysts and types of operating conditions. SASOL initially employed two types of reactors the Avgc, a fixed bed reactor and the Synthol, a circulating fluidized bed (CFR) reactor. In both of these reactors SASOL used an iron-based catalyst. The CFR reactor operated at a higher temperature (315°C versus 225°C for the Avgc) and produced a lighter distribution of products. In 1985, SASOL moved to the fixed fluidized bed (FFP) reactor as an improvement over the CFB reactor. The FFB design that became Synthol 2 was a simpler and hence was less costly to construct and had lower operating and maintenance costs. All of this SASOL FT system used an iron catalyst that produced a light spectrum of products which contain a substantial fraction of olefins along with parafins and a modest fraction of oxygenates as shown in Tables 2.3 and 2.4.

TABLE 2.4

Typical GTL composition from low temperature and high temperature FT synthesis reactors

Product	TFBR		SBR		Fluidized Bed	
	$C_5 - C_{12}$	$C_{13} - C_{18}$	$C_5 - C_{12}$	$C_{13} - C_{18}$	$C_5 - C_{10}$	$C_{11} - C_{14}$
Paraffins	53	65	44	44	13	15
Olefins	40	28	64	50	70	60
Aromatics	0	0	0	0	5	15
Oxygenates	7	7	7	6	12	10
n-paraffins	95	93	96	95	55	60

Source: Jager et al, 1995

While SASOL was building its commercial scale facilities in South Africa a number of companies including GULF Oil, Shell, EXXON and Syntroleum were working on the FT process with a different product focus. These development efforts were focused on producing a high quality distillate product as the primary product. These efforts were to produce a FT product in the very high AFS α range as shown in table 2.6. As discussed in the previous section this would be a product with a large fraction of the product being long chain linear paraffins, which would be wax at ambient temperature. Straight chain paraffins have very high octane number and are excellent as a diesel fuel. The heavier paraffins in the wax range would be hydrocracked to lower molecular weight paraffins and branched paraffins in the diesel boiling range. The heavier α FT products are produced using a cobalt paste catalyst enhanced with a variety of promoting agents based on the development efforts of the companies working on the process.

Shell Oil was the first of those working on a distillate GTL process to build a commercial plant. Shell brought its Shell middle distillate synthesis (SMDS) process on stream at a plant built in Bintulu, Malaysia in 1993. Shell considered 3 reactor types for the plant.

1. A fixed bed reactor
2. An ebulliating (boiling) fluidized bed reactor.
3. A slurry bed reactor

Shell determined that fluidized bed reactor can only be used when the reactants are in the gaseous phase, making it incompatible with producing heavy wax product (Van De Burget-1990). Of these 3 reactors considered, Shell chose the tubular fixed bed reactor for its inherent simplicity. The catalyst is packed in tubes and heat removal is accomplished by contact with the boiling water surrounding the tubes. The SMDS incorporates a hydrocracker to upgrade the heavy waxes. The final product array for the plant for two operating modes is shown in Table 2.5.

TABLE 2.5
Variation in product range and principal properties of SMDS products

Product	Gas oil mode	Kerosene mode
Tops-naphtha (wt%)	15	25
Kerosene (wt%)	25	50
Gas oil (wt%)	60	25
Property	Gas oil (Diesel)	Kerosene
Boiling range (0C)	250-360	150-250
Density (Kgm-3)	780	750
Cetane number	75	-
Smoke point (mm)	-	>50
Freezing point	-	-47
Pour point	-10	

While Shell did not choose the slurry phase reactor, many of the other developers have made that selection. The slurry phase reactor operates with a fine catalyst suspended in an oil medium. EXXON, Syntroleum and SASOL's distillate FT process all use the combination of the slurry reactor and cobalt catalyst. DOE has also participated in the slurry reactor research effort with the construction of the test facility at La Porte, Texas.

All the elements of the GTL process will continue to develop, but it will take the construction and operation of commercial facilities to really determine the best chosen for syn gas generation process, FT reactor types, catalyst and upgrading process. The decline in the estimated capital cost over time and the clear movement on commercial facilities in Nigeria and Qatar should prove the viability of capital cost estimated and provide the commercial operating experience to advance GTL process to full commercial status in the next several years.

TABLE 2.6

TABLE 2.6

Product distribution as a function of chain growth probability (α)

Distribution of Fisher Tropsch Products				Calculated distribution in two stage	
Growth Chance	Product wt%			Product Wt%	
α	$<C_{10}$	C_{10}-C_{20}	$>C_{20}$	$<C_{10}$	C_{10}-C_{20}
0.80	62.4	31.8	5.8	63.6	36.4
0.85	45.6	38.9	15.5	48.7	51.3
0.90	26.4	37.1	36.5	33.7	66.3
0.95	8.6	19.8	71.7	22.9	77.1
0.98	1.6	4.9	93.5	20.9	79.1
0.99	0.4	1.4	98.2	20.0	80.0

Source: Eilers et al, 1990

The FT process yields a wide range pf product from methane to heavy waxes (Table 2.5) with an Fe catalyst you can get a product in the 0.75 to 0.80 range that has lots of naphtha and kerosene with a CO catalyst you could get a product in the 0.90 to 0.95 range with mostly distillate (Kerosene and gas oil) and wax.

2.3.2 Type of Reactors

For Fixed bed and Slurry bed reactors the Selectivity of hydrocarbons (C_5+) is 78 to 80%. These processes can be operated in two modes namely gas oil mode and kerosene mode. Typical product composition as obtained from a tubular fixed bed reactor is given in Table 2.7 (Van Der Burgt et al, 1988).

Gas oil accounts for 60% of the total liquid fraction in the gas oil mode, Kerosene accounts for 50% of the liquid products in the kerosene mode. Fixed Bed and Slurry bed reactors yield heavier linear chain paraffins that falls in the diesel range as opposed by the fluidized bed reactors that yields comparatively lighter paraffins falling in the gasoline boiling range. The selectivities from different reactors are given in Table 2.2.

The Fischer Tropsch technology can be used to produce synthetic crude or syncrude. The syncrude mode consists of production of synthesis gas and syncrude, it does not contain the upgrading of FT products. In the syncrude mode, 25% naphtha, 57% middle distillates and 18% wax are produced. The n-paraffins, i-paraffins and olefins account for 84%, 1% and 15%. The n-paraffins are the predominant products.

TABLE 2.7
Typical composition from a tubular fixed bed reactor

Property	
Sulphur, Nitrogen	None
Pour Point	40
Typical Component Range % wt	
Naphtha	25
Wax	57
Middle distillate	18
n-paraffins	84
i-paraffins	1
Olefins	15
Aromatics	Nil

Source: Van Der Burgt et al, 1988

The fixed bed reactors produces 13.3 % C_4., 18% gasoline (C_5-C_{11}), 14% diesel (C_{12}-C_{18}), 7% C_{19}-C_{23}, 20% medium wax and 25% hard wax. The fluidized bed reactors produce 43% C_4., 40% gasoline (C_5-C_{11}), 7% Diesel, 4% medium wax. Compared to product selectivities of the fixed bed reactor, products of the fluidized bed reactor have more C_1-C_4 paraffins, C_2-C_4 olefins, Gasoline, water-soluble non-acid chemicals and water-soluble acids. They have less diesel, heavy oil and waxes. Lighter hydrocarbons (C_4.) from the fluidized bed reactors are more olefinic than that obtained from the commercial fixed bed reactor. In the fixed bed reactors as catalyst ages the selectivities of CH_4 and Diesel increase, selectivities of C_2-C_4 paraffins, C_2-C_4 olefins, heavy oil and waxes decrease, gasoline selectivity remains constant. In the Fluidized bed reactors as catalyst ages the selectivities of CH_4, C_2-C_4 olefins and gasoline decrease, selectivities of diesel, heavy oil and waxes increase. Catalyst aging results in higher diesel selectivity, and lower C_2-C_4 paraffin and C_2-C_4 olefin selectivities in both fluidized bed and Fixed bed reactors. The fixed bed reactor can be used to produce diesel and waxy products that are linear hydrocarbons. The fluidized bed can be used to produce gasoline.

A cobalt-based catalyst has a high probability of chain growth ($\alpha > 0.91$) that maximizes the wax production. Hydrocracking of the Heavy Fractions from the FT process results in minimizing the waxy product associated with the gas to liquids and hence increases the value of products by enriching it with the lower fractions obtained as a result of hydrocracking.

Product distribution is a function of the chain growth probability α. The product distribution as a function of the chain growth chance α is given in Table 2.6.

2.3.3 Catalyst
Cobalt based catalyst has lower water-gas-shift activity, higher cost, lower olefin selectivity, longer life and higher methane selectivity than the iron based catalyst under equivalent

operating conditions. It can produce high yields of oils and waxes. The Cobalt based catalyst produces lower yield of oxygenated compounds than the iron based catalyst. Iron based catalyst can be used in low H_2/CO feed ratio due to its high water gas shift activity, but it deactivates faster due to oxidation and coke deposition. Because the iron based catalyst has low water-gas-shift activity, the H_2/CO feed ratios need to be adjusted to 2. Water production reduces per pass conversion with the iron-based catalyst.

Fe based catalyst has been used to produce lower molecular weight mix, mainly by Sasol and Sasol has done it with several types of reactors, starting with average fixed bed, then they have used circulating fluid bed and fixed fluid bed. They have done so for South Africa application and stayed with Fe Catalyst as their focus was gasoline. Typical Gas to liquids (GTL) composition obtained by using different catalyst is given in Table 2.8.

For application outside South Africa (e.g. Nigeria, Qatar), Sasol is planning to use the slurry Phase reactors and Cobalt catalyst, Sasol has experience with slurry reactor and has chosen the combination of cobalt based catalyst & Slurry phase reactor for producing a diesel product in Nigeria and Qatar.

Almost if not all, the GTL process developments are going with some type of cobalt catalyst and a focus on diesel catalyst. Shell is still staying with the multi tubular fixed bed reactor. Table 2.6 shows the typical product composition from different catalysts. Catalyst activity can be increased by increasing pressure, temperature and H_2/CO feed ratio.

TABLE 2.8

Typical product composition from different catalysts

Final Product Mass %	(Fe Catalyst)		(Co Catalyst, Fixed bed)
	High Temperature F-T (Fluidized Bed)	High Temperature F-T (Fixed or slurry bed)	
"Fuel gas"	18	10	20
Gasoline	35	19	21
Diesel Fuel	47	70	58
Diesel/Gasoline Mass Ratio	1.3	3.7	2.8

Source: Dry, 1990

We are still in a technology development phase and reactor and catalyst best choices are not clear. It is partly clear that it will be some form of cobalt catalyst and that it will be primarily diesel fuel product. Paraffinic materials have high Cetane number and low octane number and don't reform easily for gasoline blend stock. The paraffinic naphtha will probably be used as an ethylene cracking stock. It could be used as a fuel cell in automobiles but not likely in the near future.

2.3.4 Pressure

As pressure increases, the (H_2 + CO) conversion, the oxygenate content and the heavier hydrocarbon selectivity increases. When pressure is increased the fresh and recycle feed are increased in proportion, and the percentage conversion remains unchanged, Thus increasing pressure means that the reactor's production capacity increases in proportion. For FT synthesis the operating pressure is dependent upon the pressure at which syngas is produced because gas compression accounts for a significant portion of the overall costs.

2.3.5 Temperature

As temperature increases the product selectivity is oriented toward the lower molecular weight products. At high temperature, secondary reaction occur, aromatics are formed, higher Ketones, acid and ring compounds are produced. The high temperature of the FT synthesis produces more olefins, less paraffins and more gasoline than the low temperature FT synthesis. As temperature increases the degree of branching in FT products and the percentage conversion increase, the low temperature FT synthesis yield the larger fraction of straight-chained products.

High temperature CFBR and FFBR have the same FT product distribution of 36.9% C_4., 16.5% C_5+C_6, 20% C_7- 320^0F cut, 15.5% 320-662°F cut, 6% 662^0F cut and 5.1% oxygenates. Low temperature slurry bed reactor produces more C_4., C_5+C_6, C_7.320^0F cuts and oxygenates, and less 320-662^0F and +662^0F cuts than the low temperature fixed bed products. C_2-C_4 cuts obtained from low temperature slurry bed process are more olefinic than that from the low temperature fixed bed process. High temperature CFBR/FFBR process produces more C_4., C_5+C_6, C_7-320^0F cuts and oxygenates and less 320-662^0F and +662^0F cuts than that of low temperature fixed bed and slurry bed processes.

Low temperature FT products are free from aromatics. The high temperature FT products contain 5% aromatics in the range of C_5-C_{10} and 15% aromatics in the range of C_{11}-C_{14}. Low temperature fixed bed reactor produces 53% paraffin, 40% olefins, 7% Oxygenates in the range of C_5-C_{12}. Low temperature slurry bed reactors produce 29% paraffins, 64% olefins and 7% oxygenates. Low temperature fixed bed reactors produce more paraffins than the slurry bed reactors in the range of C_5-C_{18}. Typical GTL composition from low temperature FT and high temperature FT synthesis reactors are shown in Table 2.4.

High temperature FT process produces more olefins and oxygenates and less paraffins than the low temperature FT process. At low temperature, the n-paraffins are predominant in paraffins.

2.3.6 H_2/CO Ratio

As H_2/CO ratio decreases, the product selectivity shifts to higher molecular weight products. As H_2/CO ratio increases the syngas conversion increases.

2.3.7 Space Velocity

As space velocity of the fresh feed gas increases, the oxygenate content and the olefinity of the F-T product increase and the (H_2 + CO) conversion decreases.

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2.4 FT PRODUCTS REFINING AND UPGRADING

The FT synthesis produces a wide range of products from methane to heavy paraffin waxes. Therefore, product refining is an important process to upgrade the FT products and to increase the quality of the end products that would have high premium in the markets. The FT products are condensed. Water and liquid hydrocarbons are obtained. All water is fed to FT water work-up unit to extract alcohols, ketones, aldehydes and acids. The olefins of C_3 and C_4 products separated from the tail gas are oligomerized to gasoline over a phosphoric acid/Kieshelguhr catalyst at about 190^0C and 38 bar. The C_3 and C_4 paraffinic products are sold as light petroleum gas (LPG). The light oil (C_5 to C_{12}), which contains about 75% olefin, is processed over an acid zeolite catalyst at about 400^0C and 1 bar. The oxygenates are converted into olefins and olefins are isomerized. The process improves the research octane number (RON) of the gasoline from about 65 to 86(Pb free). The liquid hydrocarbons from the FT reactor are treated to extract gasoline (RON 35) and diesel. The gasoline can be improved by catalytic isomerization, which forms a product with RON of about 65. The waxes from the reactor are vacuum distilled and the residue is fed to the hydrocracking unit. The wax cuts are hydrorefined to eliminate all oxygenates and olefins over nickel catalyst at about 270^0C and 70 bar. The tailgas includes methane, ethane, ethylene, CO_2 and the unreacted synthesis gas. A part is blended with methane from the cryogenic unit produce town gas. The H_2 extracted from the tailgas in the cryogenic unit is used to produce NH_3. The remaining tailgas is catalytically reformed over nickel catalyst at about 1000^0C with steam and oxygen to produce synthesis gas, which is water scrubbed to remove CO_2 and recycled to the reactor.

Upgrading of the FT products from the fixed bed and slurry bed reactors becomes necessary for the conversion of heavier waxes into usable and lighter products in the diesel range. Product upgrading is an optional stage added to the FT synthesis of GTL to increase the conversion and premium of the end product, it might not be necessary when a fluidized reactor is used for Paraffin synthesis. The products will be upgraded as a part of the GTL process in the conversion of the heavy wax fraction. This assumes that the GTL plant will be producing a high α product with a significant fraction of the reactor production being high molecular weight (720 carbon number) straight chain paraffins.

The primary reactions that govern the upgrading process is hydrocracking. After obtaining heavy paraffin from the Tubular fixed bed reactor, they are converted into middle distillates to produce kerosene and diesel in the hydrocracking process, which is a mildly selective process using a dual function catalyst. Small amount of oxygenates which exists in the waxy products must be removed. Hydrogenation of the olefins, the hydroisomerisation and the hydrocracking are performed in the trickle flow reactor to produce the desired middle distillates.

Hydrocracking is executed in a conventional column and the product fraction boiling above the diesel range is recycled back to the unit. Desired products are obtained by varying the recycling rate and cut-points as well as the single pass conversion. The process operates at a temperature between 300 and 350°C and at 30-35 bar.

The upgrading process with multi tubular fixed bed reactors for FT synthesis includes hydroisomerization and hydrocracking of heavy paraffins. Straight chain paraffins have excellent cetane number and are great diesel fuel (CI engine) except when in cold temperature they solidify in tank or worse get in the vehicle fuel lines. Thus a desirable fuel is one that has high cetane but also has good cold flow properties. That is we need to sacrifice a little of the cetane so that we don't have a fuel that will form solid at cold temperatures.

Hydroisomerization converts the straight long chain paraffins to branched chain paraffins, which have improved "cold flow" properties. Some process uses oligomerization of the C_3 to C_6 olefins as a part of upgrading the products. C_3/C_5 isomerization, catalytic reforming of the hydrorefined C_7/C_{10} cut and hydrocracking of waxes are also practiced as a part of upgrading the GTL. Table 2.9 shows the GTL production from low-temperature fixed bed and high-temperature fluidized bed Fischer-Tropsch operations. Separation and upgrading of the FT products account for 10-15% of the total investment cost. The syncrude mode using fluidized bed reactors eliminates the upgrading process. No matter what alternate batch or transportation mode is used, there is some mixing between crude oil and liquid fuels or syncrude, which requires facility to separate crude oil and liquid fuel at the receiving end of pipeline. The cost of separation between crude oil and liquid fuels is more expensive than that between crude oil and syncrude.

In order to improve liquid selectivity and reduce the cost of synthesis gas production, the combination of both oligomerization of the C_2 to C_6 olefins and hydrocracking of the waxes are preferable.

TABLE 2.9
GTL production from low-temperature fixed bed and high-temperature fluidized bed Fischer-Tropsch operations

Product selectivity after workup	Fixed bed Scheme (%)	Fluidized bed Scheme (%)
CH_4	3	11
C_2	2	8
$C_3 + C_4$	6	4
Light naphtha	10	14
Diesel Fuel	77	57
Water soluble oxygenates	2	6
Origin of Diesel		
Straight-run	28	38
Olefin oligomerization	7	54
Wax hydrocracking	65	8

Source: Dry, 1983

The upgrading process in a GTL plant will be a hydrocracking process that will crack the heavier wax molecule and will form isomers, i.e. branched chain paraffins as opposed to linear

paraffins. A branched chain paraffin of the same molecular weight as a straight chain paraffin stay as a liquid at a cold temperature compared to the straight chain paraffin that will for a waxy solid. So the operating temperature of hydrocracking and its catalyst are chosen to hydroisomerize the heavy wax feed. Sasol has combined with chevron that is providing their isocracking (a hydrocracking technology) for the Nigeria and Qatar projects. Shell and Exxon employs similar upgrading.

2.5 SUMMARY

Amongst the syngas production techniques autothermal reforming seems to be highly attractive because of the excellent thermal efficiency, desired H_2/CO ratio as required for the F-T synthesis. Autothermal reforming operates at a lower temperature and has a lower capital investment as compared to steam reforming and partial oxidation. It has a greater potential for economics of scale.

The slurry bed reactor is best suited for the FT synthesis process because of its higher CO conversion capacity, Normal operation at higher temperatures without carbon deposition on the catalyst surface and it's online removal and addition of catalyst ability. Besides this Slurry Bed Reactor gives higher productivity at a lower cost.

Cobalt based catalyst is more promising for the FT synthesis. It has higher paraffin selectivity as desired for the end GTL products, has lower olefin selectivity and a longer life as opposed by the iron-based catalyst. It can produce higher yields of oils and waxes. Cobalt based catalyst has lower water gas shift activity as compared to iron based catalyst which enables it deliver the desired H_2/CO ratio required for F-T synthesis.

The FT synthesis yields liquid fuels in the diesel and gasoline boiling range. It also yields undesirable high molecular weight paraffinic products and olefins. These undesirable products can be upgraded to useful fuels. The combination of both oligomerization of C_2 to C_6 olefins and hydro cracking of the waxes in the Upgrading process can be used to improve liquid fuel selectivity with higher paraffin productivity. Oligomerization of the C_2 to C_6 olefins into gasoline and diesel can be used to reduce the cost of synthesis gas production.

GTL products obtained from FT synthesis and upgrading of olefins and waxes are free from sulphur and aromatic compounds. These products are paraffinic in nature and have similar liquid density and viscosity as that of diesel and gasoline.

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CHAPTER 3

GTL MATERIAL TESTING

3.1 INTRODUCTION

Transportation of GTL products from the Alaskan North Slope through the Trans Alaska Pipeline System (TAPS) poses several operational problems dependent on the physical and chemical properties of the GTL products and their blends with the Alaskan North Slope Crude oil (ANSC). It may be noted that TAPS was originally designed for carrying a specific type of crude oil. The pipeline being located in an extremely cold region adds to the limitations on moving different fluid types through it.

Two possible modes of transporting GTL through TAPS are being considered. These modes are (i) batch and (ii) commingled with crude oil. In the batch or slug mode, crude oil and GTL are moved as alternating batches or slugs. A second mode is to blend or commingle the GTL products and crude oil into a single liquid phase prior to entering TAPS. In either mode of transportation, the feasibility of moving GTL products through TAPS will depend on the physical properties of the GTL and GTL-crude oil mixtures. For example, fluid properties such as density and viscosity are required to calculate the pressure and horsepower requirements. Since these fluid properties depend significantly on temperature, it is necessary to estimate the properties as functions of temperature as a part of evaluating the feasibility of flowing the fluids through TAPS.

Additional problems in transportation of GTL products through TAPS may be created by components of the transported material being deposited inside the pipe. Buildup of such deposits may impede fluid flow to the extent where the deposits need to be mechanically removed. Furthermore, deposited solids from the pipe walls can re-dissolve into the pure GTL slug in the batch mode of transport, causing serious contamination problems. Finally, one of the most important issues involved in studying the feasibility of moving GTL products through TAPS is cold restart of the pipeline. The material flowing through TAPS must be such that flow through the pipeline can be safely initiated following an extended winter shutdown.

One of the objectives of this project is to select tests and evaluate samples of GTL products and GTL-crude oil blends in order to assess the feasibility of transporting such materials through TAPS. This chapter will discuss some of the tests that can be used to assess the feasibility of transporting GTL products through TAPS and to evaluate the likelihood of cold flow problems. Density and viscosity measurements as functions of temperature are necessary for calculating horsepower requirements. Experimental data on density and viscosity of naturally occurring hydrocarbon mixtures are not well documented. However, measurements on true boiling point (TBP) fractions of various Arab (Amin and Beg 1994) and North Sea Crudes (Dandekar et. al., 1998), Alberta bitumen (Miadoyne et al., 1994) and Saskatchewan oils (Singh et al., 1994) have been reported. Additionally, the predictive capabilities of various viscosity correlations still remain a weak link. Therefore, the need for accurately measured experimental data is indispensable for evaluating the feasibility of transporting GTL products through the TAPS.

Another important GTL testing parameter is the gel strength, which is one of the most important properties necessary to evaluate the feasibility of cold restart of TAPS. The measurement of gel strength gives an indication of the so-called 'cold restart pressure' at which the liquid in the pipeline can yield under the given arctic conditions in Alaska. Thus, bearing in mind the significance of this parameter, gel strengths of various GTL and GTL-ANSC blends need to be determined by the rotating vane technique at different temperatures.

As far as the transport of GTL through TAPS is concerned, there still exists an uncertainty as to exactly which particular type of GTL product will be the potential candidate for flow through TAPS. This chapter describes the types of GTL products that were tested. A number of GTL product matrix possibilities exist, which are dependent on GTL process options such as catalyst used, process employed, operating conditions and type of GTL product upgrading, and other factors such as gas quality, Alaskan North Slope (ANS) logistics etc. In this study, a GTL sample product was obtained from the US Department of Energy and different fractions of the sample material were used to simulate variation of GTL product types. Thus, we were able to represent a wide range of GTL material that could potentially be produced from a North Slope GTL plant. Since GTL can be transported through TAPS as slugs or as commingled fluid, the tests were performed on GTL as well as GTL-crude oil blends.

Experimental studies on measurement of density and viscosity of GTL, crude oil, and GTL-crude oil blends as a function of temperature at atmospheric pressure were carried out. In this chapter, the viscosity and density measurement equipment, procedures, and all other supporting activities are presented in detail. All measurements were performed at the Petroleum Development Laboratory of The University of Alaska Fairbanks (UAF).

The gel strength measurements on the samples were performed at Westport Technology Center International (WTCI) facilities in Houston, Texas. In addition to the gel strength measurements WTCI also carried out Gas Chromatographic (GC) analysis of one of the GTL and TAPS crude oil blends to define the quantitative compositions from C2 to C30+. The work performed by WTCI is summarized in this chapter. The detailed reports written by WTCI on the gel strength measurement work are attached in the Appendices B and C.

The results from these tests are discussed in this chapter. Preliminary considerations as to what type of GTL product will likely have to be produced on the North Slope in order to be transportable over TAPS are also presented.

3.2 QUALITY ASSURANCE PROJECT PLAN

As part of this study, a Quality Assurance Project Plan (QAPjP) was prepared in connection with the potential collaborative project work between UAF and the Alyeska Pipeline Service Company (APSC). The collaborative work includes various aspects of transporting crude oil and/or GTL blends. The QAPjP is presented in Appendix A.

3.3 DESCRIPTION OF THE DENSITY AND VISCOSITY MEASUREMENT APPARATUS

3.3.1 Anton-Parr Densitometer

All density measurements were carried out in the Anton-Paar vibrating tube densitometer. The densitometer (DMA 45) is designed to measure the density of liquids at atmospheric pressures and for temperatures up to 60°C. The sample temperature is controlled by circulating constant temperature mineral oil through the densitometer. The apparatus is shown in Figure 3.1.

The Anton-Paar vibrating tube densitometer contains a U shaped oscillating sample tube. The electronic part of the meter excites the oscillator. A built-in quartz clock measures the period of oscillation; approximately every two seconds and transmits the value to the built-in processor. Then the processor calculates density and displays the value in the digital display. There are two injection ports to the right of density meter on the side. The lower filling inlet is used to inject samples while the upper filling inlet is used to flush the samples and to dry the U-tube. There is an in-built pump, which can be switched on to pass air through the upper filling inlet for drying purposes.

The density meter has a constant buffer where the values of the calibration constants A and B are stored. There is also a display selector, which is used to select the display between A, B, time period of oscillation, T and density values. After every density measurement, the samples are flushed out of the sample tube by giving a proper toluene wash followed by acetone wash and drying the tube using air.

The calibration and density measurement procedure is explained in Section 3.6.1.

3.3.2 Brookfield Rotational Viscometer

In this section a brief description of the Brookfield Rotational viscometer used for the measurement of rheological properties of GTL, crude oil and their blends is provided. The basic unit of the Brookfield Rotational viscometer possesses the capability of using three different kinds of attachments/measuring systems. These are (i) LVDV-II+ Cone Plate Viscometer (ii) LVDV-II+ Viscometer with Small Sample Adapter and (iii) LVDV-II+ Viscometer used with a 500 ml working volume of the sample.

All viscosity measurements reported in this report were carried out using the LVDV-II+ Cone Plate Viscometer. A picture of the apparatus is shown in Figure 3.2. There are three main components of this experimental setup: (i) Cone Plate Viscometer (ii) Refrigerated Bath and (iii) Wingather Data Gathering Software.

The LVDV-II+ cone plate viscometer has a cone and plate arrangement for viscosity measurement. The electronic cone spindle CPE-40 is used to measure viscosity values as low as 0.2cp. Besides, the sample volume needed to measure viscosity is only 0.5cc. This feature is useful when the sample volume availability is limited. The measurement range of LVDV-II+

cone plate viscometer with a CPE-40 spindle is 0.2cp to 3070cp. This fits in well with the GTL and crude oil viscosity measurement span.

The refrigerated bath is connected to the viscometer sample cup by a rubber tubing. The bath fluid temperature can be precisely controlled using a temperature-adjusting knob. The temperature of the sample is controlled by circulating the bath fluid from the bath to the sample cup jacket.

The viscosity data is gathered using a software called "Wingather" manufactured by Brookfield. Wingather can be used to record, save, edit, plot and analyze the testing data. The cone plate viscometer also has an electronic gap-setting feature, which has to be used to set the right gap between the cone and plate (Figure 3.2). It is desirable to set the gap every time the spindle is replaced. The viscometer can be controlled using the touch screen functions available on the face of the viscometer.

The calibration and viscosity measurement procedure is explained in Section 3.6.2.

3.4 TEST FLUIDS FOR DENSITY AND VISCOSITY MEASUREMENTS

The density and viscosity measurements were performed using the light GTL sample supplied by the US DOE (from LaPorte facility). The light GTL sample consisted mostly of C5 to C12 and progressively decreasing amounts of the heavier alkanes (Table 3.1). The rationale for these tests was to establish trends in GTL properties with temperature and to estimate how mixing with crude oil will affect the properties. The GTL sample was used on "as-is" basis, i.e., the sample was not treated in any way at UAF because only preliminary results were desired as a screening tool. The composition of the GTL is shown in Table 3.1. The crude oil samples were supplied in pressurized 1000 cc, CP2-M Welker cylinders. The aliquots of the crude oil were subsequently drawn from the Welker sample cylinder, into glass receptacles for carrying out all the required tests. However, prior to the collection of samples from the Welker cylinders a crude oil reconditioning and aliquoting procedure was followed. The crude oil composition is provided in Table 3.2. This procedure is described in detail in the following section.

3.4.1 Crude Oil Reconditioning and Aliquoting

It is necessary to recondition the samples back to original pipeline sampling conditions before taking the sample out of the Welker cylinder. This is carried out to ensure that the original composition of the sample is retained in all the test aliquots produced from the Welker cylinder sample. Aliquot samples are the sub-samples produced from original test samples. A sequential procedure to recondition the crude oil followed by aliquoting is outlined in this section.

Welker constant pressure sample cylinders are used to maintain the sampled product at pipeline pressures, provide adequate mixers, and facilitate easy and safe removal of sample.

Welker cylinder has two ends that are separated internally by a floating piston. One end is marked "product inlet" or "sampling end" and the other is the "precharge end". It also has two pressure gauges to read the pressure at both the ends of the cylinder. Quick connects and disconnects are used on both the ends to allow easy removal of sample without any leak. On top of the precharged end is an indicator rod. The mixing rod is used to homogenize the sample by mixing.

In the crude oil reconditioning procedure, the Welker cylinder is immersed vertically in a 55-gallon drum filled with water. The water filled drum is heated using two industrial belt heaters that are wrapped around the drum. The re-conditioning temperature is raised between 70 to 80°C. The crude oil re-conditioning is carried out for a period of at least two hours. During heat up and reconditioning, the crude oil sample is mixed manually to ensure homogenization.

After completion of the re-conditioning procedure the Welker is removed from the water filled drum and nitrogen supply line is opened to the precharge end of the Welker cylinder. This pressure source from the nitrogen cylinder, which is set at the original Welker sampling pressure, acts as the driving force for expelling the sample. The whitey valve at the sample end of Welker cylinder is opened with the metering valve closed. Subsequently, the metering valve is opened and sample aliquots are directly transferred into the storage containers immediately and sealed with pressure tight caps. The crude oil re-conditioning and aliquoting assembly is shown in Figure 3.3.

3.5 TEST CONDITIONS FOR DENSITY AND VISCOSITY MEASUREMENTS

All density and viscosity measurements were carried out at atmospheric pressure. The density data were measured in the temperature range of 23.8 to 60°C, whereas the viscosity data were measured in the temperature range of 20 to 60°C.

3.6 EXPERIMENTAL PROCEDURES FOR DENSITY AND VISCOSITY MEASUREMENTS

3.6.1 Density Measurements

3.6.1.1 Calibration

Prior to carrying out any density measurements, the densitometer is calibrated as follows-

i. After a stable temperature is reached, the calibration constants A and B are determined and entered into the constant buffer before measurement is started. Both constants are dependent on temperature and must therefore be re-determined if a change in measuring temperature occurs.
ii. To calculate the calibration constants, the display selector (under the cover plate on top of the DMA 45 meter) is set to position T; the value of the period of oscillation "T" is then shown on the numerical display.
iii. The illumination light is turned on to observe sample through the observation window. Now, a small volume (about 0.7 cc) of de-ionized water is introduced into the sample

tube from the bottom opening using a suitable syringe. This can be observed through the observation window with the light being switched on.

iv.   Filling is complete when the liquid has exceeded the upper of two thickenings on the oscillator. The syringe is left in the lower filling inlet and the light is switched off to maintain temperature stability. The temperature is allowed to reach an equilibrium value; this is indicated by a stable value on the display.

v.   The reading is taken for the period of water and recorded. This value T_w corresponds to the period for the water-filled oscillator. For this first measurement, the water should be left in the oscillator for approximately 15 minutes, checking that no variation in the T_w value occurs during this time. If the value of T_w varies by a maximum of one unit in the last significant digit, then it is an indication that the temperature control is working efficiently.

vi.   Next, the water is washed out of the measuring cell using alcohol. The air outlet is connected to the upper filling inlet using a piece of tubing. The pump is switched on and dry air is blown through the tubing into the sample tube until the displayed values remain constant for some time.

vii.   Then, the pump is switched off and the dry air in the U-tube is allowed to come to thermal equilibrium with the test temperature. The value for the period of air, T_a is recorded. The density of air at the temperature of test is calculated using the following equation:

$$d_a, g/ml = 0.001293[273.15/T]*[P/760]$$

Where,

T = temperature, °K
P = barometric pressure, torr.

The density of water, d_w at the test temperature is determined by referring to Table 3.3.

Using the observed T-values and the reference density values for water and air, the values of the constants A and B are calculated using the following equations:

$$A = [T_w^2 - T_a^2]/[d_w - d_a]$$

$$B = T_a^2 - (A)(d_a)$$

where:
T_w = observed period of oscillation for cell containing water,
T_a = observed period of oscillation for cell containing air,
d_w = density of water at test temperature, and
d_a = density of air at test temperature.

viii.   These values (A and B) and the corresponding measuring temperature should be noted down in pencil on the panel situated at the back of the cover of the constant buffer. The

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constants are stored in the buffer by adjusting the corresponding switches with a small screwdriver.

ix. The correct settings of the switches can be checked once more by adjusting the display selector (by checking the values of A, B separately).

x. If the selector switch is set at position "ρ", then the equipment may carry out density measurement.

The above calibration procedure was consistently followed for all the test temperatures.

3.6.1.2 Test Sample Density Measurement

The density measurements on GTL, crude oil, and their blends is carried out in the following manner –

i. The illumination light is turned on to observe the sample through the observation window.

ii. During measurement, a small amount (about 0.7 cc) of sample is introduced into the clean, dry sample tube of the instrument using a suitable syringe. At this point, it is made sure the sample introduced is enough to fill beyond the suspension point on the right-hand side.

iii. The sample is allowed to equilibrate to the test temperature before proceeding to evaluate the test sample for the presence of unseen air or gas bubbles.

iv. The sample tube is examined carefully. It is made sure that no bubbles are trapped in the tube, and that it is filled to just beyond the suspension point on the right-hand side. The sample must be homogeneous and free of even the smallest bubbles.

v. The illumination light is turned off immediately after verification, because the heat generation can affect the measurement temperature.

vi. After the instrument displays a steady reading to four significant figures for density and five for T-values, indicating that temperature equilibrium has been reached, the density value is recorded. This recorded value if the final result.

vii. The sample tube is flushed with a suitable solvent and dried and the calibration is checked as described previously prior to introducing another sample.

3.6.2 Viscosity Measurements

3.6.2.1 Calibration

The following calibration procedure is adopted for the LVDV-II+ Cone Plate Viscometer. In this work, Brookfield certified viscosity standards were used for calibration.

i. The sample cup inlet/outlet ports are connected to the water bath inlet and outlet and the bath is set to the desired test temperature.

ii. The appropriate sample volume is determined. For spindle CPE-40, the sample volume of 0.5 ml is used. For other types of spindles, Brookfield's Instruction Manual is referred to determine the correct sample volume required for the spindle to be used.

iii.   A viscosity standard fluid that will give viscosity readings between 10% and 100% of full-scale range is selected. It is best to use a viscosity standard fluid which is closer to the maximum viscosity for a given cone spindle/speed combination.

iv.   With the motor off, the sample cup is removed and the necessary volume of viscosity standard fluid is placed into the sample cup.

v.   The sample cup is attached back to the viscometer and sufficient time is allowed for the sample, cup and the cone to reach the temperature equilibrium.

vi.   The temperature of the bath fluid is measured using a calibrated thermometer and it is made sure that it is within ± 0.1°C of 25°C (or specific calibration temperature). A waiting period of at least 15 minutes is given to make sure that sample temperature is same as bath fluid temperature.

vii.   The temperature is recorded.

viii.   If the fluid is at the test temperature, the motor is turned on. For the desired speed(s), viscosity is measured and data recorded using the Brookfield Wingather Software Program. Note: The spindle must rotate at least five (5) times before readings are taken.

ix.   The viscosity reading should equal the cP value on the fluid standard to within the combined accuracies of the viscometer and the viscosity standard, in steps shown below as the acceptable calibration.

STEP 1: The full-scale viscosity range (A) is calculated using the equation:

Full Scale Viscosity Range, "A" [cP] = TK * SMC * 10,000/RPM

Where,
     TK = Viscometer torque constant, 0.09373
     SMC = Current spindle multiplier constant, 0.327
     The viscosity is accurate to ±1% of A (cP) = "X" cP

STEP 2: If the viscosity standard fluid is "B" cP, its accuracy is ±1% of B (cP) = "Y" cP

STEP 3: If the precision of the thermometers, above -50°C, is within ±0.3% and if the variation of Brookfield viscosity standards is approximately 2% with 0.3° C variations in temperature, let that variation (approximately ±2% of "B" cP) be "Z" cP.

STEP 4: So, total allowable error is (X+Y+Z) cP = ± (X+Y+Z) cP.

STEP 5: Therefore, a viscometer is assumed to be calibrated if the error in the viscosity reading lies within ±(X+Y+Z) cP of the standard fluid viscosity. Any reading outside these limits indicates a viscometer problem.

3.6.2.2 Test Sample Viscosity Measurement

A systematic procedure followed to (i) set up the cone plate viscometer, (ii) set the gap between the cone & plate and (iii) measure viscosity using cone plate viscometer is explained as follows:

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Setting Up The Cone Plate Viscometer

i.   It is made sure that the cone plate viscometer is securely mounted to the laboratory stand. The instrument is leveled by referring to the viscometer bubble level and adjusting the leveling screws. The viscometer is then zeroed with no cone or cup attached so that the face of the viscometer displays 0% torque.

ii.   The sample cup inlet/outlet ports are connected to the water bath inlet and outlet.

iii.   All measurements are made as close to target test temperature as possible. The initial water bath temperature is set to test temperature. Sufficient time is allowed for bath to reach the test temperature.

iv.   The cone plane viscometer comes with a special cone spindle(s) (for example CPE-40 spindle) which contains the Electronic Gap Setting feature.

v.   With the motor off, cone spindle is threaded by using the spindle wrench to secure the viscometer coupling nut; the spindle is gently pushed up on the coupling nut, which is held securely with the wrench. The cone spindle is threaded by hand. Note: Left Handed Threads.

vi.   The cup is attached, taking care not to hit the cone with the cup.

Setting the "Gap" between the Cone and the Plate

i.   The toggle switch is moved to the right; this turns on (enable) the Gap Setting Feature. The Pilot (red) light is illuminated.

ii.   If the contact light (yellow) is illuminated, the micrometer adjustment ring is turned clockwise until the light is no longer illuminated.

iii.   If the yellow contact light is not illuminated, the micrometer adjustment ring is slowly turned in small increments in the counter-clockwise direction. The micrometer adjustment ring is continuously moved slowly in the counter-clockwise direction until the contact light (yellow) first turns on. This is the "Hit Point".

iv.   The sliding reference marker is adjusted, right or left, to the closest full-scale division mark.

v.   The micrometer adjustment ring is adjusted one scale division to the left to meet the line on the sliding reference marker. The yellow contact light goes OFF.

vi.   Now, the gap space needed for viscosity measurement is established. The toggle switch is turned OFF (left); the red pilot light goes off.

vii.   The sample cup is carefully removed. The motor is turned OFF when the sample cup is being removed.

Viscosity Measurement using Cone and Plate Viscometer

i.   The appropriate sample volume is determined. For spindle CPE-40, the sample volume of 0.5 ml is used.

ii.   With the motor off, the sample cup is removed and appropriate sample volume is injected into the cup.

iii.   The sample cup is attached to the viscometer and sufficient time is allowed for the sample, cup and cone to reach temperature equilibrium.

iv. The spindle/speed combination that will give a minimum scale reading of 10% (% torque reading displayed at the bottom right of the viscometer touch screen) at the lowest speed to be tested is selected.

v. The temperature is monitored by measuring the temperature of the bath fluid using a calibrated mercury thermometer. Sufficient time is given so that sample temperature is the same as the bath fluid temperature.

vi. When the stable test temperature is reached, the temperature is recorded.

vii. The motor is turned on. The desired speed(s) selected is set for the material under test. The viscometer is allowed to run until the reading has stabilized. The data is recorded using Wingather Software Program.

viii. The viscometer speed is increased stepwise and the viscometer reading is recorded after it stabilizes at each speed. After an observation has been made at the top speed, the speed is decreased in steps to the slowest speed, recording the viscometer reading after stabilizing at each speed. Note: It is preferable to change speed when the motor is running.

ix. After the last reading has been taken at the slowest speed, the viscometer is shut off and the viscometer and the specimen are allowed to stand undisturbed for 5 minutes. At the end of the rest period, the viscometer is started at the slowest speed and the initial maximum and equilibrium scale readings are recorded.

x. The Wingather program is stopped and the data is saved.

xi. If the data indicate that at the top speed, measured viscosity as a function of shear rate is stable, the measurements are complete. However, if the data indicate that viscosity is still decreasing as shear increases additional measurements are carried out, including a new sample with temperature pre-treatment, at higher shear rates.

3.7 EXPERIMENTAL RESULTS AND DISCUSSIONS

3.7.1 Density Measurements

The density data were measured in conjunction with the American Society For Testing and Materials (ASTM) D 4052 method using the Anton-Paar digital densitometer. All the density measurements were carried out at various temperatures in the range of ambient to 60 °C. The test temperatures were accurate to ± 0.1 %. The test samples were 100 % ANSC oil, three blends of ANSC: GTL in the volumetric proportions of 3:1, 1:1, and 1:3, and 100 % GTL. Sample requirements for the densitometer were as low as 0.7 cc. The measured density data are presented in Table 3.4 and represented in Figure 3.4. The reproducibility of the experimental density data is determined to be ± 0.005 g/cc.

For liquids, density decreases with increasing temperature, since the liquid molecules move away from each other. As seen in Table 3.4 and Figure 3.4 the reduction in density with temperature follows a similar trend for different samples tested. The presented data also indicate that as GTL proportion in the blend increases, the density gradually decreases because of the fact that lighter hydrocarbons are added to the heavier ANSC oil. Since density decreases with increasing GTL proportion, the pressure or horsepower requirements for commingled flow should fall within the feasible limits. Also, the effect of temperature on density appears to be fairly small, indicating that density is not likely to be a critical factor in low temperature pumpability of the material. The density data thus provide crucial information

for determining the hydraulic parameters (Akwukwaegbu, 2001) that are required in studying the transportation of GTL products through the Trans Alaska Pipeline System (TAPS).

3.7.2 Viscosity Measurements

All viscosity measurements were carried out in an LVDV-II+ cone and plate Brookfield Viscometer. The electronic cone spindle CPE-40 was used. The cone angle and radius were 0.8° and 2.4 cm respectively. The test temperatures ranged from 20 to 60°C. The accuracy of temperature measurement was within $\pm$ 0.1 %. Similar to the density measurements, test samples were 100 % ANSC oil, ANSC: GTL blends in the volumetric proportions of 3:1, 1:1, and 1:3, and 100 % GTL. Sample requirements were as low as 0.5 cc. The measured viscosity data are furnished in Table 3.5 and graphically represented in Figure 3.5. The reproducibility of the experimental viscosity data is determined to be $\pm$ 0.05 mPa.s.

As seen from the data presented in Table 3.5 and Figure 3.5, viscosity decreases as temperature increases for all the samples. For the 100 % ANSC oil, the effect of temperature on viscosity is much more pronounced than in other test samples. At the lowest temperature of 20 °C, the 100 % ANSC oil viscosity is higher than the 100 % GTL by almost 13 times, whereas the difference is as low as 8 times when results at 60 °C are compared. The significantly high viscosity of 100 % ANSC oil at 20 °C does indicate an approach towards a non-Newtonian type behavior. However, the measured data clearly indicates that GTL addition to the ANSC oil significantly reduces the overall viscosity of the blend. This is attributed to the low viscosity of the 100 % GTL sample due to the absence of long chain of heavy hydrocarbon molecules unlike the ANSC oil, in which they dominate the composition and thus impart high viscosity characteristics. The measured viscosity data in conjunction with the density data will facilitate the calculations of friction factors, Reynolds numbers, and pressure drops required in comparing the calculations of batching or blending mode of transporting GTL products through TAPS (Akwukwaegbu, 2001).

In summary, it is observed that crude oil viscosity is a stronger function of temperature than light GTL viscosity. Viscosity of the light GTL being much smaller than crude oil viscosity and the effect of temperature on light GTL viscosity being small (Fig 3.5), it appears that viscosity is not likely to be a controlling factor GTL or GTL-crude blend pumpability.

3.8 CORRELATIONS OF THE MEASURED DENSITY AND VISCOSITY DATA

The measured density (ρ, in g/cc) and viscosity (μ, in mPa.s) data of ANSC, GTL, and their blends were correlated as a function of temperature (T, in °C) and ANSC oil composition by volume (x, in fraction) in the blend. The experimental data were correlated using non-linear regression technique (SigmaPlot 2000, v6.1).

The density data were correlated as shown below:

$$\rho = aT^2 + bT + c$$

$$(1)$$

Where,

$$a = a_1x^2 + b_1x + c_1 \qquad (2)$$

$$b = a_2x^2 + b_2x + c_2 \qquad (3)$$

$$c = a_3x^2 + b_3x + c_3 \qquad (4)$$

with the following values of the coefficients –

$a_1 = 1.48633e^{-5}$ $c_1 = 1.29317e^{-5}$ $b_1 = -1.83177e^{-5}$
$a_2 = -0.00146545$ $c_2 = -0.00159337$ $b_2 = 0.00188374$
$a_3 = -0.0581009$ $c_3 = 0.770151$ $b_3 = 0.207453$

The density correlation reproduced the experimental data with an average absolute deviation (AAD) of 0.3 % and the regression coefficient (R^2) of 0.998. A comparison of the correlated and measured density data is shown in Figure 3.6.

The viscosity data were correlated as shown below:

$$\mu = dx^2 + ex + f \qquad (5)$$

Where,

$$d = d_1T^2 + e_1xT + f_1 \qquad (6)$$

$$e = d_2T^2 + e_2T + f_2 \qquad (7)$$

$$f = d_3T^2 + e_3T + f_3 \qquad (8)$$

with the following values of the coefficients:

$d_1 = 0.000913208$ $f_1 = 3.23332$ $e_1 = -0.106364$

$d_2 = -0.000514859$ $f_2 = -0.213413$ $e_2 = 0.0668998$

$d_3 = 0.00020135$ $f_3 = 1.10379$ $e_3 = -0.036443$

The viscosity correlation reproduced the experimental data with an average absolute deviation (AAD) of 5.6 % and regression coefficient (R^2) of 0.997. A comparison of the correlated and measured viscosity data is given in Figure 3.7.

$$\frac{v^2}{2} + \frac{P}{\rho} + gz = C_e \qquad (4.2.11)$$

where C_e is a constant. Equation (4.2.11) is more commonly known as the Bernoulli equation of pressure in steady flow or the equation of energy for steady flow.

For flow between points 1 and 2, equation (4.2.11) is written as,

$$\left(\frac{V_2^2}{2g} + \frac{P_2}{\rho_2 g} + z_2 \right) - \left(\frac{V_1^2}{2g} + \frac{P_1}{\rho_1 g} + z_1 \right) = C_e \qquad (4.2.12)$$

In equation (4.2.12), $V^2/2g$, and $P/(\rho g)$ are the velocity and pressure heads respectively. The last term, z, is the elevation or geometric head of the fluid above an arbitrary reference plane (Kaufmann, 1963; Holland, 1973).

4.3.2.2.1 Energy Losses
Since most natural liquids are very nearly incompressible (i.e. constant density), they are not inviscid (frictionless). Internal friction (viscosity) converts part of the flow energy into other energy forms such as sound, heat etc. and it is "lost" (Kaufmann, 1963). This loss is normally considered as a "head", the friction head, h_f, and is given by the Darcy-Weisbach equation (Smith et al, 1960) as:

$$h_f = 4f \frac{LV^2}{2gD} \qquad (4.2.13)$$

Therefore, equation (4.2.12) is re-written as,

$$\left(\frac{V_1^2}{2g} + \frac{P_1}{\rho\, g} + z_1 \right) = \left(\frac{V_2^2}{2g} + \frac{P_2}{\rho g} + z_2 \right) + h_f \qquad (4.2.14)$$

For steady incompressible flow through a pipe, between points 1 and 2, with a pump at one end, equation (4.2.14) can be re-written as,

$$\left(\frac{V_2^2}{2g} + \frac{P_2}{\rho g} + z_2 \right) - \left(\frac{V_1^2}{2g} + \frac{P_1}{\rho g} + z_1 \right) = \Delta h_p - h_f \qquad (4.2.15)$$

where Δh_p, is the head imparted to the fluid by the pump (Holland, 1973).

This then implies that the total pressure drop across the streamline is given as

3.9 TEST FLUIDS FOR GEL STRENGTH MEASUREMENTS

The gel strength measurements were carried out on two different GTL samples and crude oil. The properties of the two GTL samples are presented in the following sub-sections. The crude oil composition has already been presented in Table 2.

3.9.1 GTL 1

The first set of gel strength measurements were performed using the light GTL sample (from LaPorte) mixed with varying wax fractions. The wax and the light hydrocarbon GTL sample were supplied by the University of Alaska Fairbanks (UAF) to WTCI after receiving them from US DOE. The paraffin wax (heavier alkanes) was separated from Shellwax® 200 by a modified ASTM-1160 Vacuum Distillation process to produce only a 20% overhead fraction. This wax fraction was then gravimetrically mixed with the light hydrocarbon GTL liquid in the proportions of 25% wax distillate + 75 % light GTL and 50 % wax distillate + 50 % light GTL. The compositions of the light GTL and the blends with the wax are shown in Table 6.

The purpose of mixing the wax distillate with the light GTL in different proportions was to create GTL samples of varying wax content. Since gel strength has significant dependence on wax content, it was necessary to use samples with varying wax content so that the limiting conditions for flow through TAPS can be estimated.

3.9.2 GTL 2

The second GTL sample was a Fischer-Tropsch (FT) diesel sample, also supplied by US DOE. The characteristics of this GTL are presented in Table 7. A compositional analysis of this GTL sample (FT Diesel) was also carried out at UAF. The detailed composition of GTL 2 is provided in Table 8. A plot of the PIANO (Paraffins, Iso-paraffins, Aromatics, Naphthenes, Olefins) analysis of GTL 2 is presented in Figure 8. The FT diesel was used by itself (i.e. without mixing with wax fractions) in this set of tests. This represents a scenario where clean diesel is produced and transported from the North Slope.

3.10 GTL AND TAPS CRUDE OIL BLENDS

As far as GTL 1 is concerned, twenty-four samples were, gravimetrically prepared for gel strength measurements, prepared as per the ratios listed below:

(a)   25 % wax distillate + 75 % light hydrocarbon (LH) GTL
(b)   50 % wax distillate + 50 % light hydrocarbon GTL
(i)   sample (a) + crude oil (1:4)
(ii)   sample (a) + crude oil (1:3)
(iii)   sample (b) + crude oil (1:4)
(iv)   sample (b) + crude oil (1:3)
(v)   LH sample + crude oil (1:4)
(vi)   LH sample + crude oil (1:3)

The gel strength measurements were carried out on twenty-four test samples for the GTL 2. The samples were gravimetrically prepared, eight each at the three ratios listed below:

(a)   100% Light Hydrocarbon GTL
(b)   25% Light Hydrocarbon GTL + 75% Crude Oil
(c)   20% Light Hydrocarbon GTL + 80% Crude Oil

In the GTL:crude oil blend ratios, smaller proportions of GTL were used because it was assumed that GTL production in the North Slope will be far less than crude oil production in the foreseeable future. Thus, blends containing lower proportions of GTL are likely to flow through TAPS.

3.11 COMPOSITIONAL ANALYSIS OF GTL 1 AND CRUDE OIL BLENDS

The quantitative compositions of various GTL and crude oil blends and pure crude oil were determined by WTCI. Prior to the GC analysis of blends, the apparatus is subjected to an extensive quality control check using normal alkane standard, aromatic standard, reference gas oil, and two crude oil standards. The gas chromatographs on the GTL and crude oil blends were quantified by employing iso-octane as an internal standard. The Standard Laboratory Procedure (2001) of WTCI explains the GC analysis procedure in further details.

The compositions of GTL and crude oil blends (samples v and vi, Section 3.10) were not measured, these values were calculated from the individual GTL and crude oil compositions. All the compositional data are presented in Table 3.9. In order to assess the internal consistency of the measured compositional data on the blends, the TAPS crude oil composition was back calculated using a simple component mass balance approach as shown below (example for C30+):

$$C_{30+[50:50]GTL} * M_{[50:50]GTL} + C_{30+CRUDEOIL} * M_{CRUDEOIL} = C_{30+BLEND} * M_{BLEND}$$

or

$$C_{30+[25:75]GTL} * M_{[25:75]GTL} + C_{30+CRUDEOIL} * M_{CRUDEOIL} = C_{30+BLEND} * M_{BLEND}$$

Where,

$C_{30+[50:50]GTL}$ and $C_{30+[25:75]GTL}$ is the weight % of C30+ in 50:50 (wax:GTL) and 25:75 (wax:GTL) respectively

$C_{30+CRUDEOIL}$ and $C_{30+BLEND}$ is the weight % of C30+ in the crude oil and the blend respectively

$M_{[50:50]GTL}$ and $M_{[25:75]GTL}$ is the mass of 50:50 (wax:GTL) and 25:75 (wax:GTL) respectively

$M_{CRUDEOIL}$ and M_{BLEND} is the mass of crude oil and the blend respectively

The calculated crude oil composition and the ones measured on the crude oil were compared as shown in Figure 3.9. As seen in Figure 3.9, all the data lies neatly on a 45° line, which clearly indicates that the measured compositional data on GTL and crude oil blends is reliable, and internally consistent.

3.12 COMPOSITIONAL ANALYSIS OF GTL 2 AND CRUDE OIL BLENDS

Compositional data for the GTL 2 and TAPS crude oil blends were not measured. However, the compositions of the GTL 2 were measured using gas chromatography at UAF. For this purpose, a known calibration sample was analyzed over a wide range of solvent to sample concentrations. Based on this, a relationship between the molar composition, retention time, and the areas under the chromatograph peaks was developed. The known calibration sample is primarily normal paraffins. Although, this may not be a good relationship for other substances, it does not pose a problem for a substance like FT diesel as it is mainly composed of alkanes or saturates (Table 3.7 and 3.8). The developed correlation was applied to the area percents from a sample of GTL 2, which was run through the GCMS, to determine its composition.

The individually measured compositions of the GTL 2 and the TAPS crude oil were numerically recombined to determine the blend compositions. The calculated blend compositions are presented in Table 3.10.

3.13 TEST TEMPERATURES FOR GEL STRENGTH MEASUREMENTS

The GTL 1 and TAPS crude oil blend gel strength measurements were carried out at temperatures ranging from 60 to 0°F, whereas for the GTL 2 and TAPS crude oil blends the gel strength measurements were performed at temperatures in the range of 27 to –20°F.

These test temperatures were selected on the basis of the cold ramping of the GTL and crude oil blends from 90 to –20°F over a twenty one day period. The temperature decay curve used for the sample preparation and test temperature selection was based on the Trans Alaska Pipeline cold restart data supplied by the Alyeska Pipeline Service Company. The temperature ramp is presented in Figure 3.10, along with the selected test temperatures. The temperature ramp simulates cooling of the fluids inside TAPS under winter shutdown conditions. Thus, by choosing test temperatures from the TAPS cooling ramp, one would be able to estimate the feasibility of cold restart for a given GTL product or its blend.

3.14 GEL STRENGTH MEASUREMENT PROCEDURE

The gel strengths of various GTL crude oil blends were determined by the rotating vane method. The tests for determining the yield stress, or yield stress value, of the cooled crude oil blends, were performed following Westport's Standard Laboratory Procedure 307 (WTC, July 2000). This method is based on Brookfield rotary viscometers and vane spindles, which extend horizontally through a sample, minimizing the impact of slippage at the spindle wall. This method determines the minimum amount of torque necessary to initiate oil movement at low shear, and subsequent gel breakdown after initiation of flow.

The method basically consists of determining the yield stress value of a crude oil by measuring the torque on a spindle, using a Brookfield viscometer, rotating at 0.01 rpm in the material. The spindle to be used consists of four rectangular vanes dimensioned (0.75"w x 2.25"h) and oriented at 90 degree increments around the central axis. The sample cup is dimensioned (1.5" id x 4.0"h). Vertical orientation of vanes within the sample cup is dimensioned (1.00" from top and 0.75" from bottom).

The crude oil blends were initially heated to 150°F to destroy all temperature and shear histories and then cooled to 100°F at which stress value they were loaded into the vane closed-cup apparatus. The closed-cup apparatus holds the vanes rigidly during cooling and aging and prevents loss of light ends through evaporation. After loading into the cup apparatus the samples were cooled in an environmental chamber at a controlled rate to below 0°F. The cooling rate mimics the expected rate of cooling of the Trans-Alaska pipeline oil in the case of shut-in.

Samples were withdrawn from the environmental chamber at selected test temperatures and transferred to a refrigerated circulator that maintains the sample at test temperature. The spindle was attached to the Brookfield viscometer (LV, RV or HB) before the spindle clamping mechanism was released. The clamping mechanism was released and the viscometer was started at 0.01 rpm and torque as a function of time was measured, until a maximum reading was obtained. The maximum torque (dyne-cm) obtained is divided by a vane parameter constant K to obtain the yield stress (dynes/sq.cm). The constant K is calculated based on the dimensions of the vanes. (K=36.19 cm^3, for a Vane with D=0.75 inch (1.905 cm) and H=2.25 inch (5.715 cm).

3.15 EXPERIMENTAL RESULTS AND DISCUSSIONS

3.15.1 Gel Strength Measurements on GTL 1 and TAPS Crude Oil

The results on the gel strength measurements for the GTL 1 and TAPS crude oil mixtures are presented in Table 3.11 and Figure 3.11, respectively.

Initial test temperatures were set at 9, 0 and –20°F. However, tests at 9°F indicated gel strength beyond measurable limits of the viscometer for samples (i) to (iv) and relatively high strengths for samples (v) and (vi). Because of this the cold ramp was continued and testing resumed at 0°F for samples (v) and (vi). Similarly, due to the high yield stress values encountered, no tests were conducted at –20°F and the remaining samples were tested at higher temperatures in order to determine the gelation onset and build-up.

Thus, from the very high gel strengths of samples (i) to (iv) at 9°F, it appears that GTL products with moderate to high wax content are not likely to be acceptable for transporting via TAPS from the cold restart point of view. Even the pure light GTL-crude samples (v) and (vi), which did not have any added wax fraction, exhibited very high gel strength at 0°F. Cold restart may be a problem for even the light GTL.

All remaining samples were re-heated to 150°F and then cold ramped to a 21-day cycle. The high wax content/GTL samples (iii, iv) were tested at 60°F resulting in low yield values.

The tests at 40°F were performed on samples (i) to (iv). Samples (ii) and (iii) resulted in maximum torque readings beyond the limits of the initially selected viscometers. Therefore, repeat tests were performed for samples (ii) and (iii) using viscometers with higher spring ratings, which permitted the measurement of yield stress.

All the tests at 20°F on samples (i), (ii), (iv), (v) and (vi), were carried out using the strongest viscometer (HB). Due to the higher wax content, the test on sample (iv) was beyond the strongest viscometer limit, and yield stress is reported as > 1589 dyne/cm^2.

The gel strength measurements on sample (vi) at 9°F were repeated. The repeat measurements indicated some variability, as far as the results are concerned (229 vs. 438 dyne/cm^2). This variation is not attributed to procedural variations. Moreover, due to the lack of sample quantity a third repeat test could not be performed. However, the value of 438 dyne/cm2 is considered as more reliable as it falls within the data trend.

3.15.2 Gel Strength Measurements on GTL 2 and TAPS Crude Oil

The results on the gel strength measurements for the GTL 2 and TAPS crude oil mixtures are presented in Table 3.12 and Figure 3.12, respectively.

Initially, the planned test temperatures were set at 20°F, 0°F and –20°F. However, the simple spot test of the pure GTL sample (a) indicated possible gel strength onset at earlier temperatures. Therefore, tests were performed on the 100% GTL at 27°F and a yield stress of 171 dynes/sq.cm was obtained. At a temperature of 20°F the pure GTL sample had reached its pour point value. Therefore, no gel strength measurements could be carried out at the other temperatures of 20, 0, and –20°F as these were beyond the measurable limits of the strongest viscometers. Thus, these values are reported as >1589 dyne/sq.cm.

The gel strength measurements for sample (b) and (c) at 20°F resulted in averaged yield stress values of 2.91 dynes/sq.cm and 1.13 dynes/sq.cm respectively. At a temperature of 0°F sample (b) produced an average yield stress value of 694 dynes/sq.cm, whereas sample (c) produced an average yield stress value of 389 dynes/sq.cm.

All tests performed at a temperature of –20°F on sample (b) and (c) produced yield stress values beyond the measurable limits of the Vane test equipment, or values greater than 1589 dynes/sq.cm.

These results indicate that gel strength of FT diesel can increase abruptly once the temperature falls below a threshold. The threshold temperature can be as high as 20°F. From the pipeline cold restart point of view, this poses a serious problem. Since the pure GTL sample gels at a higher temperature than GTL-crude oil blends, batching of GTL could be more troublesome than commingled flow. Although these results are preliminary, they clearly indicate the need

for further studies to identify exactly what amounts and molecular weights of paraffins in GTL will be acceptable for transportation through TAPS.

3.16 COMPARISON OF GEL STRENGTH MEASUREMENTS ON GTL 1 AND GTL 2 AND TAPS CRUDE OIL BLENDS

In order to evaluate the effect of GTL type or its characteristics on gel strength measurements of GTL and crude oil blends, a comparative study of the two GTL's, i.e., GTL 1 and GTL 2 was carried out. For the sake of fare comparison, gel strengths measured for the blends having the ratios of 1:3 (GTL:crude oil) and 1:4 (GTL:crude oil) or 25% GTL and 75% crude oil and 20% GTL and 80% crude oil, were compared. These results are shown Figure 3.13 and Figure 3.14 respectively. This comparison indicated that the trend of gel strengths were similar for samples (b) and (vi) and (c) and (v) having 1:3 and 1:4 GTL to crude oil ratios respectively.

3.17 CONCLUSIONS

3.17.1 Density and Viscosity Measurements

Based on the experimental study carried out at UAF on density and viscosity measurements of GTL, crude oil and their blends, the following main conclusions are drawn.

- Standard laboratory testing procedures are developed and used to determine the density and viscosity data of Alaskan North Slope Crude (ANSC) oil, Gas-to-Liquids (GTL), and their blends, in the temperature range of ambient to 60 °C at ambient pressure.

- A Quality Assurance Project Plan (QAPjP) has been developed.

- Appropriate calibration procedures are followed for calibrating the Anton-Paar densitometer and the cone and plate Brookfield viscometer used for measuring the density and viscosity respectively.

- The reproducibility of the measured density and viscosity data was found to be ± 0.005 g/cc and ± 0.05 mPa.s respectively.

- It was found that GTL and crude oil readily developed miscibility when mixed at ambient temperature and pressure conditions.

- The trend of both density as well as viscosity reduction as a function of increasing temperature is clearly evident for all the tested samples.

- The dilution of crude oil by GTL causes a major reduction in both density as well as viscosity.

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- The measured density and viscosity data have also been correlated using non-linear regression techniques. It is demonstrated that the developed correlations are capable of reproducing the measured data with reasonable accuracy and reliability.

- Using the measured data and the developed correlations, the hydraulics of transporting GTL and crude oil fluids either commingled or batches through TAPS can be evaluated.

3.17.2 Gel Strength Measurements

Based on the test results at WTCI on gel strength measurements and compositional analysis of various GTL and crude oil blends, the following main conclusions are drawn.

- The trend of increasing yield stress with increasing wax content was clearly observed.

- Gel strength tests at $-20°F$ for the GTL 1 and TAPS crude oil blends had to be abandoned owing to very high yield stress (beyond measurable limits of the apparatus) already encountered at temperatures higher than $-20°F$.

- The results indicate that light samples had measurable gel strength whereas significant gel strength problems were observed for heavier GTL and crude oil blends.

- The formation of weak gel structures at higher temperatures (sample vi, $9°F$) posed some repeatability problems. The lack of additional samples precluded the possibility of further repetition of the test.

- The measured compositional data on GTL 1 and crude oil blends was found to be accurate and reliable.

- As far as the 100% GTL 2 sample is concerned, it produces high gel strength, but when it is blended with TAPS crude oil a significant reduction is observed.

- It is also evident that the decrease in GTL 2 in the blend ratio also contributes to lower yield stress values.

- No gel strength measurements could be reported at temperatures of $-20°F$ for the GTL 2 and TAPS crude oil blends due to very high yield stress (beyond measurable limits of the apparatus).

- In general, the gel strength behavior observed with the GTL 2 and TAPS crude oil blends can be attributed to the chemical composition of the GTL 2, which indicates a highly paraffinic characteristic.

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- The developed methodology of GCMS calibration at UAF was found to be accurate and reliable for the determination of FT diesel (GTL 2) compositions.

- It can be concluded that the ratio blending may be effective in lowering the ultimate yield stress values of GTL/crude oil blends. However, it will be worthwhile to determine if the effect of ratio blending is reproducible with varying crude oil composition.

- A similarity in the gel strength trend was observed when the results for GTL 1 and GTL 2 and TAPS crude oil blends were compared.

- This study indicates that even light GTL (LaPorte type) or FT diesel can pose problems for transportation through TAPS from the cold restart point of view. Rigorous studies are needed to identify the upper limit on the quantity and nature of paraffins in GTL that can be accepted for TAPS flow. Since high molecular weight paraffins are clearly not acceptable, it may be necessary to include a hydrocracking unit in the GTL plant considerations for the North Slope.

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TABLE 3.1
Composition of the Gas-To-Liquids (GTL) sample used in density and viscosity measurements

Component	Mole %	MW* g/mole	Density* g/cc @ 15.6 °C
H_2S	0.00	34.1	
CO_2	0.00	44.0	
Methane	0.00	16.0	
Ethane	0.00	30.1	
Propane	0.95	44.1	
i-Butane	0.00	58.1	
n-Butane	6.19	58.1	
neo-Pentane	0.00	72.2	
i-Pentane	0.00	72.2	
n-Pentane	10.80	72.2	
C6	12.41	84.0	0.6900
C7	11.48	96.0	0.7270
C8	10.49	107.0	0.7490
C9	8.87	121.0	0.7680
C10	7.48	134.0	0.7820
C11	6.16	147.0	0.7930
C12	5.62	161.0	0.8040
C13	4.24	175.0	0.8150
C14	3.36	190.0	0.8260
C15	2.65	206.0	0.8360
C16	2.11	222.0	0.8430
C17	1.68	237.0	0.8510
C18	1.32	251.0	0.8560
C19	1.05	263.0	0.8610
C20	0.87	275.0	0.8660
C21	0.60	291.0	0.8710
C22	0.46	300.0	0.8760
C23	0.33	312.0	0.8810
C24	0.25	324.0	0.8850
C25	0.18	337.0	0.8880
C25+	0.45	367.1	0.8979
Total	100.00		

* Molecular weights and densities are from the generalized values of Katz and Firoozabadi (1978) and Whitson (1984).

TABLE 3.2

Composition of the crude oil sample used in density, viscosity, and gel strength measurements

Sample Components	Composition Wt%	Mol. Wt. g/mole	Density @ 15.6 °C g/cc
C2	0.009		
C3	0.167		
IC4	0.259		
NC4	0.954		
IC5	0.630		
NC5	0.967		
C6	1.836		
BENZENE	0.340		
C7	3.358		
TOLUENE	0.792		
C8	4.078	100.0	0.7576
C9	4.115	113.0	0.7759
C10	3.715	126.0	0.7897
C11	3.174	138.0	0.8064
C12	2.989	153.0	0.8287
C13	3.308	162.0	0.8335
C14	3.160	174.0	0.8490
C15	3.108	179.0	0.8609
C16	2.757	192.0	0.8672
C17	2.636	208.0	0.8710
C18	2.590	216.0	0.8780
C19	2.543	234.0	0.8846
C20	2.296	241.0	0.8981
C21	2.134	252.0	0.9015
C22	2.037	263.0	0.9062
C23	1.898	285.0	0.9085
C24	1.781	304.0	0.9108
C25	1.669	307.0	0.9138
C26	1.624	555.0 (C_{25+})	0.9851 (C_{25+})
C27	1.557		
C28	1.532		
C29	1.482		
C30+	34.506		
Total	100.000		

TABLE 3.3

Density of water.

Temp. °C	Density, g/ml	Temp. °C	Density, g/ml	Temp. °C	Density, g/ml
0.0	0.999840	21.0	0.997991	40.0	0.992212
3.0	0.999964	22.0	0.997769	45.0	0.990208
4.0	0.999972	23.0	0.997537	50.0	0.988030
5.0	0.999964	24.0	0.997295	55.0	0.985688
10.0	0.999699	25.0	0.997043	60.0	0.983191
15.0	0.999099	26.0	0.996782	65.0	0.980546
15.56	0.999012	27.0	0.996511	70.0	0.977759
16.0	0.998943	28.0	0.996231	75.0	0.974837
17.0	0.998774	29.0	0.995943	80.0	0.971785
18.0	0.998595	30.0	0.995645	85.0	0.968606
19.0	0.998404	35.0	0.994029	90.0	0.965305
20.0	0.998203	37.78	0.993042	100.0	0.958345

TABLE 3.4
Experimental density data* of tested crude oil, GTL, and their blends

Temperature °C	Crude:GTL (1:0)	Crude:GTL (3:1)	Crude:GTL (1:1)	Crude:GTL (1:3)	Crude: GTL (0:1)
			Density g/cc		
23.8	-	-	-	-	0.7377
24.0	0.8987	-	-	-	-
24.1	-	0.8703	-	-	-
24.2	-	-	0.8385	-	-
24.4	-	-	-	0.7986	-
35.0	0.8911	0.8634	0.8315	0.7916	0.7282
40.0	0.8893	0.8618	0.8294	0.7891	0.7247
45.0	0.8880	0.8600	0.8282	0.7873	0.7224
50.0	0.8868	0.8583	0.8268	0.7853	0.7210
60.0	0.8844	0.8565	0.8257	0.7814	0.7197

*   All measurements are at atmospheric pressure.

TABLE 3.5
Experimental viscosity data* of tested crude oil, GTL, and their blends

Temperature °C	ANSC:GTL (1:0)	ANSC:GTL (3:1)	ANSC:GTL (1:1)	ANSC:GTL (1:3)	ANSC:GTL (0:1)
			Viscosity mPa.s		
20	17.30	6.80	4.10	2.30	1.30
30	10.80	5.30	3.00	1.90	1.10
40	7.70	4.30	2.30	1.60	0.96
50	6.40	3.70	1.90	1.40	0.81
60	5.50	3.40	1.70	1.30	0.68

*   All measurements are at atmospheric pressure.

TABLE 3.6
Compositional data of GTL 1 sample by gas chromatography

Sample Components	Gas to liquids product (GTL) Wt%	25% Wax / 75% GTL Wt%	50% Wax/ 50% GTL Wt%
C2	0.002	0.003	0.003
C3	0.145	0.107	0.066
IC4	0.003	0.011	0.007
NC4	1.215	0.904	0.245
IC5	0.146	0.110	0.069
NC5	2.972	2.212	1.367
C6	7.826	5.800	3.901
BENZENE	0.003	0.002	0.001
C7	8.889	6.633	4.167
TOLUENE	0.029	0.022	0.016
C8	8.764	6.570	4.147
C9	8.539	6.384	4.071
C10	7.940	6.230	3.972
C11	7.290	5.607	3.989
C12	6.446	5.241	3.827
C13	5.748	4.908	3.854
C14	5.065	4.610	3.942
C15	4.414	4.367	4.101
C16	3.811	4.189	4.348
C17	3.273	4.096	4.699
C18	2.799	4.071	5.119
C19	2.389	4.070	5.522
C20	2.026	4.001	5.753
C21	1.705	3.795	5.674
C22	1.421	3.422	5.236
C23	1.165	2.914	4.511
C24	0.939	2.353	3.650
C25	0.747	1.816	2.804
C26	0.607	1.369	2.086
C27	0.583	1.094	1.584
C28	0.433	0.764	1.104
C29	0.247	0.458	0.710
C30+	2.418	1.866	5.457
Total	100.000	100.000	100.000

TABLE 3.7

Characteristics of the GTL 2 sample.

Analysis	Values
Density, kg/L @ 15 ^{c}o	0.7845
API Gravity @ 60 ^{o}F	54
Cetane No.	73.7
Sulfur content (% mass)	20 PPB
Aromatic (% v/v)	0.1
Saturates (% v/v)	99.8
Olefins (% v/v)	0.1
Flash Point (^{o}C)	72
Cloud Point (^{o}C)	3
Water & Sediment (%)	<0.02
Carbon Residue (% mass)	0.02
Ash (% mass)	<0.001
Viscosity (cSt @ 40 ^{o}C)	3.57
Corrosion	1A
Pour Point (^{o}C)	0
Carbon/Hydrogen (% mass)	
Carbon	84.91
Hydrogen	14.94
Nitrogen	0.57
Residual	-1.09
Oxygen (by difference)	Negligible

TABLE 3.8

Compositional data of GTL 2 sample by gas chromatography

Carbon no.	n-Paraffin mole %	i-Paraffin mole %	Aromatic mole %	Napthene mole %	Olefin mole %
9	1.124	0.000	0.000	0.000	0.000
10	1.630	0.000	0.000	0.000	0.000
11	0.702	0.000	0.000	0.000	0.000
12	2.492	1.030	0.000	0.000	0.000
13	4.851	6.114	0.000	0.000	0.000
14	8.429	6.158	0.000	0.000	0.000
15	7.469	4.887	0.000	0.000	0.000
16	6.857	3.769	0.000	0.000	0.000
17	5.721	4.172	0.000	0.000	0.000
18	4.691	7.587	0.000	0.000	0.000
19	3.440	5.631	0.000	0.000	0.000
20	2.559	3.274	0.000	0.000	0.000
21	1.801	2.913	0.000	0.000	0.000
22	0.955	1.292	0.000	0.000	0.000
23	0.175	0.280	0.000	0.000	0.000
Total	52.895	47.105	0.000	0.000	0.000

TABLE 3.9
Compositional data for GTL 1 and crude oil blends

Sample Components	25:75 Wax/GTL 80g @ 320g oil Wt%	25:75 Wax/GTL 100g @ 300g oil Wt%	50:50 Wax/GTL 80g @ 320g oil Wt%	50:50 Wax/GTL 100g @ 300g oil Wt%	80g GTL @ 320g oil* (1:4) Wt%	100g GTL @ 300g oil* (1:3) Wt%
C2	0.000	0.001	0.002	0.001	0.007	0.007
C3	0.033	0.050	0.088	0.069	0.162	0.161
IC4	0.128	0.149	0.210	0.160	0.207	0.195
NC4	0.734	0.852	0.951	0.797	1.006	1.019
IC5	0.502	0.508	0.568	0.484	0.533	0.509
NC5	1.098	1.195	1.074	1.015	1.368	1.468
C6	2.758	3.050	2.443	2.463	3.034	3.333
BENZENE	0.270	0.255	0.271	0.244	0.272	0.255
C7	3.904	4.060	3.509	3.476	4.465	4.741
TOLUENE	0.623	0.588	0.621	0.565	0.639	0.601
C8	4.572	4.662	4.111	4.092	5.015	5.249
C9	4.506	4.579	4.042	4.048	5.000	5.221
C10	4.121	4.202	3.696	3.737	4.560	4.771
C11	3.663	3.760	3.291	3.367	3.997	4.203
C12	3.403	3.490	3.112	3.208	3.681	3.853
C13	3.584	3.640	3.360	3.413	3.796	3.918
C14	3.421	3.492	3.293	3.367	3.541	3.636
C15	3.312	3.359	3.266	3.342	3.369	3.434
C16	3.042	3.105	3.083	3.194	2.968	3.021
C17	2.914	2.985	3.047	3.184	2.763	2.795
C18	2.919	2.991	3.145	3.292	2.632	2.642
C19	2.847	2.923	3.154	3.329	2.512	2.505
C20	2.653	2.747	3.050	3.230	2.242	2.229
C21	2.493	2.571	2.876	3.079	2.048	2.027
C22	2.324	2.409	2.711	2.894	1.913	1.883
C23	2.122	2.169	2.456	2.600	1.752	1.715
C24	1.909	1.948	2.180	2.286	1.613	1.571
C25	1.723	1.744	1.929	1.984	1.484	1.438
C26	1.572	1.592	1.718	1.757	1.421	1.370
C27	1.470	1.478	1.563	1.570	1.362	1.314
C28	1.375	1.378	1.437	1.422	1.313	1.258
C29	1.249	1.274	1.301	1.270	1.235	1.173
C30+	28.757	26.796	28.444	27.061	28.088	26.484
Total	100.000	100.000	100.000	100.000	100.000	100.000

* Values are calculated based on the measured GTL and TAPS crude oil composition.

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TABLE 3.10

Compositional data for GTL 2 and crude oil blends

Sample Components	100g GTL @ 300g oil* (25% GTL+75% crude oil) Wt%	80g GTL @ 320g oil* (20% GTL+80% crude oil) Wt%
C2	0.006	0.007
C3	0.125	0.133
IC4	0.194	0.207
NC4	0.716	0.763
IC5	0.472	0.504
NC5	0.725	0.773
C6	1.377	1.468
BENZENE	0.255	0.272
C7	2.519	2.687
TOLUENE	0.594	0.634
C8	3.058	3.262
C9	3.244	3.418
C10	3.040	3.175
C11	2.501	2.635
C12	2.898	2.916
C13	4.692	4.415
C14	5.535	5.060
C15	5.201	4.782
C16	4.699	4.311
C17	4.578	4.189
C18	5.359	4.805
C19	4.571	4.165
C20	3.524	3.279
C21	3.129	2.930
C22	2.291	2.240
C23	1.585	1.648
C24	1.336	1.425
C25	1.251	1.335
C26	1.218	1.299
C27	1.168	1.246
C28	1.149	1.226
C29	1.112	1.186
C30+	25.879	27.605
Total	100.000	100.000

* Values are calculated based on the measured GTL and TAPS crude oil composition.

TABLE 3.11
Yield stress data of GTL 1 and crude oil blends

Test sample (mixture reference)	Test number	Brookfield Viscometer Model	Temperature (°F)	Yield Point Maximum torque (dyne-cm)	Yield stress (dyne/cm²)
(i)	14	RV	40	747	20.7
(i)	21	HB	20	26736	739
(i)	7	HB	9	>57496	>1589
(ii)	13	LV	40	>674	>18.6
(ii)	18	RV	40	2307	63.7
(ii)	22	HB	20	50079	1384
(ii)	6	HB	9	>57496	>1589
(iii)	12	LV	60	2.69	0.07
(iii)	15	RV	40	>7187	>199
(iii)	16	HB	40	19491	539
(iii)	5	HB	9	>57496	>1589
(iv)	11	HB	60	0.0	0
(iv)	17	HB	40	43122	1192
(iv)	23	HB	20	>57496	>1589
(iv)	4	HB	9	>57496	>1589
(v)	20	HB	20	2587	71
(v)	1	RV	9	>7187	>199
(v)	2	HB	9	13167	364
(v)	10	HB	0	24206	669
(vi)	19	HB	20	3335	92
(vi)	3	HB	9	8279	229
(vi)	8	HB	9	15869	438
(vi)	9	HB	0	22366	618

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TABLE 3.12
Yield stress data of GTL 2 and crude oil blends

Test sample (mixture reference)	Brookfield Viscometer Model	Temperature (°F)	Yield Point Maximum torque (dyne-cm)	Yield stress (dyne/cm²)
(a)	RV	27	-	-
(a)	RV	27	6202	171
(a)	RV	20	>7187	>199
(a)	RV	0	>7187	-
(a)	HB	0	>57496	>1589
(a)	HB	0	>57496	>1589
(a)	HB	-20	>57496	>1589
(a)	HB	-20	>57496	>1589
(b)	RV	20	108	2.98
(b)	RV	20	93.4	2.58
(b)	RV	20	115	3.18
(b)	LV	0	>674	-
(b)	HB	0	23746	656
(b)	HB	0	26506	732
(b)	HB	-20	>57496	>1589
(b)	HB	-20	>57496	>1589
(c)	RV	20	28.7	0.79
(c)	RV	20	64.7	1.79
(c)	RV	20	28.7	0.79
(c)	LV	0	>674	-
(c)	RV	0	>7187	-
(c)	HB	0	12592	348
(c)	HB	0	15581	431
(c)	HB	-20	>57496	>1589

Circulation
of oil

Vibrating U-Tube

Sample
Injection

FIGURE 3.1 The Anton-Paar Digital Density Meter.
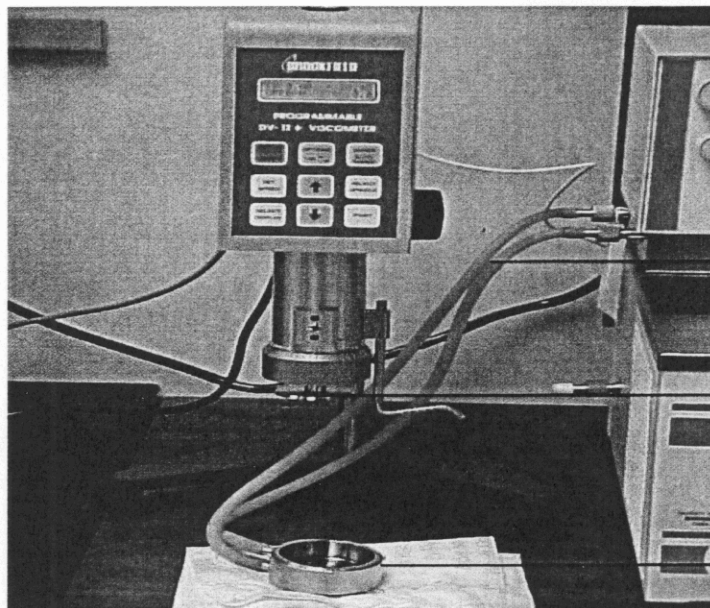
Circulation of
Bath Liquid

Cone Spindle
CPE - 40

Sample Cup
(Plate)

FIGURE 3.2 The Brookfield Cone and Plate Viscometer.

56
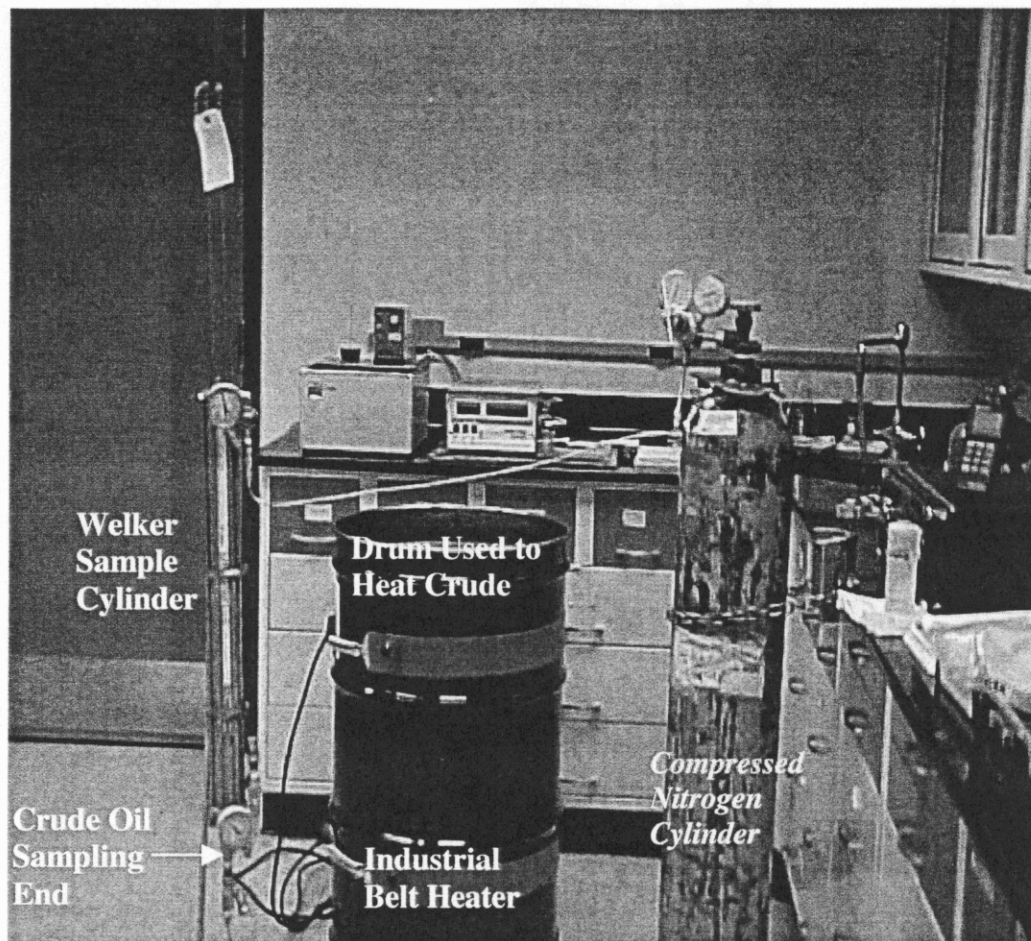
FIGURE 3.3 Crude oil re-conditioning assembly.
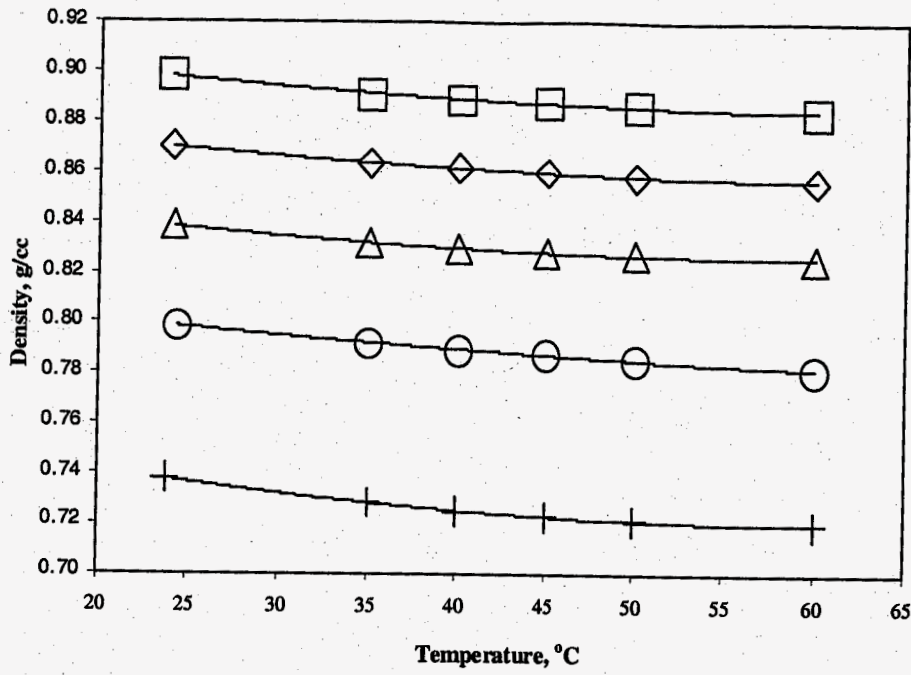
FIGURE 3.4 Experimental density data of tested crude oil, GTL, and their blends.
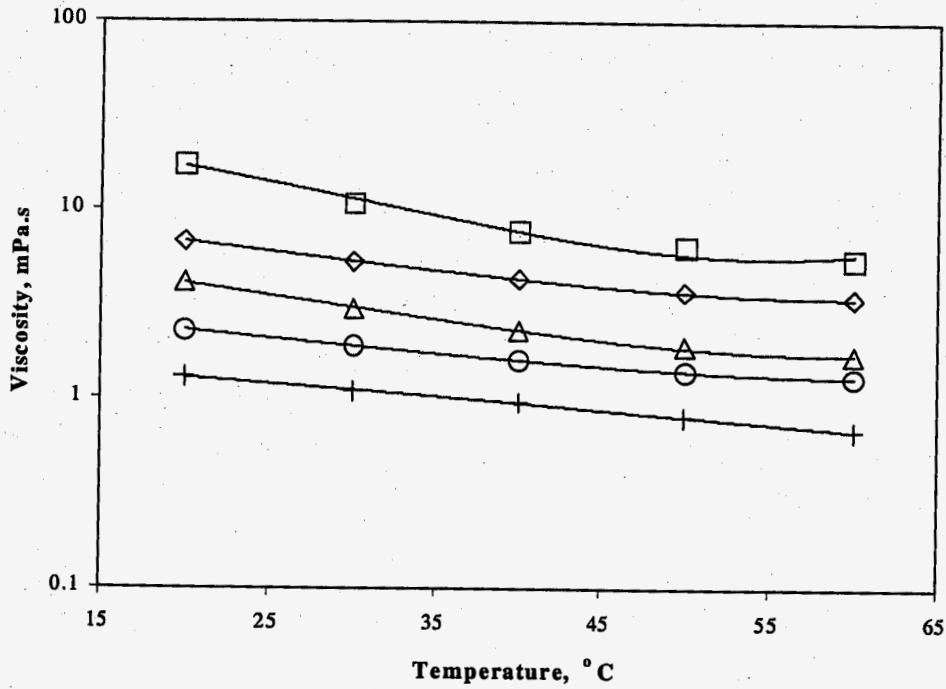
FIGURE 3.5 Experimental viscosity data of tested crude oil, GTL, and their blends.
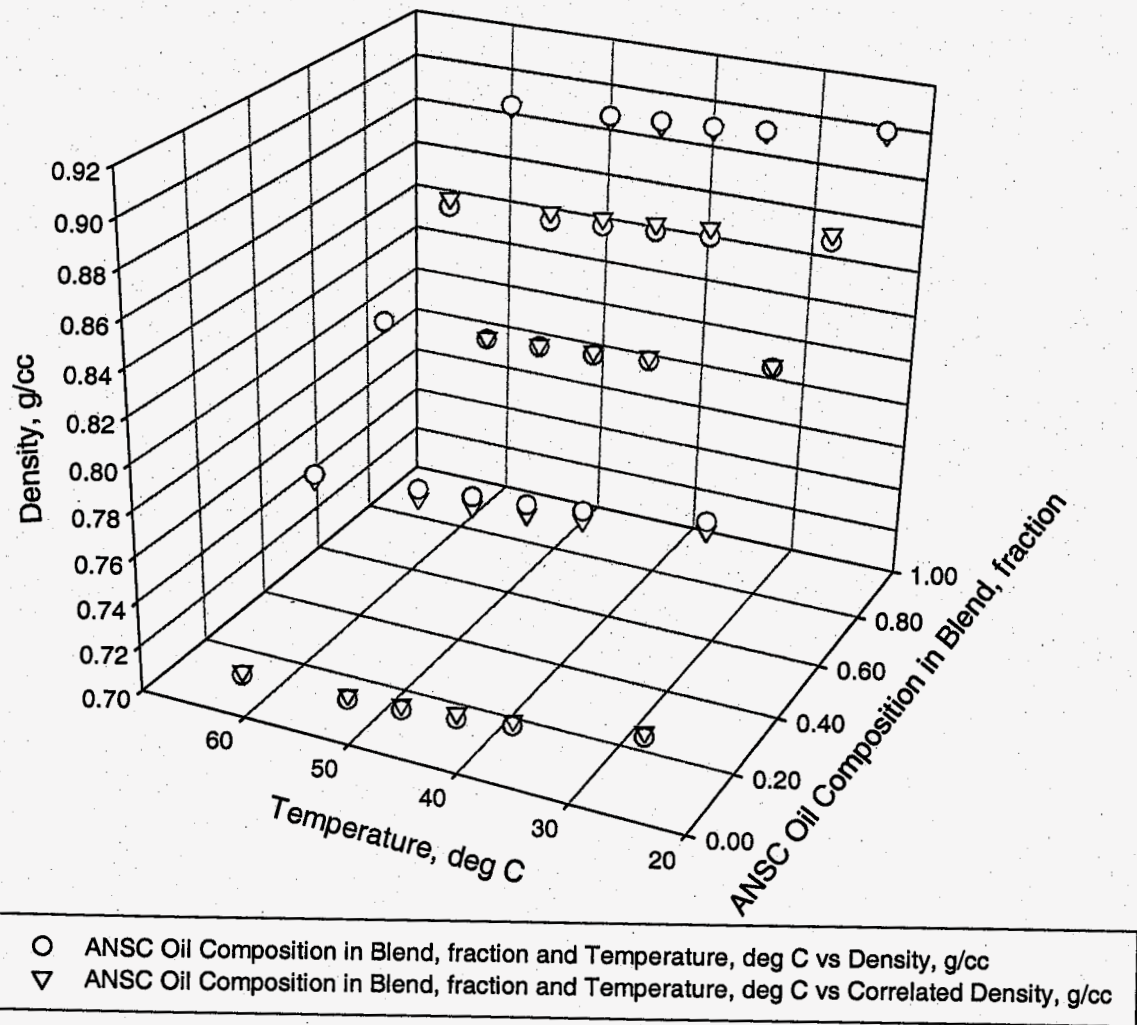
FIGURE 3.6 Comparison of correlated and measured density data for the tested crude oil (ANSC), GTL, and their blends as a function of temperature and ANSC oil composition (fraction) in the blend.
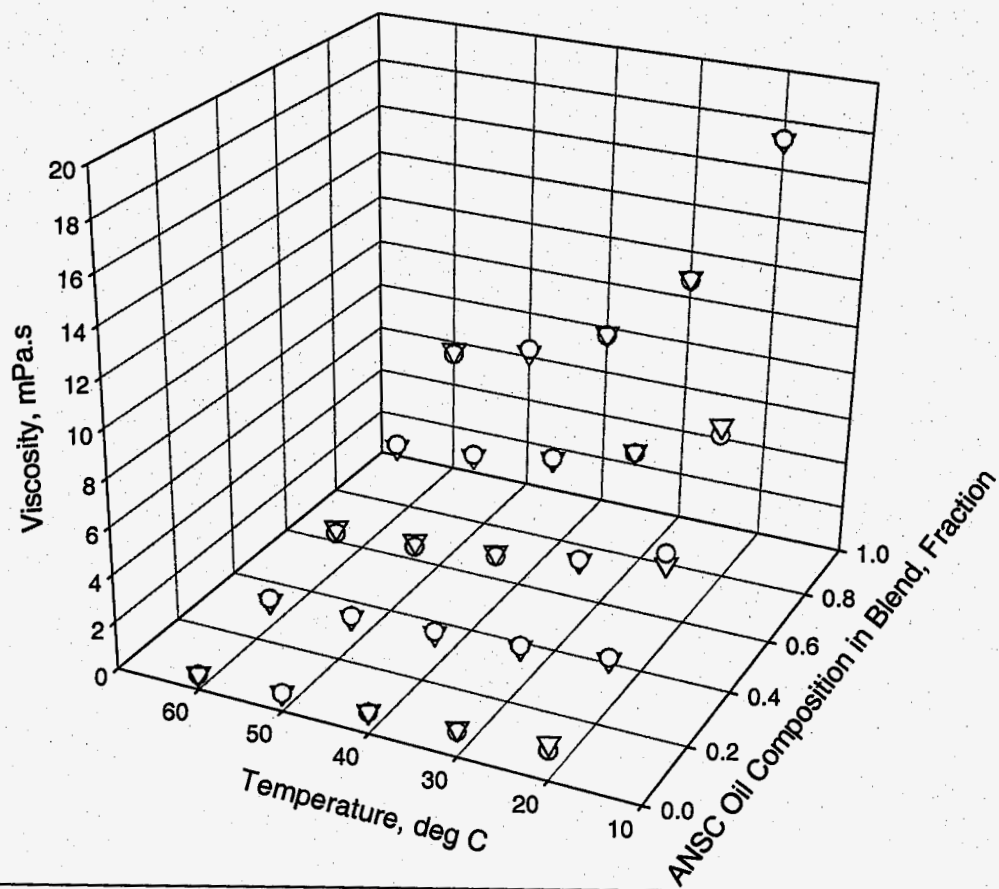
FIGURE 3.7 Comparison of correlated and measured viscosity data for the tested crude oil (ANSC), GTL, and their blends as a function of temperature and ANSC oil composition (fraction) in the blend.
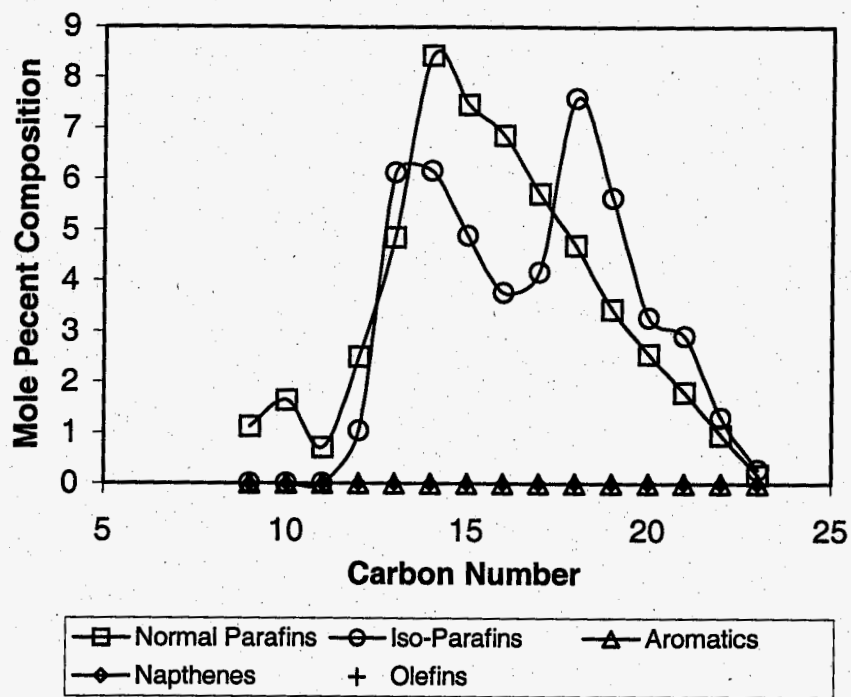
FIGURE 3.8 PIANO analysis of GTL 2 sample.
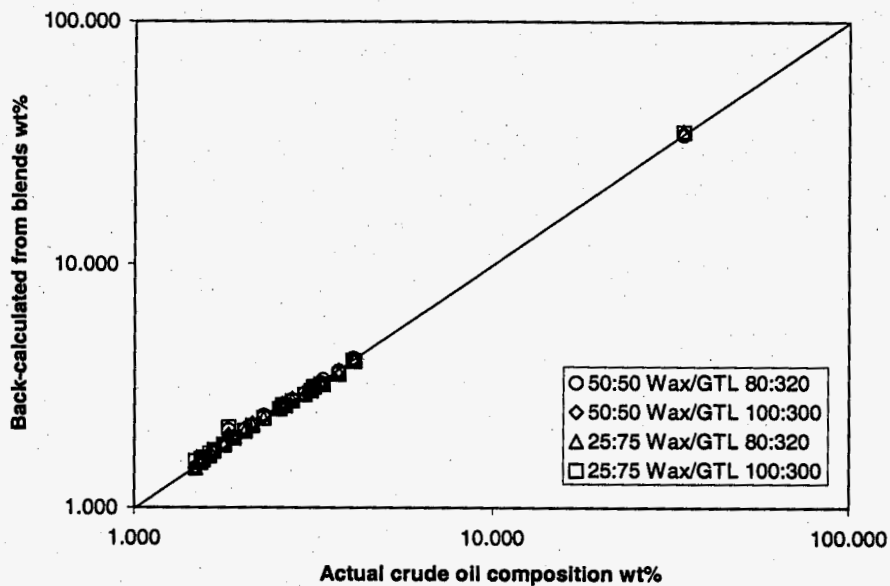
FIGURE 3.9 Comparison of back-calculated and actual TAPS crude oil composition (components having composition less than 1 wt % are not shown).
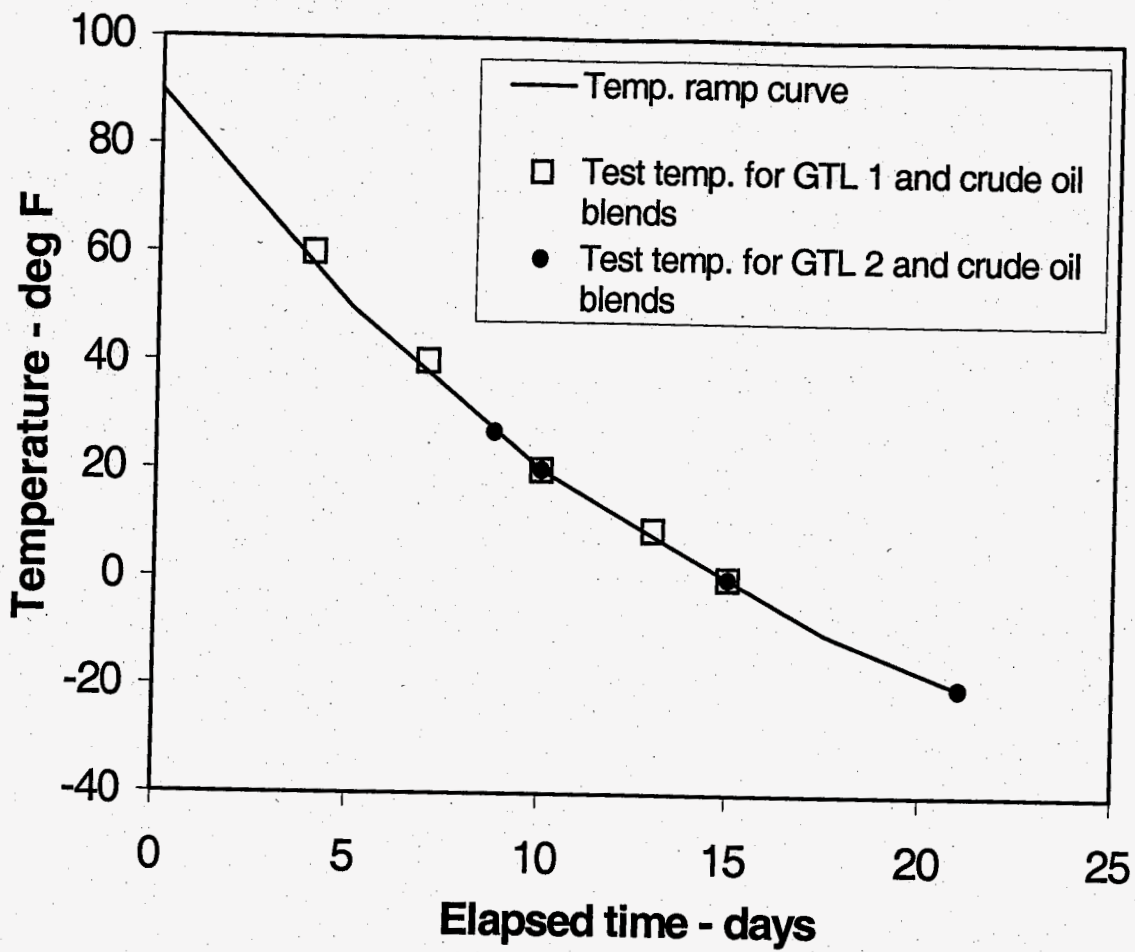
FIGURE 3.10 Cooling ramp and profile of test temperatures for gel strength measurements.

FIGURE 3.11 Yield Stress data of GTL 1 and TAPS crude oil blends as a function of test temperature
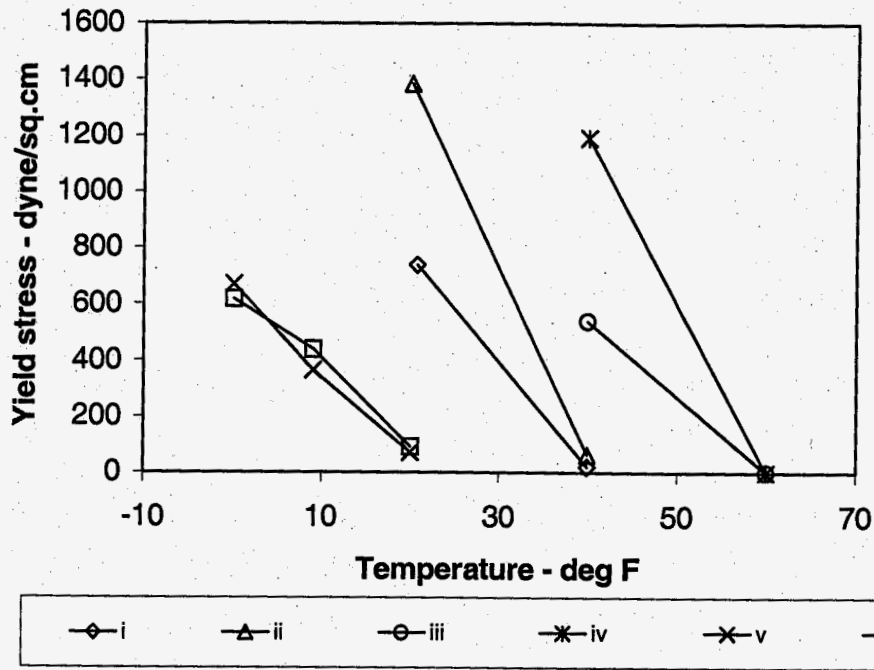
Composition of samples by %

Sample Number	ANS Crude	GTL1 Liquid	GTL1 Lt wax
1	80%	15%	5%
2	75%	18.75%	6.25%
3	80%	10%	10%
4	75%	12.5%	12.5%
5	80%	20%	
6	75%	25%	
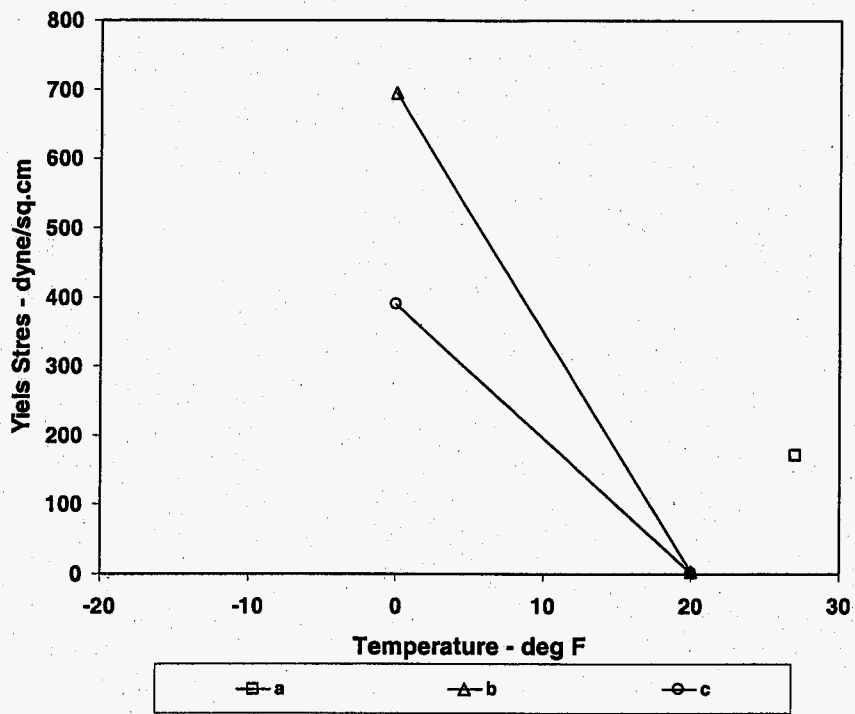
Compositions by %

Sample	GTL	ANS Crude
a	100%	0%
b	25%	75%
c	20%	80%

FIGURE 3.12 Yield stress data of GTL 2 and TAPS crude oil blends as a function of test temperature
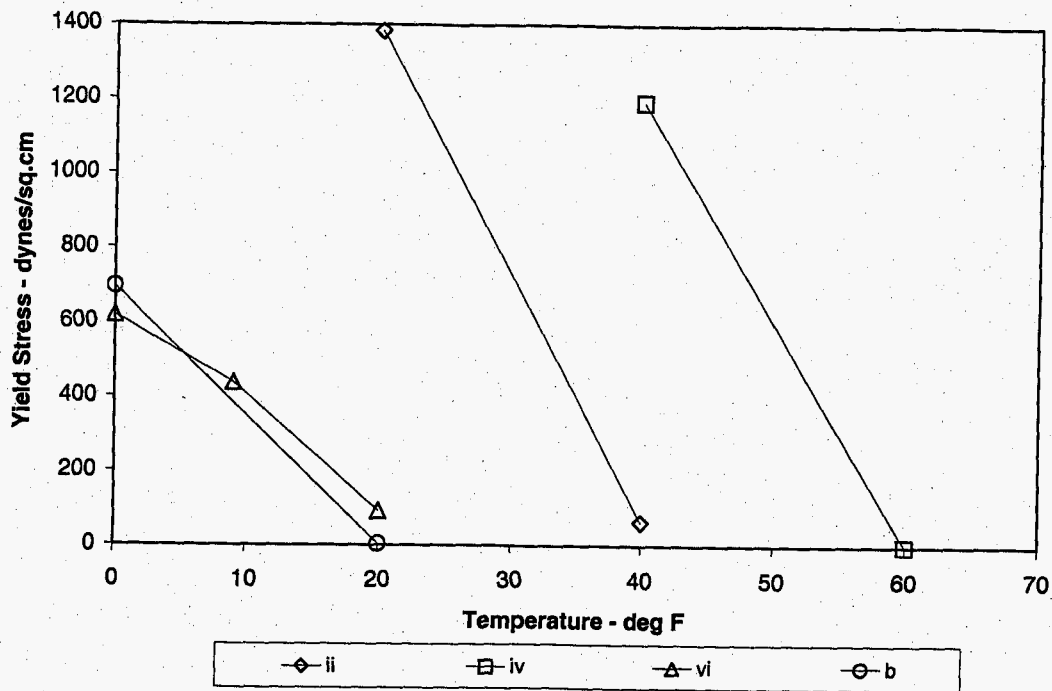
FIGURE 3.13 Comparison of gel strength measurements of GTL 1 and GTL 2 TAPS crude oil blends for the ratio of 1:3 GTL1/2:Crude oil.
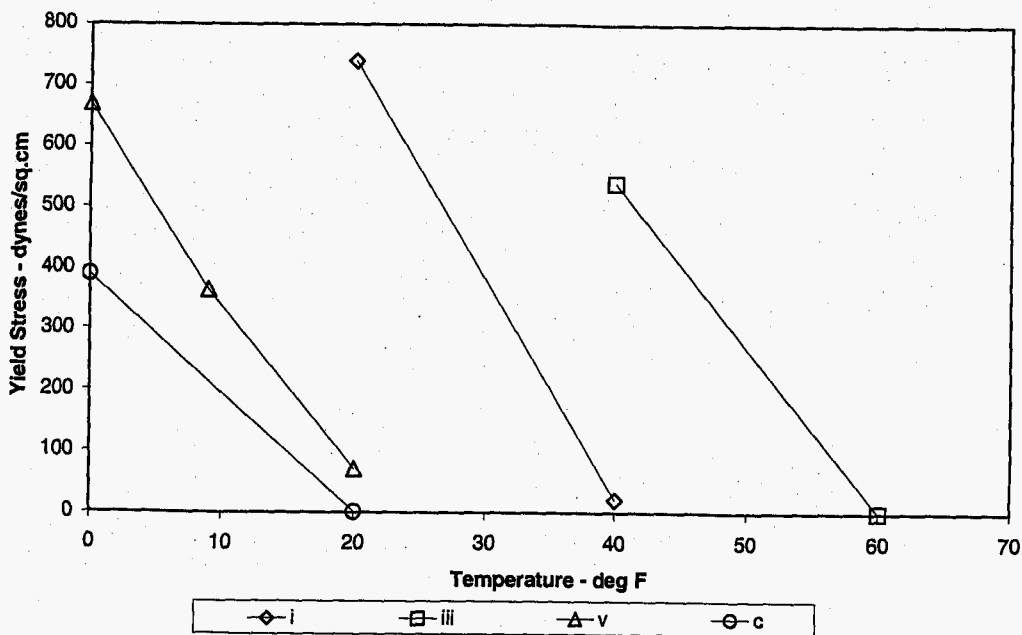
FIGURE 3.14 Comparison of gel strength measurements of GTL 1 and GTL 2 TAPS crude oil blends for the ratio of 1:4 GTL1/2:Crude oil.

CHAPTER 4

HYDRAULIC MODELING OF GTL TRANSPORTATION

Transportation of GTL products through the TAPS can be achieved by using one of the two possible modes. In the first mode, alternate batches or slugs of crude oil and GTL can be transported through the pipeline. This mode is referred to as batching or slugging. A minimum slug length will be required because some mixing between the crude oil and GTL will take place at the leading and trailing edges of the slugs. In the second mode, the GTL products can be mixed with the crude oil and sent through the pipeline as a single liquid phase. This mode is termed blending or commingling.

In order to study the feasibility of GTL transportation through TAPS, it is necessary to predict the pressure drop or gradient along the entire pipeline. Pressure drops occur due to various forces such as gravitational, frictional, etc. The objective of this study is to solve the pertinent energy equations for both batch and commingled flow modes, and to analytically determine the pressure gradients and related hydraulic flow parameters for each transportation mode. The factors that contribute to this pressure drop are examined, and the methods of accounting for them are considered. A comparison of the pressure gradient calculations is presented for the batching and the commingled flow modes.

4.1 BATCH FLOW

The transportation of GTL and Crude Oil in slugs or batches results in the creation of an interface zone between both fluids. This is analogous to two-phase slug flow in pipelines, in that each batch or slug is followed by an air pocket. This interface zone is made up of mostly air pockets, and a mixture of both fluids. The magnitude of the interface zone is a function of the fluid velocity, density differences, viscosity, pipe diameter, length, time and composition (Baum et al., 1998).

Two-phase flow is a more complex phenomenon than single-phase flow, primarily because the distribution of the two phases is unknown and difficult to specify quantitatively. When gas and liquid flow simultaneously in a pipe, the two phases can distribute themselves in a variety of flow configurations, depending on operating parameters, physical properties of the two-phases, as well as geometrical variables (for purposes of this work, any mention or reference to "gas", is in actuality, a reference to the air pockets between slugs). In addition, the flow is affected by various factors such as the liquid hold-up, void fraction, pressure loss etc.

The fundamental flow patterns in two-phase flow as classified by Baker (1954) are:
  i) Stratified flow: Flow in which the liquid flows along the bottom of the pipe and the gas flows above, over a smooth liquid interface.
  ii) Wavy flow: This is similar to stratified flow except that the gas moves at a higher velocity and the interface is disturbed by waves traveling in the direction of flow.
  iii) Slug flow: Flow in which a wave is picked up periodically by the more rapidly moving gas, to form a frothy slug which passes through the pipe at a much greater velocity than the average liquid velocity.

iv) Plug flow: Flow in which alternate plugs of liquid and gas move along the upper part of the pipe.

v) Bubble flow: Flow in which bubbles of gas move along the upper part of the pipe at approximately the same velocity as the liquid.

vi) Annular flow: Flow in which the liquid forms a film around the inside wall of the pipe and the gas flows at a high velocity as a central core.

vii) Spray flow: Flow in which most or nearly all of the liquid is entrained as a spray by the gas.

These flow patterns have been further classified into four major types: Stratified Flow (Stratified Smooth and Stratified Wavy), Intermittent Flow (Elongated Bubble Flow and Slug Flow), Annular Flow (Annular Mist Flow and Annular Wavy Flow), and Dispersed Flow (Taitel et al, 1976; Aziz et al, 1978).

Slug flow occurs because of the velocity difference in the flow of gas and liquids. The liquid phase grows in amplitude until; it succeeds in bridging the entire cross-section of the pipe to form a "slug". The slug is immediately accelerated to an average stable velocity, by the gas behind it (Govier and Aziz, 1972). The length of the gas bubble depends on the flow rates and the fluid properties, and for given flow rates, it depends on the manner in which the fluids are introduced. It also depends on the system pressure and therefore increases as the pressure declines in the direction of flow (Govier and Aziz, 1972).

Various models have been proposed to account for or describe slug flow in horizontal pipes or tubes. Kordyban (1961) was the first to propose such a model. In his model, the liquid slug moves at the average velocity of the gas bubble and "skates" over the top of the more slowly moving liquid below it. Based upon this concept, a pressure drop expression was developed. Govier and Aziz (1972) later discovered that the model was oversimplified and inadequate.

Dukler and Hubbard (1975) presented a model that until today remains the reference point for the analysis of gas-liquid slug flow in pipes (Figure 4.1). The model permits the prediction in detail of the unsteady hydrodynamic behavior of gas-liquid slug flow. It is based on the observation that a fast moving slug overruns a slow moving liquid film, accelerating it to full slug velocity in a mixing eddy located at the front of the slug. A new film is shed behind the slug ("scooping mechanism") that decelerates with time.
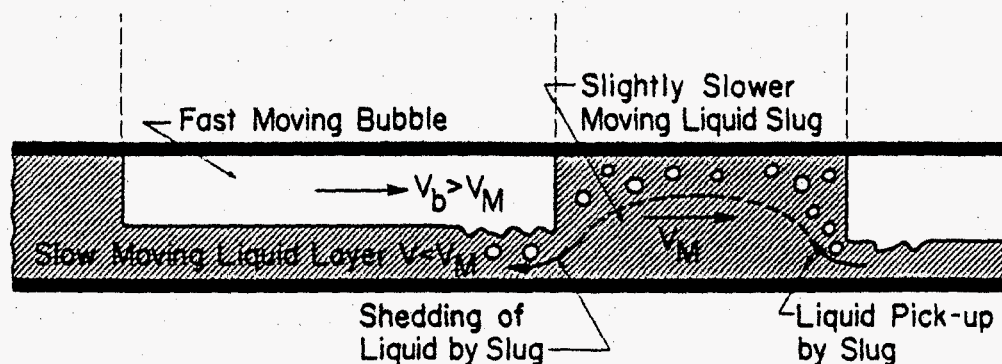
FIGURE 4.1 Schematic Representation of the Dukler and Hubbard Model

The model is based on the following assumptions:

i)   Steady state representation of the slug.

ii)   Mixing in the slug is a result of a mixing eddy and diffusion due to turbulence.

iii)   Slug length is constant.

iv)   Amount of liquid scooped at the head of the liquid is equal to the amount of liquid shed at its tail.

v)   Pressure drop across the film is negligible.

The model has the ability to predict the slug fluid velocity, length of the slug, film region behind the slug, film distance as a function of time and distance, as well as the pressure drop (containing an acceleration and frictional term) across the slug. In 1989, Kokal et al. highlighted a shortcoming of the model, in that it requires the values of slug frequency and liquid hold-up in the liquid slug, which are difficult to estimate.

Over the years, various workers have modified the basic assumptions inherent in the Dukler and Hubbard model, and have derived new models or procedures for obtaining the parameters required for the description of slug flow. A review of these models is available in the literature (Akwukwaegbu, 2001).

4.2 COMMINGLED FLOW

In this mode of transportation, the Crude Oil and GTL are blended, before being sent through the pipeline, as a single liquid phase mixture. This mode is termed blending or commingling. The transport of such fluid mixtures in horizontal or nearly horizontal pipes has become the norm, especially in the gathering and processing of hydrocarbons. This enables major cost savings in pipeline construction, and permits the centralization of processing facilities. This usually results in the improvement of processing economics and conservation of resources.

When a mixture of fluids flows in a system, the component fluids can be distributed in a variety of flow configurations or patterns, depending on the operating parameters, physical properties of the fluids, as well as geometrical variables. The flow may also be affected by pressure losses in the system, liquid holdup (as a result of density differences) etc.

Since GTL and Crude Oil are both hydrocarbons, and as such may have very similar fluid properties, the possibility exists of blending both fluids into one homogeneous mix. This is subject to laboratory testing to determine the actual fluid properties of the resulting fluid mixture.

As part of the GTL project, tests were conducted by the Petroleum Engineering Department at the University of Alaska Fairbanks, on samples of GTL and Crude Oil (Ramakrishnan, 2000). From the results of the tests, it was observed that when both fluids were mixed, they blended into a single homogeneous liquid. There was no separation into distinct layers or boundaries when the mixture was left to stand.

This then allows the flexibility of treating the mixture as a single-phase homogeneous liquid, with its own unique fluid properties. In studying the commingled flow of GTL and Crude Oil through the Trans-Alaska Pipeline System, the Bernoulli equation of pressure for the flow of fluids in pipes is used. This equation forms the basis for any analysis in the area of fluid mechanics, and has been discussed in detail, by a great number of researchers.

4.3 DEVELOPMENT OF MODEL EQUATIONS

In studying the flow of Gas To Liquids and Crude Oil through the Trans Alaska Pipeline System (TAPS), in either batch or commingled mode, the primary concern will be on the expected pressure drop or gradient along the entire pipeline. Such pressure drop may be due to a number of reasons, such as friction, hydrostatics etc. In carrying out a proper study, the various factors that contribute to this pressure drop are examined, and the methods of accounting for them are considered. This will be achieved by presenting mathematical models or equations, which are used to obtain numerical values for these factors, and as such, allow a proper understanding of the role played by these factors in the hydraulics.

4.3.1 Batch Flow Model

In this transport mode, alternate batches or slugs of crude oil and GTL can be transported through the pipeline. This mode is also referred to as batching or slugging. A minimum slug length will be required because some mixing between the crude oil and GTL will take place at the leading and trailing edges of the slugs. The study of the expected pressure drop, that occurs during transportation in slugs or batches will focus on the minimum slug length, length of the interface (or void space) between the slugs, as well as the length of the mixing zone. Development of batch flow model equations is described in the following sections.

4.3.1.1 Assumptions

In studying the batching or slugging mode of transport, the following assumptions have been made:

   i)   Incompressible fluid flow, steady state and fully developed.
   ii)   Constant slug length.
   iii)   The bubble (void) between the slugs is occupied by air.
   iv)   The liquid film has a constant thickness.
   v)   Flow is isothermal with constant fluid properties
   vi)   There is some degree of mixing between the trailing film edge and the head of the slug.

4.3.1.2 Governing Equations

The slug body is divided into two sections (see Figure 4.2), the liquid slug zone of length l_s, and the mixing zone of length, l_m. In the original work, the mixing zone was construed to consist of a liquid film, and an elongated air bubble (Taitel, et al, 1990). For this work, this definition has been modified, such that the mixing zone is the interface between slugs.

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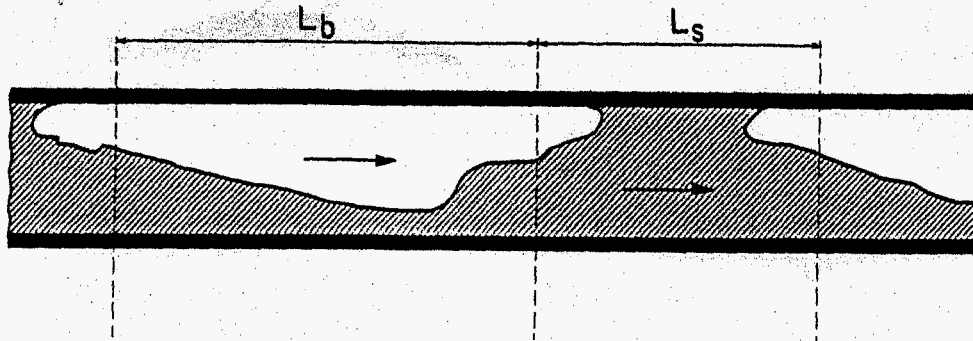
FIGURE 4.2 Schematic Representation of Slug Flow (Govier and Aziz, 1972)

The pressure drop across one slug unit is calculated from

$$\Delta P = \Delta P_f + \Delta P_a + \Delta P_h \qquad (4.1.1)$$

where $\Delta P_f, \Delta P_a, \Delta P_h$ are the pressure drops due to friction, acceleration, and hydrostatic forces respectively (Kokal, et al., 1989; Taitel, et al, 1990). The pressure drops are affected by the flow regime of the fluid i.e. laminar (streamlined) or turbulent.

4.3.1.2.1 Pressure Drop Due To Friction

This is the pressure drop due to frictional forces within the liquid slug and the void (air pocket and liquid film). Taitel and Barnea (1990) presented Equation (4.1.2) in order to determine the pressure drop due to friction. It is a combination of the friction forces produced by the individual components of a typical slug.

$$\Delta P_f = \frac{2f_s \rho_{mz} V_m^2 l_s}{D} + \frac{2f_g \rho_g V_t^2 l_f}{D_g} + \frac{2f_f \rho_l V_f^2 l_f}{D_f} \qquad (4.1.2)$$

where the friction factors of the slug, f_s, air bubble, f_g, and liquid film (fluid interface zone), f_f are based on the Reynolds number of the slug, R_{es}, air bubble, R_{eg}, and the film, R_{ef}. For this work, it is assumed that the effects of the air pocket or bubble, are negligible, hence Equation (4.1.2) then becomes;

$$\Delta P_f = \frac{2f_s \rho_l V_m^2 l_s}{D} + \frac{2f_f \rho_{mz} V_f^2 l_m}{D_f} \qquad (4.1.3)$$

The Moody friction factor is applied for laminar flow regime, and is defined as:

$$f = \frac{64}{R_e} \qquad (4.1.4)$$

The Zigrang and Sylvester (1985) equation for turbulent flow, which incorporates the pipe roughness factor, ε, can be given by:

$$\frac{1}{\sqrt{f}} = -2\log\left[\frac{\varepsilon/D}{3.7} - \frac{5.02}{N_{R_e}}\log\left(\frac{\varepsilon/D}{3.7} + \frac{13}{N_{R_e}}\right)\right] \qquad (4.1.5)$$

The Reynolds number for the slug, and film respectively, are obtained from the following expressions:

$$R_{es} = \frac{DV_m\rho_l}{\mu_l} \qquad (4.1.6)$$

$$R_{emz} = \frac{D_f V_f \rho_{mz}}{\mu_{mz}} \qquad (4.1.7)$$

Where,

$$\rho_{mz} = \rho_{l1}E_{ls} + (1 - E_{ls})\rho_{l2} \qquad (4.1.8)$$

$$\mu_{mz} = \mu_{l1}E_{ls} + (1 - E_{ls})\mu_{l2} \qquad (4.1.9)$$

ρ_{mz}, ρ_{l1} and ρ_{l2}, are the densities of the mixing zone and slugs respectively; μ_{mz}, μ_{l1}, and μ_{l2} are the viscosities of the mixing zone and slugs respectively; E_{ls}, is the liquid holdup in the liquid slug; E_{lf}, is the liquid holdup in the interface zone; D_f is the hydraulic diameter occupied by the interface zone.

4.3.1.2.2 Pressure Drop Due To Acceleration

The film velocity, V_f, just before slug pick-up, is lower than the velocity in the main body of the slug, V_s. This necessitates the acceleration of the film to match the velocity of the slug. As a result, there is a pressure drop generated by this, and it can be defined as (Kokal et al, 1989):

$$\Delta P_a = \rho_l E_{ls}(V_t - V_s)(V_s - V_f) \qquad (4.1.10)$$

4.3.1.2.3 Hydrostatic Pressure Drop

This pressure drop can be experienced in any system because of the pipe orientation or inclination. Equation (4.1.11) was presented by Kokal et al (1989) and Taitel et al (1990) to determine the pressure drop due to pipe inclination.

$$\Delta P_h = \rho_{ms}(g\sin\beta)l_s + \rho_f(g\sin\beta)l_f \qquad (4.1.11)$$

where:

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$$\rho_f = \rho_{l1} E_{lf} + (1 - E_{lf}) \rho_{l2} \tag{4.1.12}$$

β is the angle of inclination. Since $\sin \beta = h/L = \Delta z/L$, equation (4.1.11) can be re-written as

$$\Delta P_h = \left(\rho_{ms} l_s + \rho_f l_f \right) g \, \Delta z/L \tag{4.1.13}$$

For the purposes of this work, the Equation (4.1.13) is presented as

$$\Delta P_h = \left(\rho_{l1} l_s + \rho_{ms} l_m \right) g \, \Delta z/L \tag{4.1.14}$$

The schematic arrangement of the batches or slugs, is as shown in Figure 4.3.

The total pressure drop across the slug can be calculated from the sum of equations (4.1.3), (4.1.10) and (4.1.14). This would require the determination of the following quantities: slug length, l_s; liquid hold-up in the slug, E_{ls}; average fluid velocity in the slug, V_s; film velocity, V_f; and length of the mixing zone, l_m.
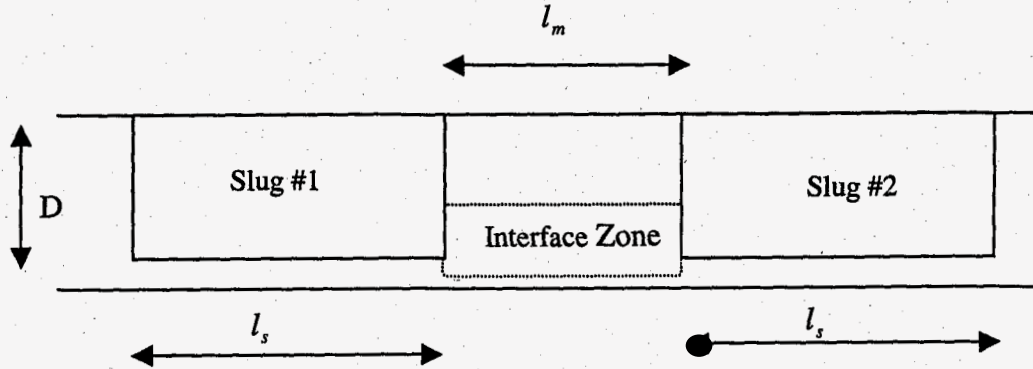
FIGURE 4.3 Schematic Representation of Batch or Slug Flow

4.3.1.2.4 Slug Length
This is the length of a slug. In 1986, Scott et al. presented a correlation for the determination of the slug length for large diameter pipes, and which is given by:

$$\ln(l_s) = -25.4144 + 28.4948 (\ln(D))^{0.1} \tag{4.1.15}$$

4.3.1.2.5 Average Fluid Velocity
By conducting a momentum balance over a slug unit, the average fluid velocity is given by (Govier et al, 1972; Kokal et al, 1989; Taitel et al, 1990; Fan et al, 1993; Sharma et al, 1998).

$$V_m = \frac{Q}{A} = \frac{Q_1 + Q_2}{A} = V_{s1} + V_{s2} \qquad (4.1.16)$$

where V_{s1} and V_{s2} are the superficial velocities of the slugs respectively. The average slug velocity, V_s, can be determined from equation (4.1.16) by setting it equal to the average fluid velocity.

$$V_m = V_s \qquad (4.1.17)$$

4.3.1.2.6 Transitional Velocity

This is the slug transitional velocity. This can also be defined as the velocity of the leading edge of the slug. In 1990, Taitel and Barnea, presented a correlation, which is actually a linear combination of the interface velocity.

$$V_t = C_o V_s + V_d \qquad (4.1.18)$$

where $C_o = 2$ for laminar flow, $C_o = 1.2$ for turbulent flow, and V_d is the propagation or drift velocity and is defined as (Kokal et al, 1989):

$$V_d = 0.345 \sqrt{\frac{gD(\rho_{l1} - \rho_{l2})}{\rho_{l1}}} \qquad (4.1.19)$$

4.3.1.2.7 Liquid Slug Hold-up

When there is a difference in phase properties (density and/or viscosity), one of them, usually the less dense phase, tends to flow at a higher in situ average velocity than does the other. This gives rise to the existence of slip of one phase past the other, or holdup of one phase relative to the other. In 1996, Abdul-Majeed presented a correlation for the determination of the liquid holdup in the slug. It is a modification of the Lockhart-Martinelli parameter (1949). Equations (4.1.20) and (4.1.21) are for turbulent and laminar flow regimes respectively.

$$(E_{ls})_{theoretical} = \exp(-0.9304919 + 0.5285852R - 9.219634 \times 10^{-2} R^2 + 9.02418 \times 10^{-4} R^4)$$
$$(4.1.20)$$

$$(E_{ls})_{theoretical} = \exp(-1.099924 + 0.6788495 R - 0.1232191 \times 10^{-2} R$$
$$- 1.778653 \times 10^{-3} R^3 + 1.626819 \times 10^{-3} R^4)$$
$$(4.1.21)$$

where $R = \ln(X)$, and

$$X = \left[\frac{V_{s2}\rho_{l2}\mu_{l1}}{V_{s1}\rho_{l1}\mu_{l2}} \right]^m \frac{\rho_{l1}V_{s1}^2}{\rho_{l2}V_{s2}^2} \qquad (4.1.22)$$

X is the Lockhart-Martinelli parameter (1949), and $m = 0.2$ for turbulent flow and $m = 1$ for laminar flow. Due to assumptions made in the development of the model, a correction was made to the value of the liquid holdup obtained from both equations:

$$(E_{ls})_{actual} = C(E_{ls})_{theoretical} \qquad (4.1.23)$$

where

$$C = 0.528(V_{s2}V_{s1})^{-0.215121} \qquad (4.1.24)$$

4.3.1.2.8 Interface Velocity
From the original data of Dukler and Hubbard (1975) model, the film velocity is given as

$$V_f = V_m \left(\frac{1}{1 + \dfrac{0.2V_m}{\omega}} \right) \qquad (4.1.25)$$

where ω is the slug frequency, and is given by equation (4.1.26) as (Govier et al, 1972)

$$\omega = 0.0226 \left[\frac{V_{sl}}{gD} \left(\frac{19.75}{V_m} + V_m \right) \right]^{1.2} \qquad (4.1.26)$$

4.3.1.2.9 Length of the Mixing Zone
This is the interface region between slugs. This interface zone is made up of mostly air pockets, and a mixture of both fluids. The magnitude of the interface zone is a function of the fluid velocity, density differences, viscosity, composition, time, pipe diameter and length. It is characterized by a rapidly varying liquid hold-up. This was originally presented in the Dukler and Hubbard model (1975) as,

$$l_m = \frac{0.15}{g}(V_m - V_f)^2 \qquad (4.1.27)$$

It is observed that at large values of V_m, equation (4.1.27) largely over predicts l_m. In 1993, Andreussi et al. proposed a new correlation that corrects such over predictions, and is given by:

$$l_m = k_m(1 - E_{ls})D \qquad (4.1.28)$$

where k_m is a factor for the length of the mixing zone and is approximately equal to 30.

4.3.1.2.10 Liquid Hold-up in the Mixing Zone

At steady state, the mass exchange rate between the liquid slug and the film is expressed as (Govier et al., 1972; Dukler and Hubbard, 1975; Nicholson et al., 1978; Kokal et al., 1989; Taitel et al., 1990):

$$\rho_l A E_{ls}(V_t - V_s) = \rho_l A E_{lf}(V_t - V_f) = m_e^R \tag{4.1.29}$$

From equation (4.1.28), the film hold-up can be obtained as

$$E_{lf} = E_{ls}\frac{(V_t - V_s)}{(V_t - V_f)} \tag{4.1.30}$$

4.3.1.2.11 Interface Hydraulic Diameter

This is fraction of the actual pipe diameter occupied by the film (interface). In calculating the hydraulic diameter, the approach presented by Darby (1996) will be followed. If the height of the interface within the pipe is given as h (which can either be smaller or larger than the radius of the pipe, R), then the cross-sectional area can be obtained from equation (4.1.31a)

$$A = R^2\left[\cos^{-1}\left(1 - \frac{h}{R}\right) - \left(1 - \frac{h}{R}\right)\sqrt{1 - \left(1 - \frac{h}{R}\right)^2}\right] \tag{4.1.31a}$$

From equation (4.1.31b), the wetted perimeter can be calculated as:

$$W_p = 2R\cos^{-1}\left(1 - \frac{h}{R}\right) \tag{4.1.31b}$$

As a result, the interface hydraulic diameter can then be calculated from:

$$D_f = \frac{4AE_{lf}}{W_p} \tag{4.1.31c}$$

Setting the change in elevation equal to the head loss due to friction initializes this iterative procedure,

$$\Delta z = h_f = \frac{2f_f L Q^2}{gD_f A^2} \tag{4.1.31d}$$

which is outlined as follows:

i)   A value is assumed for h/R, and the parameters A, W_p and D_f are determined from equations (4.1.31a), (4.1.31b) and (4.1.31c) respectively.

ii)   From equation (4.1.7) the interface Reynolds number, $N_{R_{ef}}$, is calculated.

iii) The interface frictional factor, f_f, can be computed as function of $N_{R_{ef}}$ by using equations (4.1.4) and (4.1.5).

iv)   By assuming values for h/R, an iterative procedure is applied to obtain solutions to the right hand side (RHS) of equation (4.1.31d). The guessed values of h/R are continuously adjusted until a tolerance limit is reached.

4.3.1.2.12 Average Pressure Gradient

The average pressure gradient is determined for one complete slug unit, by dividing the total pressure drop across a slug, by the effective slug length. This is given by equation (4.1.32) as

$$\frac{\Delta P}{L} = \frac{\Delta P_f + \Delta P_a + \Delta P_h}{l_s}$$

(4.1.32)

4.3.2 Commingled Flow Model

In this transport mode, the GTL and Crude Oil are pre-mixed before shipment through the TAPS as a single phase. For the purpose of this analysis, it is assumed that the fluids are homogeneously mixed, and that due to the envisioned throughput, there will be no separation into distinct layers.

4.3.2.1 Assumptions

In studying the commingled mode of transport, the following assumptions will have to be made:

i)   Incompressible fluid flow, steady state and fully developed
ii)   Flow is isothermal with constant fluid properties.
iii) Fluid exhibits Newtonian behavior
iv)   No separation into constituent fluids.

4.3.2.2 Governing Equations

Consider a finite element of an inviscid (frictionless) fluid, subject only to the action of gravity, (i.e. the fluid is at rest). Applying Newton's third law of motion to this fluid element (Landau et al., 1959; Bird et al., 1960; Kaufmann, 1963; Streeter et al., 1985)

$$F_s = dm \frac{dv}{dt}$$

(4.2.1)

where F_s, is the resultant of all external forces in the direction of the streamline; v, is the fluid velocity; and, dm, is the mass of the element.

The forces acting on the element are the weight and the end forces (pressure difference between the upper and lower faces), as shown in Figure 4.4. Thus, from equation (4.2.1),

$$\rho g \cdot ds \cdot dA \cdot \cos\theta + P \cdot dA - dA\left(P + \frac{\partial P}{\partial s} ds\right) = \rho \cdot ds \cdot dA \frac{\partial v}{\partial t} \tag{4.2.2}$$

Since θ is the angle sustained by the particle with the horizontal,

$$\cos\theta = -\frac{\partial z}{\partial s} \tag{4.2.3}$$

Equation (4.2.2) then becomes:

$$-\left(\rho g \cdot ds \cdot dA \frac{\partial z}{\partial s}\right) - \left(ds \cdot dA \cdot \frac{\partial P}{\partial s}\right) = \rho \cdot ds \cdot dA \frac{dv}{dt} \tag{4.2.4}$$

Equation (4.2.4) then simplifies to:

$$\frac{dv}{dt} = -g\frac{\partial z}{\partial s} - \frac{1}{\rho}\frac{\partial P}{\partial s} \tag{4.2.5}$$

In general, the fluid velocity, v, is a function of both time and location, s, along the streamline. Therefore, the total derivative for the velocity term is given as,

$$dv = \frac{\partial v}{\partial s} ds + \frac{\partial v}{\partial t} dt \tag{4.2.6}$$

Since, velocity is the rate of change of distance with time, the actual acceleration of the particle in the direction of flow becomes:

$$\frac{dv}{dt} = v\frac{\partial v}{\partial s} + \frac{\partial v}{\partial t} \tag{4.2.7}$$
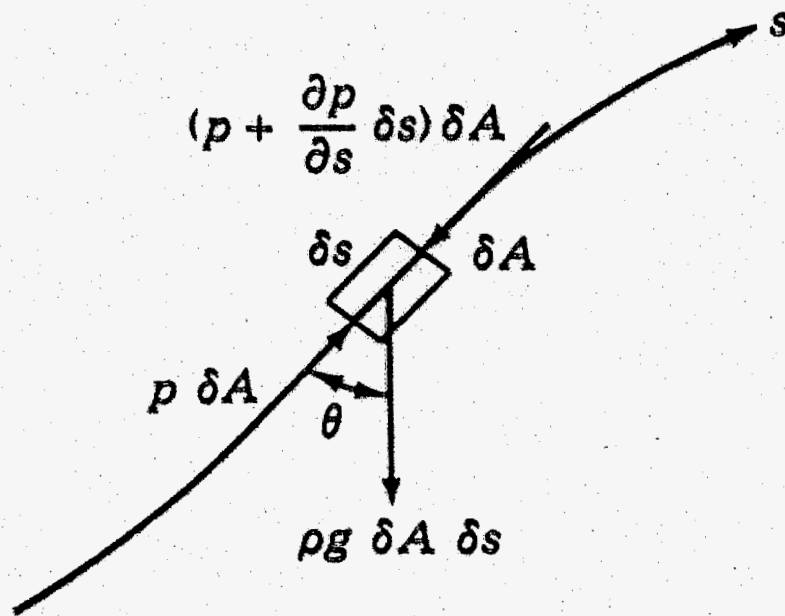
FIGURE 4.4 Force Balance on a Finite Element (Streeter, 1985)

On the assumption of steady state flow, equation (4.2.7) reduces to,

$$\frac{dv}{dt} = v \frac{\partial v}{\partial s} \tag{4.2.8}$$

This on substitution into equation (4.2.5) and re-arranging yields

$$v \frac{\partial v}{\partial s} + g \frac{\partial z}{\partial s} + \frac{1}{\rho} \frac{\partial P}{\partial s} = 0 \tag{4.2.9}$$

Since the distance, s, is the only independent variable, the partial derivatives are replaced by total derivatives, and thus, equation (4.2.9) then becomes

$$\frac{1}{\rho} \frac{dP}{ds} + g \frac{dz}{ds} + v \frac{dv}{ds} = 0 \tag{4.2.10}$$

Equation (4.2.10) is best known as the Euler's equation of motion along a streamline (Landau et al, 1959).

All the terms in equation (4.2.10) are derivatives with respect to distance, s. This then enables the integration along the streamline to obtain

$$\frac{v^2}{2} + \frac{P}{\rho} + gz = C_e \tag{4.2.11}$$

where C_e is a constant. Equation (4.2.11) is more commonly known as the Bernoulli equation of pressure in steady flow or the equation of energy for steady flow.

For flow between points 1 and 2, equation (4.2.11) is written as,

$$\left(\frac{V_2^2}{2g} + \frac{P_2}{\rho_2 g} + z_2\right) - \left(\frac{V_1^2}{2g} + \frac{P_1}{\rho_1 g} + z_1\right) = C_e \tag{4.2.12}$$

In equation (4.2.12), $V^2/2g$, and $P/(\rho g)$ are the velocity and pressure heads respectively. The last term, z, is the elevation or geometric head of the fluid above an arbitrary reference plane (Kaufmann, 1963; Holland, 1973).

4.3.2.2.1 Energy Losses
Since most natural liquids are very nearly incompressible (i.e. constant density), they are not inviscid (frictionless). Internal friction (viscosity) converts part of the flow energy into other energy forms such as sound, heat etc. and it is "lost" (Kaufmann, 1963). This loss is normally considered as a "head", the friction head, h_f, and is given by the Darcy-Weisbach equation (Smith et al, 1960) as:

$$h_f = 4f\frac{LV^2}{2gD} \tag{4.2.13}$$

Therefore, equation (4.2.12) is re-written as,

$$\left(\frac{V_1^2}{2g} + \frac{P_1}{\rho\, g} + z_1\right) = \left(\frac{V_2^2}{2g} + \frac{P_2}{\rho g} + z_2\right) + h_f \tag{4.2.14}$$

For steady incompressible flow through a pipe, between points 1 and 2, with a pump at one end, equation (4.2.14) can be re-written as,

$$\left(\frac{V_2^2}{2g} + \frac{P_2}{\rho g} + z_2\right) - \left(\frac{V_1^2}{2g} + \frac{P_1}{\rho g} + z_1\right) = \Delta h_p - h_f \tag{4.2.15}$$

where Δh_p, is the head imparted to the fluid by the pump (Holland, 1973).

This then implies that the total pressure drop across the streamline is given as

$$\frac{P_1 - P_2}{\rho g} = \frac{\left(V_2^2 - V_2^2\right)}{2g} + \left(z_2 - z_1\right) + \left(h_f - \Delta h_p\right) \tag{4.2.16}$$

or simply

$$\Delta P = \rho g \left[\left(\frac{V_2^2 - V_1^2}{2g}\right) + \left(z_2 - z_1\right) + \left(h_f - \Delta h_p\right)\right] \tag{4.2.17}$$

Equation (4.2.17) will form the basis for the study of the commingled flow of GTL and Crude Oil through TAPS.

4.4 APPLICATION OF MODEL EQUATIONS

In choosing the appropriate mode for transporting GTL through TAPS, i.e. either batch or commingled flow, the derived model equations will have to be applied to estimate the expected pressure drop for each mode. Based on the results obtained from the computations, a reasonable choice can then be made.

4.4.1 Calculation Algorithm
The Trans-Alaska Pipeline System (TAPS) is an 800 miles long and 48" diameter pipeline. For computational purposes, it has been divided into six (6) major pipe sections. These sections are as follows:

i.   Pump Station #1 to Pump Station #3 (Length, L = 104.27 mi.; Change in elevation, Δz = +1344.3 ft.)
ii. Pump Station #3 to Pump Station #4 (L = 39.79 mi.; Δz = +1380 ft.)
iii. Pump Station #4 to Pump Station #7 (L = 270.02 mi.; Δz = -1859.1 ft.)
iv. Pump Station #7 to Pump Station #9 (L = 134.66 mi.; Δz = + 604.3 ft.)
v.   Pump Station #9 to Pump Station #12 (L = 186.36 mi.; Δz = +312.6 ft.)
vi. Pump Station #12 to Valdez Terminal (L = 65.1 mi.; Δz = -1655.4 ft.)

The successful application of the model equations requires a prior knowledge of fluid properties, such as density and viscosity. Also important, is the knowledge of the pipe parameters (diameter, length, geometry), as well as current operating conditions (flow rate, pump information, pipe specifications). The systematic procedures necessary for the determination of the total pressure drop, as well as the average pressure gradient, are outlined in the following sections.

4.4.1.1 Batch Flow
For this transport mode, the focus will also be on the determination of the average slug length, length of the mixing zone, and liquid holdup in the slug.

The sequential steps, which are carried out for each pipe section, are outlined as follows:

i.   From equation (4.1.16), the mixture velocity, V_m, is calculated as a function of the fluid flow rates.

ii. The transitional velocity, V_t, is calculated by combining equations (4.1.18) and (4.1.19).

iii. The determination of the liquid holdup in the slug is a four (4) step process, which can be listed as;
   a) Determine the Lockhart-Martinelli parameter, X, from equation (4.1.22)
   b) From equation (4.1.24), the correction factor, C, is obtained.
   c) The theoretical liquid holdup is obtained from either equations (4.1.20) or (4.1.21).
   d) Using the value obtained for C from (b) above, the true liquid holdup is calculated using equation (4.1.23).

iv. The length of the slug, l_s, is obtained by using equation (4.1.15).

v.   From equation (4.1.28), the length of the mixing zone, l_m, is calculated.

vi. The interface velocity, V_f, is obtained from equation (4.1.25), as a function of V_m, and the slug frequency, ω, obtained from equation (4.1.26).

vii. From equations (4.1.31a-d), a value for the effective diameter of the interface or film, is obtained.

viii. Using equations (4.1.6) and (4.1.7), the Reynolds number, N_{R_e}, for the slug, and film, are calculated as functions of densities, velocities, diameters, and viscosities.

ix. Depending on the flow regime, the appropriate friction factor, f, is calculated as a function of the Reynolds' number, using either equation (4.1.4) or (4.1.5).

x.   The pressure drop due to friction, ΔP_f, is calculated from equation (4.1.2).

xi. The pressure drop due to acceleration, ΔP_a, is calculated from equation (4.1.10).

xii. The hydrostatic pressure drop, ΔP_h, is calculated from equation (4.1.14).

xiii. The average pressure gradient, $\Delta P/L$, is calculated from equation (4.1.32).

Finally, the total pressure drop is computed as the sum of the individual pressure drops across each pipe section.

4.4.1.2 Commingled Flow

In this mode, since there is prior mixing of both GTL and Crude Oil before transport, the analysis will be conducted similar to that of a single-phase fluid. The focus will also be on the expected pressure drop across each pipe segment.

The sequential steps, which are carried out for each pipe section, are outlined as follows:

i.   The initial fluid velocity, V_1, is calculated as a function of fluid flow rate, Q, and pipe cross-sectional area, A (similar to equation (4.1.15)).

ii. From equation (4.1.5), the Reynolds' number, N_{R_e}, is calculated, in order to determine the appropriate flow regime (for laminar flow, $N_{R_e} \leq 2000$, and for turbulent flow, $N_{R_e} > 2000$).

iii. Depending on the flow regime, the appropriate friction factor, f, is calculated as a function of the Reynolds' number, using either equation (4.1.3) or (4.1.4).

iv. From equation (4.2.13), the head loss due to friction, h_f, is calculated as a function of the friction factor.

v. Based on the flow rates and number of pumps in service, the head imparted to the fluid by the pumps, Δh_p, can be determined (Note: Since this analysis is based on already existing equipment, this data would have to be obtained from the pump design and specification sheet).

vi. The pressure drop, ΔP, is determined from equation (4.2.17) (Note: Steady state flow, therefore, $V_1 = V_2 = V$).

The total pressure drop is the sum of the individual pressure drops across each pipe section. In general, the total pressure drop, ΔP_t, is calculated as follows:

$$\Delta P_t = \Delta P_1 + \Delta P_2 + \Delta P_3 + \Delta P_4 + \Delta P_5 + \Delta P_6$$

4.5 RESULTS

The calculation path for each mode has been transcribed into computer code for use in the Microsoft Excel® Spreadsheet program. The code is written in the Visual Basic environment. Pressure profiles along the entire length of TAPS for the batch mode and the commingled mode are calculated using the procedures described above. The input data and the results are summarized below.

For batch mode, the pressure gradients in each of the six pipeline sections are calculated for a daily throughput of 1.1MMBPD of both Crude Oil and GTL. Other necessary data are shown below.

Inlet Temperature = 90°F
Crude Oil Specific Gravity = 0.8614
Crude oil viscosity = 6.2 cp
GTL Specific Gravity = 0.73
GTL viscosity = 1.0 cp
Pipe Diameter = 48 in. = 4 ft
Pipe roughness = 0.00001 ft
Interface Diameter ratio = 0.3

For commingled flow mode, a daily throughput of 1.1 MMBPD of total commingled fluid is considered. For a GTL to crude oil ratio of 1:1, the other input data are shown below.

Inlet Temperature = 90°F
Fluid: Specific Gravity = 0.833
Fluid specific gravity = 2.8 cp
Pipe Diameter = 48 inch
Pipe Roughness = 0.00001 ft

The sample calculations were also carried out for different blending ratios of GTL and Crude Oil. The ratios considered were:

i. 100% Crude Oil
ii. 75% Crude Oil + 25% GTL (3:1 ratio)
iii. 50% Crude Oil + 50% GTL (1:1 ratio)
iv. 100% GTL

The pressure gradients for commingled flow obtained from these computations are as shown in Figure 4.5. For comparison, pressure gradients from batch mode and commingled mode are plotted together in Figure 4.6. These results indicate that the pressure gradients obtained from the batch flow calculations are higher than those obtained from that of commingled flow. The reason for this difference is that for batch flow, the pressure gradient is the ratio of the total pressure drop across the slug to the slug length, whereas for commingled flow, it is the ratio of the total pressure drop to the length of the pipe segment.
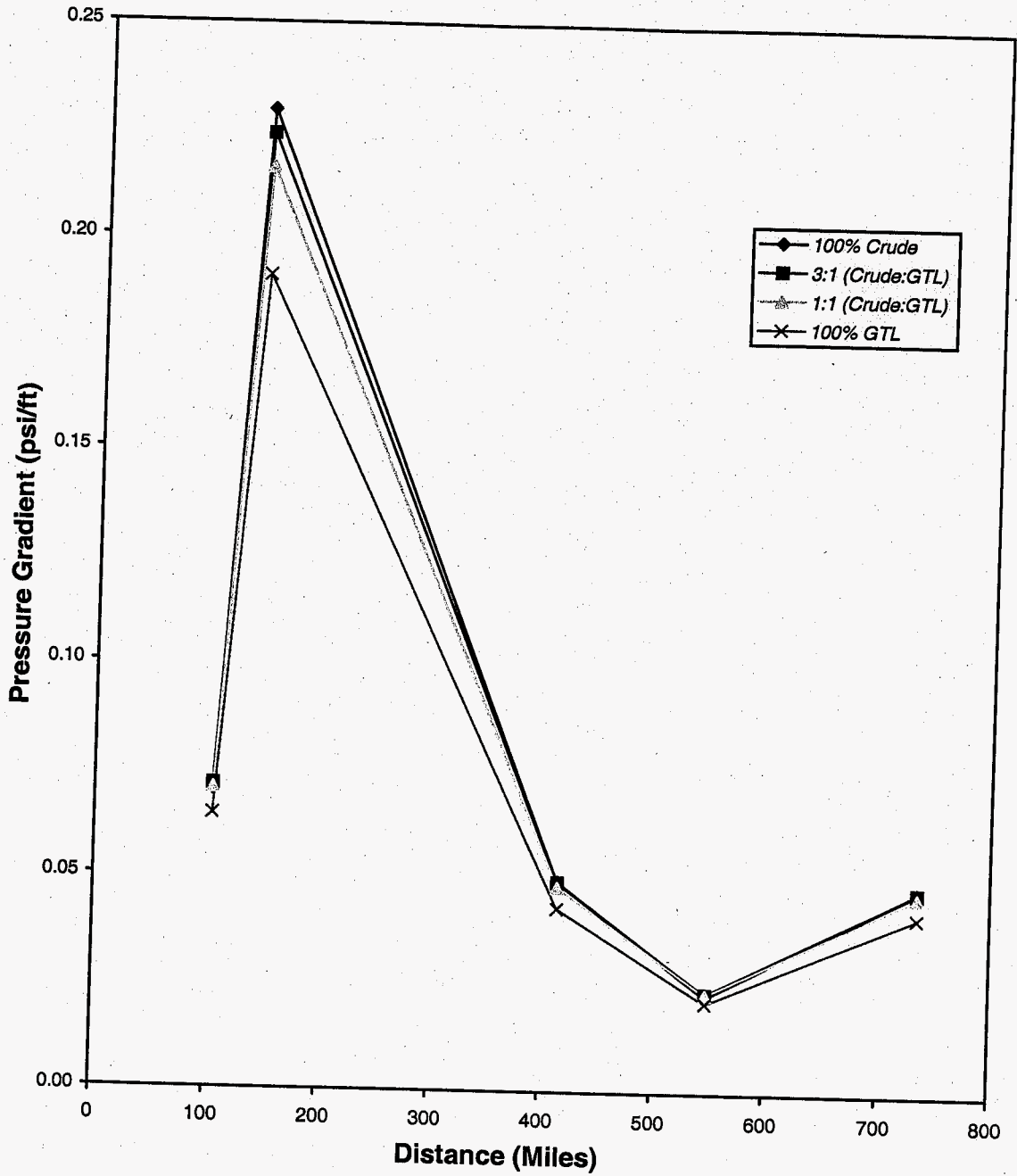
FIGURE 4.5 Pressure Gradient Plot for Commingled Flow
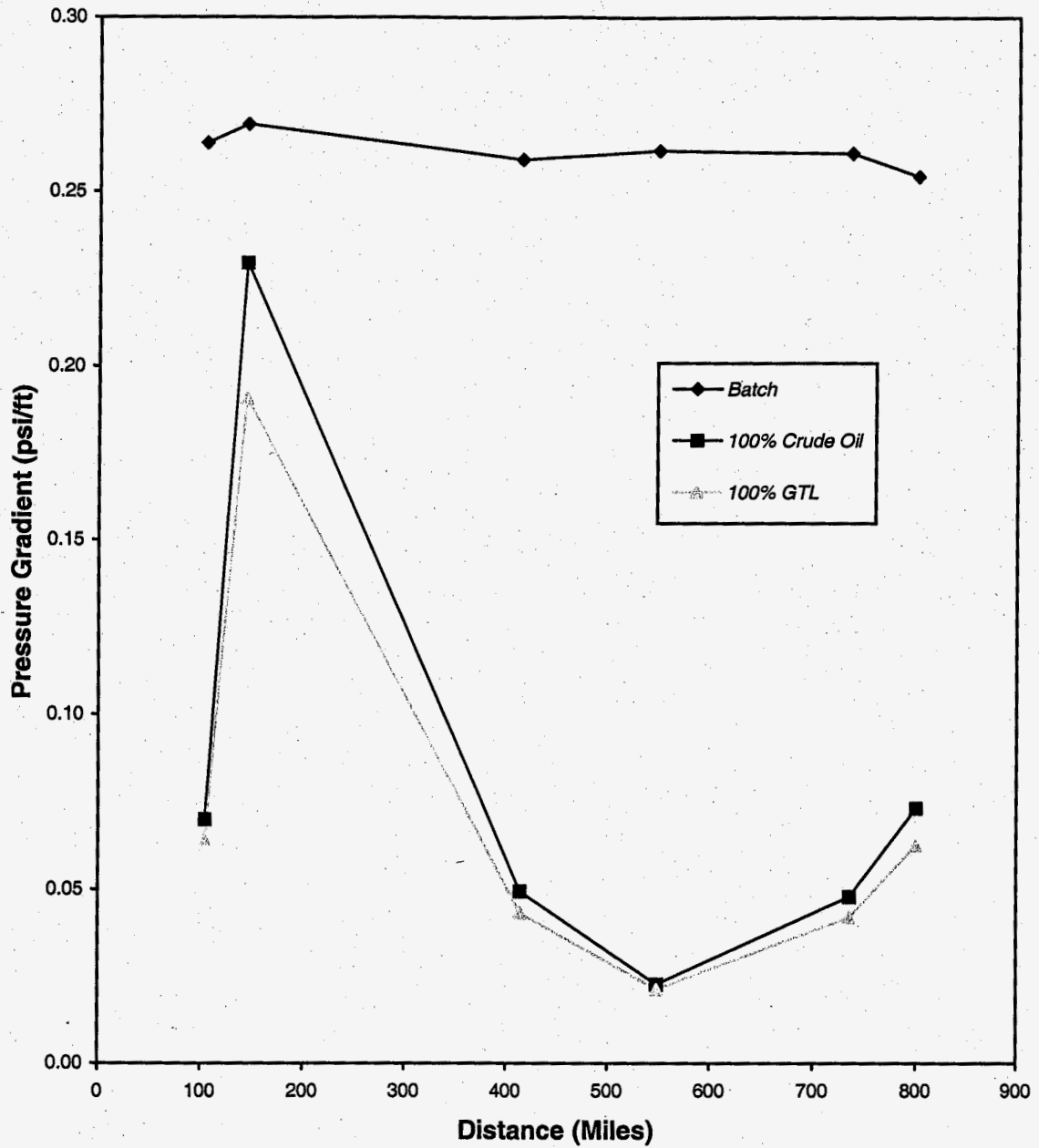
FIGURE 4.6 Comparison Plot of Batch and Commingled Flow Modes

4.6 CONCLUSIONS

The following conclusions are made, based on the results presented in this study.

1. Using the equations presented in this work, batch and commingled flow models can be analytically solved for predicting the pressure gradients encountered when considering the transport of GTL products and Crude Oil through the Trans-Alaskan Pipeline System (TAPS).

2. The derived flow equations presented here can be modified under specified operating conditions or constraints of the Trans-Alaskan Pipeline System (TAPS), using live GTL or Crude Oil data.

3. Mixing at the Oil-GTL interface in the case of batch mode transportation poses flow modeling and simulation difficulties.

4. The pressure gradients obtained from the batch flow calculations are higher than those obtained from that of commingled flow.

4.7 NOMENCLATURE

A	cross-sectional area of the pipe, m^2 [ft^2]
C	correction factor for the liquid hold-up in the slug
C_e	constant in Euler's equation
C_o	film distribution parameter
D	pipe diameter, m [inch.]
D_f	hydraulic diameter occupied by the film, m [inch.]
E_{lf}	liquid holdup in the film
E_{ls}	liquid holdup in the slug
F_S	resultant of forces
f_f	friction factor for the interface zone based on R_{emz}
f_s	friction factor in the liquid slug based on R_{es}
g, g_c	acceleration due to gravity, 9.81 m/s^2 or 32.2 ft/s^2
h, z	height or elevation, m [ft]
h_f	head loss due to friction, m [ft]
Δh_p	pump head, m [ft]
k_m	factor for the length of the mixing zone
L	length or distance, m [ft]
l_m	length of the mixing zone, m [ft]
l_s	length of the slug, m [ft]
m_e	mass exchange rate, kg/s [lbm/s]
N_{R_e}	Reynolds number
ΔP	pressure drop, N/m^2 [psi]
ΔP_a	acceleration pressure drop, N/m^2 [psi]
ΔP_f	frictional pressure drop, N/m^2 [psi]
ΔP_h	hydrostatic pressure drop, N/m^2 [psi]
$\Delta P/L$	average pressure gradient, N/m^2 [psi]

R_{emz}	Reynolds number for the interface zone
R_{es}	Reynolds number for the liquid slug
V_d	drift velocity, m/s [ft/s]
V_f	Interface zone velocity, m/s [ft/s]
V_m	mixture velocity, m/s [ft/s]
V_s	average velocity of the slug, m/s [ft/s]
V_{sl}	superficial liquid velocity, m/s [ft/s]
V_t	transitional velocity, m/s [ft/s]
W_p	Liquid wetted perimeter of the pipe wall, m [inch]
X	Lockhart-Martinelli parameter
Δz	change in elevation , m [ft]
β	angle of inclination, °
ε	pipe roughness, m [ft]
μ_l	Liquid viscosity, cp.
μ_{mz}	Viscosity of the interface zone, cp.
ρ_l	liquid density, kg/m^3 [lb/gallon]
ρ_{mz}	Density of the interface zone, kg/m^3 [lb/gallon]
ω	slug frequency

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CHAPTER 5

THERMODYNAMICS OF GTL TRANSPORTATION

In order to evaluate the feasibility of GTL transportation through TAPS, flow behavior of GTL products and GTL-crude oil blends needs to be accurately modeled. Hydraulic modeling of fluid flow through TAPS has been covered in the previous chapter. In this chapter, thermodynamic considerations in flow of GTL material through TAPS are studied. It is important to study the thermodynamic aspects of fluid flow through TAPS for the following reasons.

    i)   Variation of properties of the fluid due to temperature changes along the pipeline
    ii)   The temperature of the fluid coming out of the TAPS
    iii)   Heat loss from the fluid as it flows through 800 miles of pipe

In studying the flow of GTL products through the TAPS, in either batch or commingled mode, it will be necessary to know the expected heat loss along the entire pipeline. This heat loss is dependent on a number of factors, such as different temperatures of the fluid as it passes through different sections of the pipe, different ambient conditions to which the pipe is exposed, the location of the pipe above or below ground etc. In carrying out a proper study the various parameters that contribute to this heat loss should be examined and the methods of accounting them must be considered. Moreover, the horsepower required to pump the medium between pipeline pump stations are dependent on fluid properties which are functions of temperatures. Thus, thermodynamics of GTL flow through TAPS is important in understanding the flow behavior.

OBJECTIVE OF THIS STUDY

The objective of this study is to develop the heat transfer and fluid dynamic equations and apply them to determine the heat loss and pumping power required for different modes of crude oil and GTL transportation and compare the results. This study helps us in evaluating the capability of TAPS pumping equipment and other auxiliary components at different pump stations to transport GTL considering heat transfer and fluid dynamic aspects. The theory of heat loss from pipeline and governing equations that are used to obtain numerical values are described in the following sections.

5.1 HEAT TRANSFER ANALYSIS

5.1.1 Below Ground Pipe Line

Figure 5.1 shows the configuration of the below ground pipeline.

The heat transfer rate can be calculated by:

$$q = \frac{T_{iav} - T_\infty}{[1/(h_i 2\pi R_1 L)] + [\ln(R_2/R_1)/(2\pi k_p L)] + [1/(k_s S)] + [d_2/(k_{sn} LH)] + [1/(h_o LH)]} \quad (5.1)$$

It includes inside convective heat transfer coefficient, resistance due to pipe wall, soil, snow and outside convective heat transfer coefficient. H represents the width over which heat is transmitted, here it is assumed to be fifteen times the diameter of the pipe, due to two-dimensional nature of heat flow through the soil and snow. For calculation of heat transfer in summer, resistance due to snow $d_2/k_{sn} LH$ is set to zero.

Heat flux is obtained from

$$q'' = q/2\pi R_1 L = U_i (T_{iav} - T_\infty) \quad (5.2)$$

All terms are described in the nomenclature.

Overall Heat Transfer Coefficient

The heat flux can be expressed as the product of the reciprocal of the resistance for a unit area, and an appropriate temperature difference. The reciprocal of resistance for a unit area U, is termed as overall heat transfer coefficient.

U_i based on inside surface area is given by

$$\frac{1}{[1/h_i] + [(R_1/k_p)\ln(R_2/R_1)] + [(2\pi R_1 L)/(k_s S)] + [(2\pi R_1 L d_2)/(k_{sn} LH)] + [(2\pi R_1 L)/(h_o LH)]}$$

$$(5.3)$$
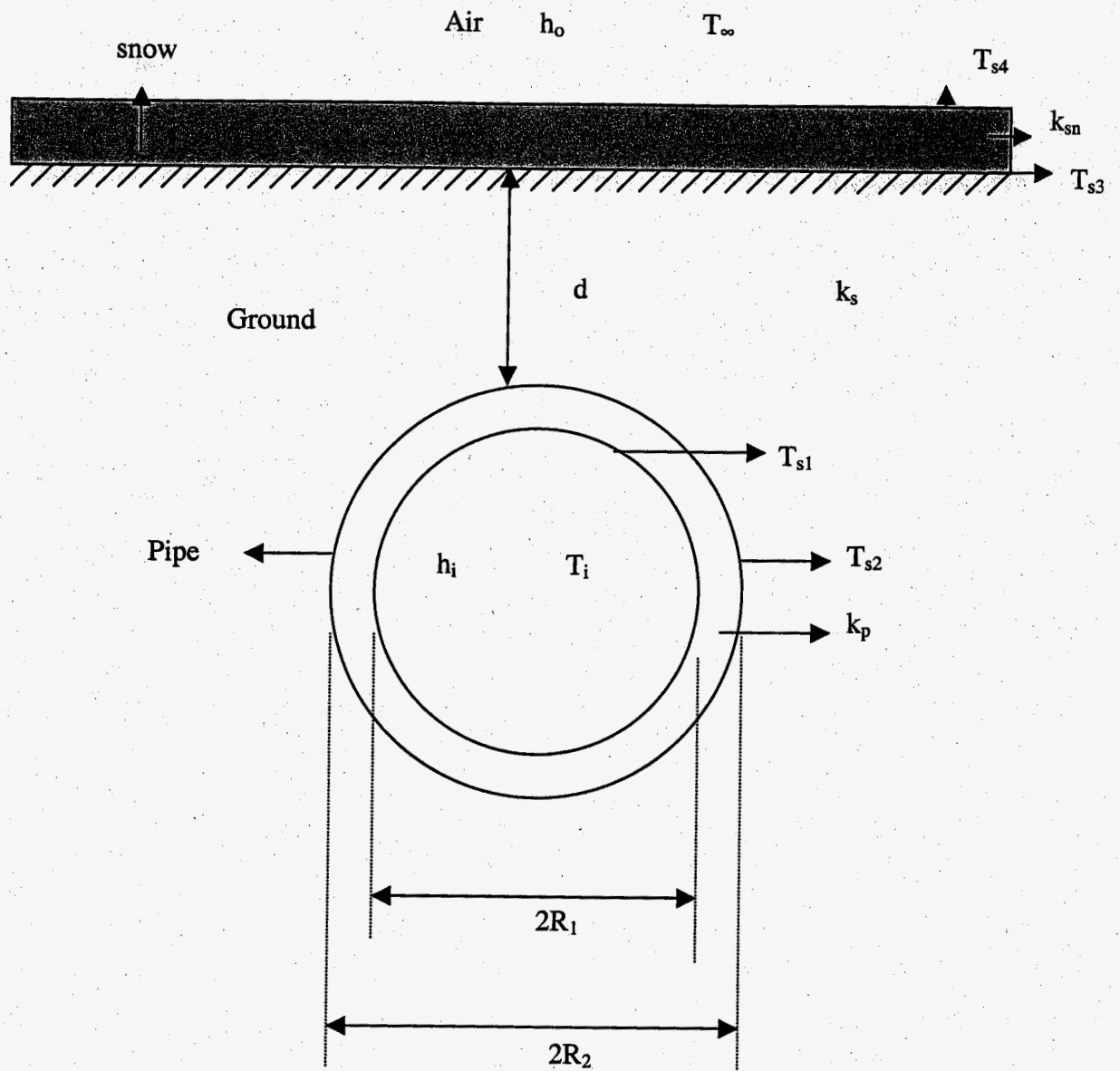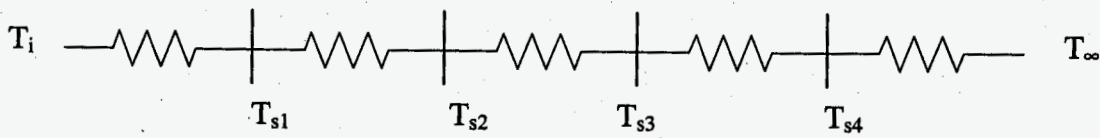
FIGURE 5.1. Below Ground Configuration of the Pipe Line

Conduction shape factor

For cases like a pipe buried underground, the temperature of the soil is two-dimensional and the resistance concept is applied by relating the heat transfer rate to the geometry and thermal conductivity of the soil medium. This is called the conduction shape factor and is denoted by S.

For a cylinder buried in a semi infinite medium the conduction shape factor can be determined from the equations presented in Suryanarayana (1995)

$$
S = \begin{cases}
\dfrac{2\pi L}{\cosh^{-1}(d/R)} & \text{if } L \gg R & (5.4.a) \\[3mm]
\dfrac{2\pi L}{\ln(2d/R)} & \text{if } L \gg R \,;\, d > 3R & (5.4.b) \\[3mm]
\dfrac{2\pi L}{\ln(L/R)\left[1 - \ln(L/2d)/\ln(L/R)\right]} & \text{if } d \gg R;\ L \gg d & (5.4.c)
\end{cases}
$$
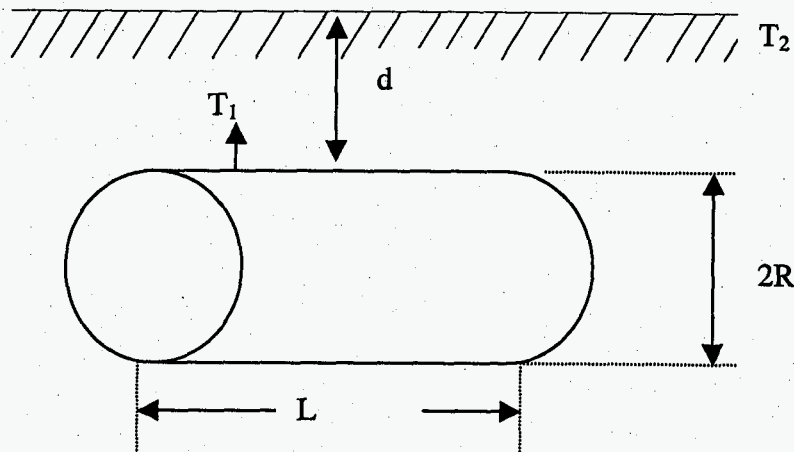
FIGURE 5.2 Pipe Orientation for Conduction Shape Factor

Resistance due to Pipe Wall:

Due to the cylindrical shape of the pipeline the resistance offered by the pipe wall is

$$\ln(R_2/R_1)/2\pi k_p L$$

Resistance due to the snow

Resistance due to the layer of the snow on the earth surface can be determined by

$d_2 / k_{sn} LH$, assuming heat transfer over fifteen diameters.
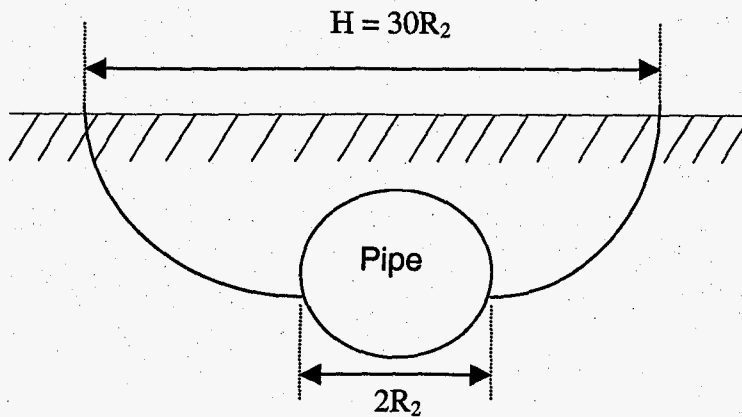
FIGURE 5.3 Assumed Zone of Heat Loss from the Pipe

Resistance due to convection

Heat transfer occurs between the oil and its boundary solid surface of the steel pipe wall. The heat flux from or to the solid surface is proportional to the difference between the surface temperature and a characteristic temperature of the fluid. The coefficient of proportionality is known as convective heat transfer coefficient.

The convective heat transfer coefficient when the heat transfer is from fluid to the surface can be determined by

$$h_i = \frac{Nu_d k_f}{2R_1} \tag{5.5}$$

The above equation is based on the inside diameter for cylindrical passage.
Where Nu is called the Nusselt number and k_f is the thermal conductivity of the fluid flowing through the pipeline, h_i is the internal convective heat transfer coefficient

For heat transfer from the snow in winter or bare surface in summer to the outside air, the convective heat transfer is given by

$$h_o = \frac{Nu_H k_a}{H}$$ (5.6)

The Nusselt number is based on the linear dimension H of a rectangular surface, h_o is the outside convective heat transfer coefficient and k_a is the thermal conductivity of air.

The equation to determine the value of the Nusselt number depends on various parameters like Reynolds number and Prandtl number. Reynolds number is the non dimensional number that is related to the motion of the fluid. It is the ratio between the inertia and viscous forces acting upon the fluid.

Reynolds number for internal flows is given by $\quad Re_d = \dfrac{\rho V d_1}{\mu}$

We can write mass flow rate as $\quad \dot{m} = \rho V \dfrac{\pi d_1^2}{4}$

which gives $\quad \rho V d_1 = \dfrac{4\dot{m}}{\pi d_1 \mu}$

Therefore, Reynolds number can be written as:

$$Re_d = \frac{4\dot{m}}{\pi d_1 \mu}$$ (5.7)

The above equation is written taking diameter as the characteristic length for cylindrical surfaces and V is the average velocity of fluid flowing through the pipe.

Depending on the value of the Reynolds number the flow is characterized as laminar flow or turbulent flow.

For flow inside pipes, if the value of Reynolds number is less than 2100 then the flow is characterized as laminar flow and if the Reynolds number is greater than 2100 then the flow is turbulent flow.

To calculate the Reynolds number for external flows V is replaced by V_∞ which is the ambient air velocity

Therefore Reynolds number can be written as $\quad Re_H = \dfrac{\rho_a V_\infty H}{\mu}$ (5.8)

The above equation is written taking length as the characteristic length for a rectangular surface.

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For external flows over rectangular surfaces, if the value of the Reynolds number is less than $5*10^5$ then the flow is characterized as laminar flow and if the Reynolds number is greater then $5*10^5$ then the flow is turbulent flow.

Prandtl number represents the relative effectiveness of molecular transport of momentum and energy within the hydrodynamic and thermal boundary layers.

$$Pr = c_p \mu / k_f \tag{5.9}$$

Where c_p is the specific heat of the fluid and μ is the coefficient of dynamic viscosity of the fluid and k_f is the thermal conductivity of the fluid, all at a particular temperature.

Depending upon the values of Reynolds number and Prandtl number for flow of the fluid inside the pipe, Nusselt number can be calculated and from Nusselt number the convective heat transfer coefficient can be determined. Equations of Nusselt number for internal flows can be found from Suryanarayana (1995).

For fully developed laminar flow $Nu_d = 3.66$ (uniform surface temperture) $\tag{5.10}$

$$Nu_d = 4.36 \text{ (uniform surface heat flux)} \tag{5.11}$$

For turbulent flows with entry length effects the equations are

$0.5 < pr < 1.5$; $2300 < Re_d < 10^6$; $0 < d/L < 1$

$$Nu_d = 0.0214 (Re_d^{4/5} - 100) \, Pr^{2/5} \, [1 + (d/L)^{2/3}] \tag{5.12}$$

$1.5 < Pr < 500$; $2300 < Re_d < 10^6$; $0 < d/L < 1$

$$Nu_d = 0.012(Re_d^{0.87} - 280) \, Pr^{2/5} \, [1 + (d/L)^{2/3}] \tag{5.13}$$

The hydrodynamic entry length equations can be found from Suryanarayana (1995).

For laminar flow $\quad L_e/d = 0.0565 \, Re_d$ $\tag{5.14}$

For turbulent flow $\quad L_e/d = 1.359 \, Re_d^{1/4}$ $\tag{5.15}$

The thermal entry length equations can be found from Suryanarayana (1995)

For laminar flow $\quad L_{e,th}/d = 0.037 \, Re_d \, Pr$ (uniform surface temperature) $\tag{5.16}$

$$L_{e,th}/d = 0.053 \qquad \text{(uniform heat flux)} \tag{5.17}$$

For turbulent flow $L_{e,th} = 10\,d$ (5.18)

For turbulent flow it is considered to be fully developed if $x/d > 10$, where x is the distance from the entrance.

The Nusselt number equations can be used for fully developed case by setting $d/L = 0$
Equations of Nusselt number for external flows over rectangular surfaces can be found from Suryanarayana (1995)

For Mixed flow (entry laminar flow and turbulent thereafter)

$$Nu_H = (0.037\,Re_H^{4/5} - 871)\,Pr^{1/3} \qquad 5*10^5 < Re_H < 10^7 \qquad (5.19)$$

$$Nu_H = [1.967\,Re_H\,(\ln Re_H)^{-2.584} - 871]\,Pr^{1/3} \quad 10^7 < Re_H < 10^9 \qquad (5.20)$$

For fully turbulent flows

$$Nu_H = 0.037\,Re_H^{4/5}\,Pr^{1/3} \qquad Re_{cr} = 0,\ Re_H < 10^7 \qquad (5.21)$$

$$Nu_H = 1.967\,Re_H\,(\ln Re_H)^{-2.584}\,Pr^{1/3} \qquad Re_{cr} = 0,\ 10^7 < Re_H < 10^9 \qquad (5.22)$$

By using the above equations (1) through (22) all the heat transfer parameters can be calculated.

Exit Temperature of the Fluid

Due to the heat loss, the fluid temperature must diminish in the direction of flow (Figure 5.4).

The exit temperature of the fluid from the pipeline can be determined by using the equation presented by Suryanarayana (1995)

$$\frac{T_\infty - T_e}{T_\infty - T_i} = \exp\left(\frac{-U_i A_i}{\dot{m}c_p}\right) \qquad (5.23)$$

$$T_\infty$$

Pipe and outer resistance

$$\dot{m} \qquad U_i$$

$$\dot{m}$$

$$T_i \qquad \qquad A_i \qquad \qquad T_e$$

T+ΔT   T
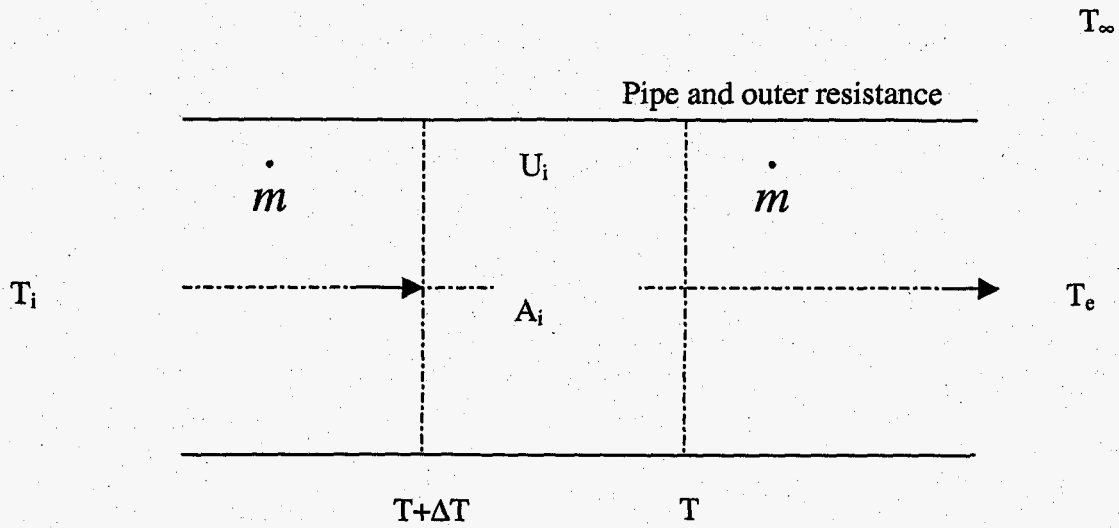
FIGURE 5.4 Relationship Between Fluid Inlet and Exit Temperatures and Overall Heat Transfer Coefficient

5.1.2 Above Ground Pipe Line

Figure 5.5 shows the configuration of the above ground pipeline.

The heat transfer rate for above ground pipeline can be determined by:

$$q = \frac{T_{iav} - T_\infty}{[1/(h_i \, 2\pi R_1 L)] + [\ln(R_2/R_1)/(2\pi k_p L)] + [\ln(R_3/R_2)/(2\pi k_i L)] + [1/(h_0 \, 2\pi R_3 L)]} \qquad (5.24)$$

Heat flux is defined as the heat transfer rate per unit area, which is given as

$$q'' = \frac{q}{2\pi R_1 L} = U_i (T_{iav} - T_\infty) \qquad (5.25)$$

Where U_i can be written based on the inner surface of the pipe

$$U_i = \frac{1}{[(1/h_i)] + [\ln(R_2/R_1)(R_1/k_p)] + [\ln(R_3/R_2)(R_1/k_s)] + [(1/h_o)(R_1/R_3)]} \qquad (5.26)$$

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The outside film coefficient h_o can be determined from the Nusselt number

$$h_o = \frac{Nu_D k_a}{2R_3}$$

(5.27)

Nusselt number for external flow over cylinders can be found from the equations summarized by Suryanaraya (1995)

$$Nu_D = 0.3 + \frac{0.62 \, Re_D^{1/2} \, Pr^{1/3}}{\left[1 + (0.4/Pr)^{2/3}\right]^{1/4}} \left[1 + \left(\frac{Re_D}{282000}\right)^{5/8}\right]^{4/5} \qquad Re_D > 400000 \qquad (5.28)$$

$$Nu_D = 0.3 + \frac{0.62 \, Re_D^{1/2} \, Pr^{1/3}}{\left[1 + (0.4/Pr)^{2/3}\right]^{1/4}} \left[1 + \left(\frac{Re_D}{282000}\right)^{1/2}\right] \qquad 20000 < Re_D < 400000 \quad (5.29)$$

$$Nu_D = 0.3 + \frac{0.62 \, Re_D^{1/2} \, Pr^{1/3}}{\left[1 + (0.4/Pr)^{2/3}\right]^{1/4}} \qquad Re_D < 20000 \qquad (5.30)$$

For all of the above equations, the product $Re_D Pr > 0.2$ must be satisfied.

In the previous three equations the Reynolds number is based on the wind velocity across the pipeline and can be obtained from the relation

$$Re_D = \frac{2V_\infty R_3 \rho_a}{\mu}$$

(5.31)

where R_3 is the outer radius of the pipe with insulation.

Air

T_∞

T_{s3}'

T_{s2}'

T_{s1}'

T_i h_i

h_o

k_i

Insulation

k_p

Pipe

$2R_1$

$2R_2$

$2R_3$
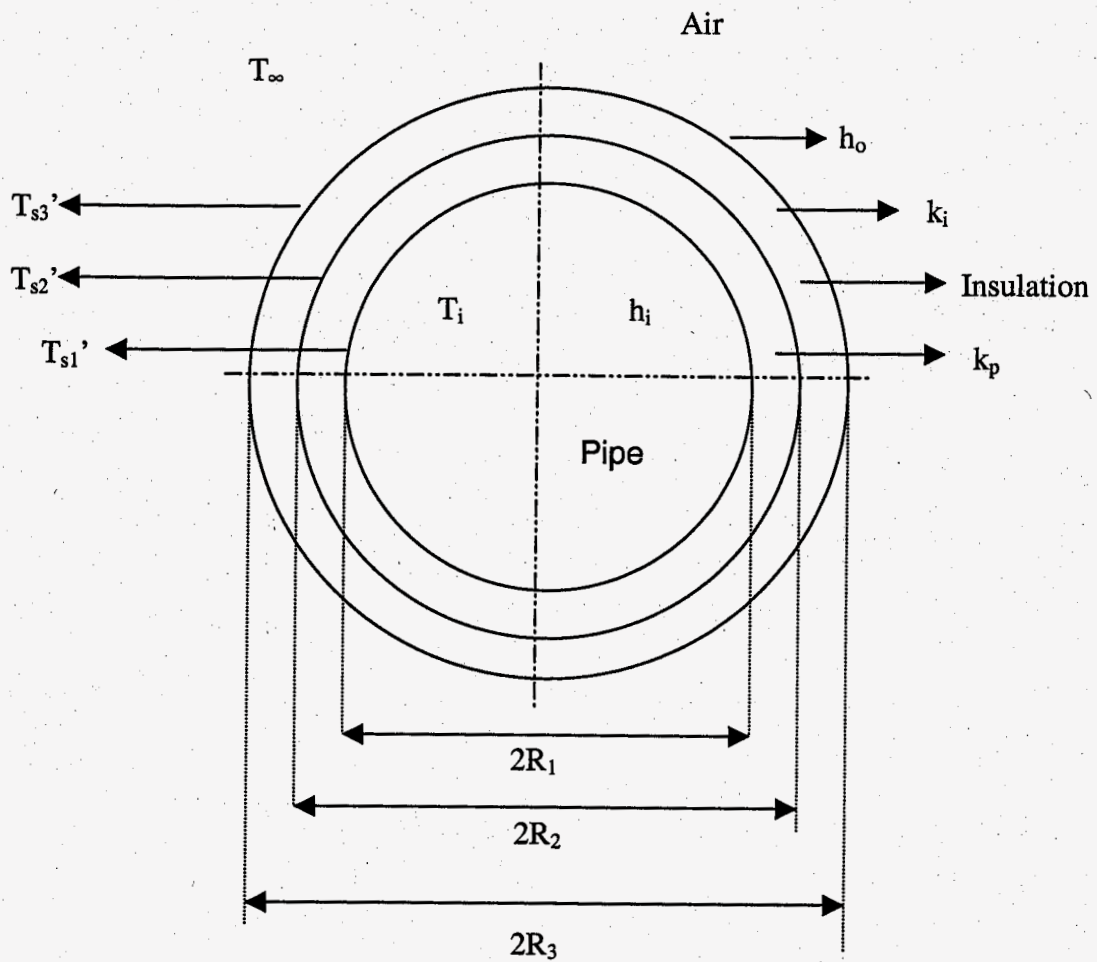
Thermal Resistance Circuit

Ti ——⌇⌇—|——⌇⌇—|——⌇⌇—|——⌇⌇—— T_∞

T_{s1}' T_{s2}' T_{s3}'
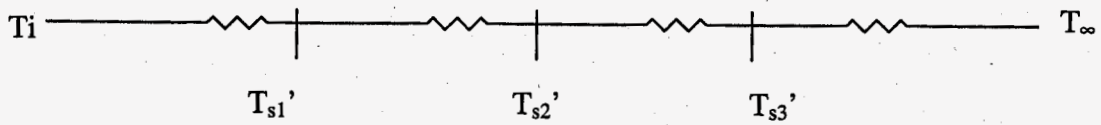
FIGURE 5.5 Above Ground Configuration of the Pipe Line

5.2 FLUID DYNAMIC ANALYSIS

Pressure drop in the pipeline is due to various factors like friction and hydrostatic head.

Pressure drop due to frictional head is given by White (1986)

$$\Delta p = \rho g h_f \tag{5.32}$$

where h_f is the head loss due to friction.

The head loss in terms of friction factor is given by White (1986)

$$h_f = \frac{f L V^2}{2 g d_1} \tag{5.33}$$

Where,
   f is called the Darcy friction factor,
   V is the average velocity of the fluid flowing through the pipe,
   d_1 is the inside diameter and
   L is the length of the pipe.

Friction factor f for turbulent flow depends upon Reynolds number and the roughness of the pipe. The friction factor f is given by Haaland (1983)

$$\frac{1}{f^{1/2}} = -1.8 \log \left[\frac{6.9}{\mathrm{Re}_d} + \left(\frac{\varepsilon / d_1}{3.7} \right)^{1.11} \right] \tag{5.34}$$

Velocity of the fluid flowing through the pipe is given by

$$V = \frac{\dot{m}}{\rho A}$$

Where A is the cross sectional area of the pipe which is given by $\frac{\pi}{4} d_1^2$ and $\dot{m}$ is the mass flow rate of the fluid flowing through the pipe.

The pressure difference due to hydrostatic head is given by

$$\Delta p_h = \rho g (z_2 - z_1) \tag{5.35}$$

The hydrostatic head is given by $(z_2 - z_1)$, where this term represents the elevation difference between two pump stations.

The pressure loss in pipe fittings between pump stations has been presented by Akwukwaegbu (2001)

$$\Delta p_m = \rho g h_m \qquad (5.36)$$

where h_m is the head loss due to pipe fittings.

The minor losses can be written as

$$h_m = k \frac{v^2}{2g} \qquad (5.37)$$

Where,

        k is a constant and

        v is the velocity of the fluid

h_m is constant for all fluids flowing through the pipe as the volumetric flow is taken to be 1.1 MMBPD, the velocities are the same for all the fluids. So head loss due to pipe fittings is constant.

Finally, the power required to pump the medium between the pipeline pump stations against these heads is given by Thomas (1993).

$$P = \overset{\bullet}{m} g[h_f + (z_2 - z_1) + h_m] \qquad (5.38)$$

5.3 PIPELINE SPECIFICATIONS AND FLUID PROPERTIES

The application of governing equations to determine the heat transfer and fluid dynamic parameters for the fluid flowing through Trans Alaska Pipeline requires the basic knowledge of the pipe specifications and the current operating conditions of the pipe. It is also important to know the properties of the fluid like viscosity, thermal conductivity, density, specific heat of the fluid as well as the properties of the air to determine heat loss to the surroundings.

5.3.1 Pipe Specifications

The heat transfer and fluid dynamic models work on certain basic information of the Trans Alaska Pipeline System (TAPS). The TAPS is an 800.302 miles long pipeline with 48 inch outer diameter. The pipeline is made of steel with thickness of 0.462 inches in some sections and 0.562 inches in other sections. The pipeline starts at Prudhoe Bay and ends at the Valdez terminal. Some sections of the pipeline are above ground and the other sections are below ground. The above ground sections have 3.5 inch thick insulation and the below ground sections do not have any insulation. The current crude oil flow rate in TAPS is approximately

1.1 million barrels per day (MMBPD). The pipeline has 12 pump stations (PS) numbered from PS1 to PS12, in which PS11 was deemed unnecessary and was never built. Pump station 5 is a relief station with no pumping capacity. Pump stations 2,6,8 and 10 have been placed in standby mode. Therefore the pipe is divided into 7 major sections for the purpose of thermodynamic calculation. These sections are listed below and a schematic of the pipeline along with the pump stations is shown in Figure 5.6.

    i)   PS1-PS3

    ii)   PS3-PS4

    iii)   PS4-PS5

    iv)   PS5-PS7

    v)   PS7-PS9

    vi)   PS9-PS12

    vii)   PS12-VALDEZ

Certain thermodynamic properties associated with the calculations don't show a significant change in the values with variation of temperature. Therefore, these are assumed to be constant throughout the length of the pipe as shown in Table 5.1

TABLE 5.1
Constant Properties

Thermal Conductivity of Insulation	0.0462 W/m K	(Chrisman, 2001)
Thermal Conductivity of Snow	0.19 W/m K	(Thomas, 1993)
Thermal Conductivity of Pipe	60.5 W/m K	(Suryanarayana, 1995)
Thermal Conductivity of Gravel	2 W/m K	(Andersland & Anderson, 1978)

FIGURE 5.6 The Trans Alaska Pipeline System and the pump stations

5.3.2 Fluid Properties

The crude oil is pumped from pump station 1 approximately at temperature of $115.7\ ^\circ F$ (Chrisman, 2001). As the oil flows through the 800 miles pipe, there is some heat loss from the oil to the surroundings. Because of heat loss, there will be a decrease in temperature, which in turn affects certain properties of the oil.
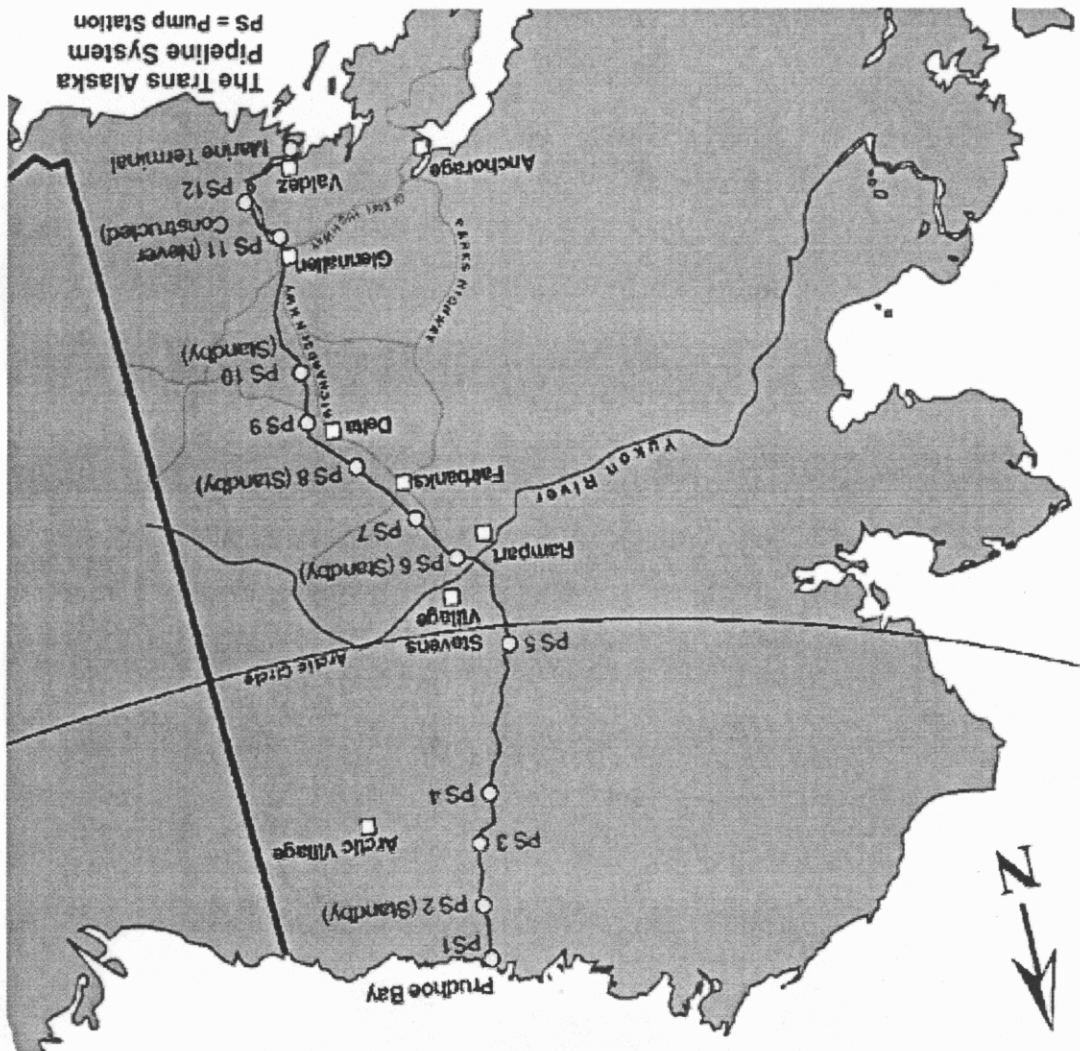
The properties of the fluid that are sensitive to the temperature change are:

   i)   Density of fluid
   ii)   Thermal Conductivity of fluid
   iii)   Viscosity of fluid

Since we will have to consider the flow of crude oil, GTL and a mixture of oil and GTL called commingled mixture properties variation for these three different types of fluids needs to be determined. Densities and viscosities of crude oil, GTL, and GTL-crude oil blends as a function of temperature are already described in Chapter 3. Thermal conductivities of these fluids are discussed below.

In the present study, three different types of fluids are considered: crude oil, GTL, and a 3:1 blend of crude oil and GTL. Variation of thermal conductivity of crude oil and GTL with temperature is shown in the following Table 5.2 (Dandekar, 2001).

TABLE 5.2
Variation of Thermal Conductivity with Temperature

Temperature	Thermal Conductivity (W/m K)	
(°F)	Crude Oil	GTL
120 (48.88 °C)	0.1418	0.1299
100 (37.78 °C)	0.1442	0.1325
80 (26.67 °C)	0.1466	0.1351
60 (15.56 °C)	0.1491	0.1377
40 (4.45 °C)	0.1503	0.1432
20 (-6.67 °C)	0.1529	0.1485

The thermal conductivity of the GTL-crude oil mixture can be determined by the mass average method.

$$k_{mix} = \frac{m_g k_g + m_o k_o}{m_o + m_g} \tag{5.39}$$

We know that one kg of mixture contains 0.7789 kg of crude oil and 0.2210 kg of GTL. Thus,

$$kmix = \frac{0.2210(0.1322) + 0.7789(0.1440)}{1}$$

$$k_{mix} = 0.14139 \text{ W/m K}$$

Variation of thermal conductivity of the fluids under consideration with temperature is shown in Figure 5.7.
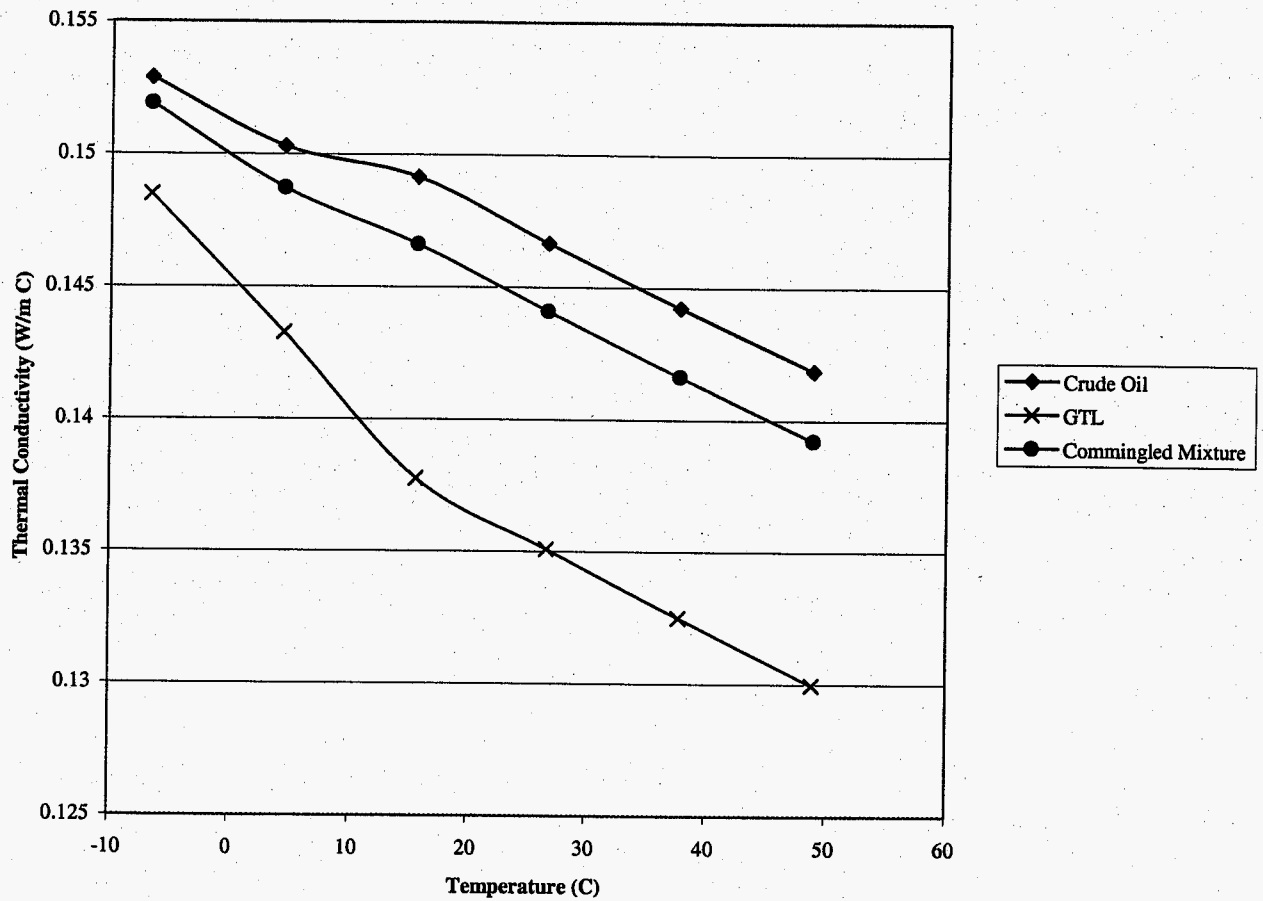
FIGURE 5.7 Variation of Thermal Conductivity of Fluids with Temperature

5.3.3 Properties of Air

The fluid flowing through the Trans Alaska Pipeline System loses heat to the outside air by convection. The outer convective heat transfer coefficient of air depends on Nusselt number and Reynolds number, which in turn depends on the viscosity and the density of air. The ambient air temperature along TAPS varies for different months and for different locations of the pipe. So the variation of the properties of the air needs to be determined with temperature.

The properties of the air that vary with temperature are:
i)   Density of air
ii) Viscosity of air
iii) Thermal Conductivity of air
iv) Prandtl number of air

The variation of properties of air at different ambient temperatures are shown graphically in Figures 5.8 through 5.11 (Suryanarayana, 1995).

Variation of Prandtl Number with Temperature
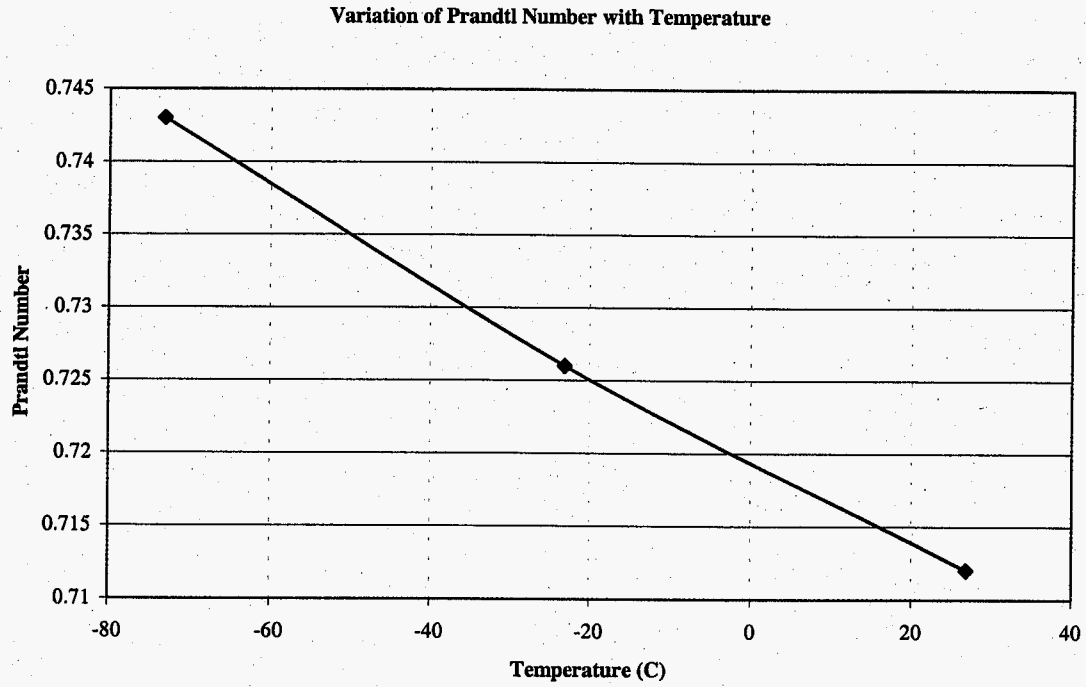
FIGURE 5.8 Prandtl Number Variation for Air With Temperature
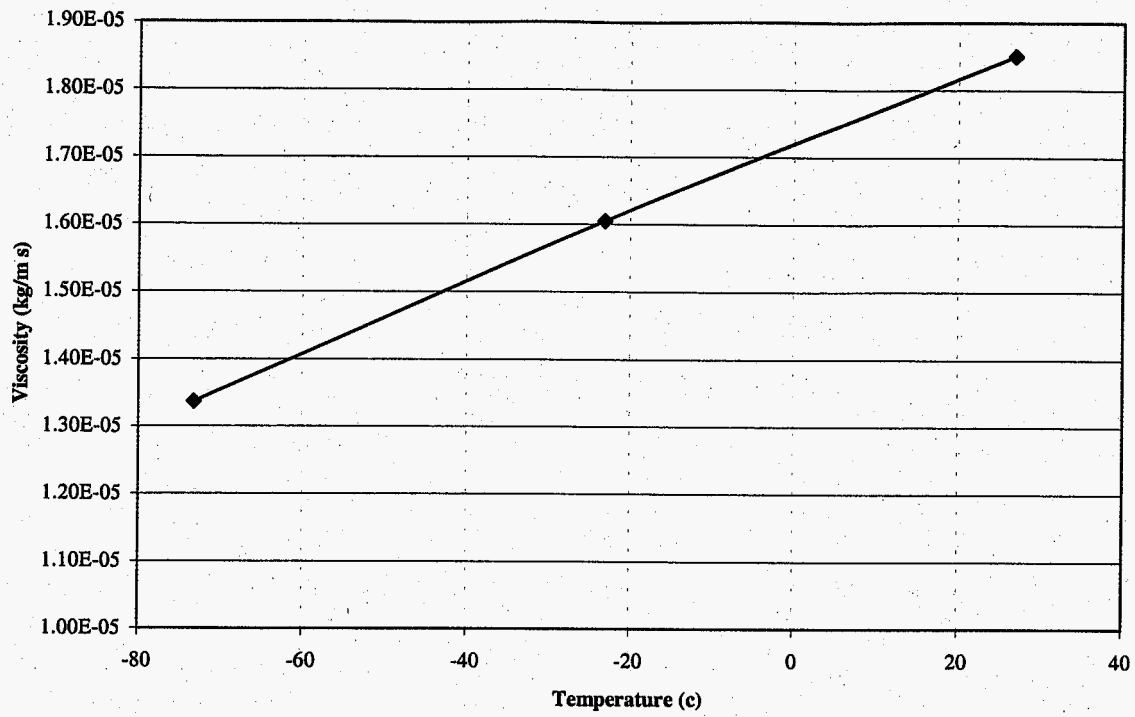
FIGURE 5.9 Viscosity Variation for Air with Temperature
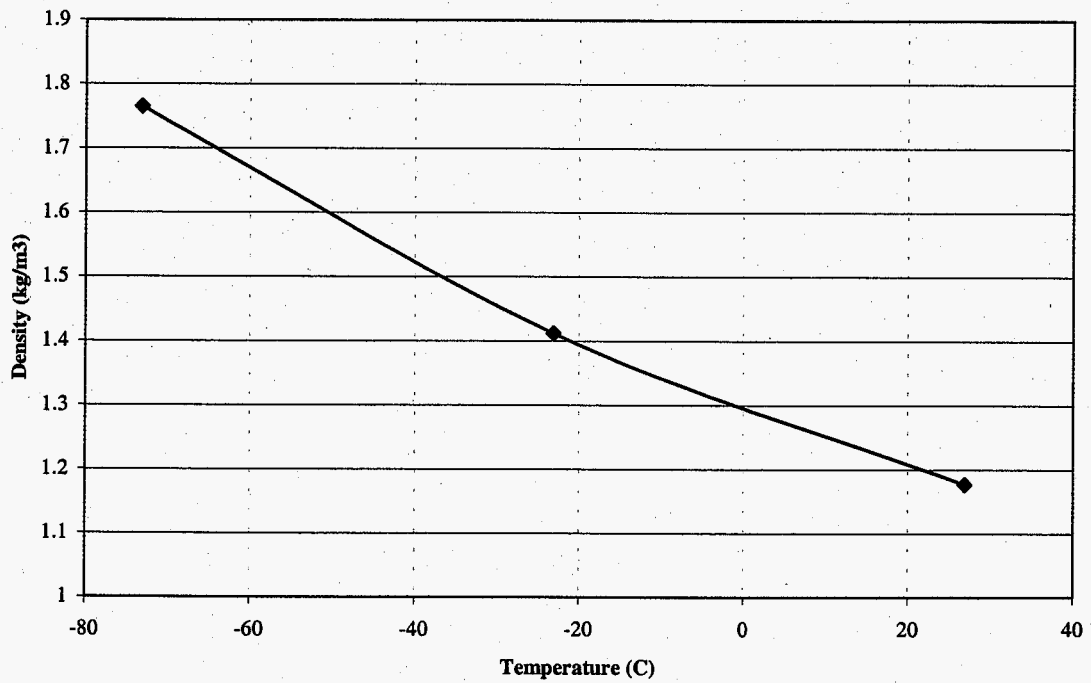
FIGURE 5.10 Density Variation for Air with Temperature
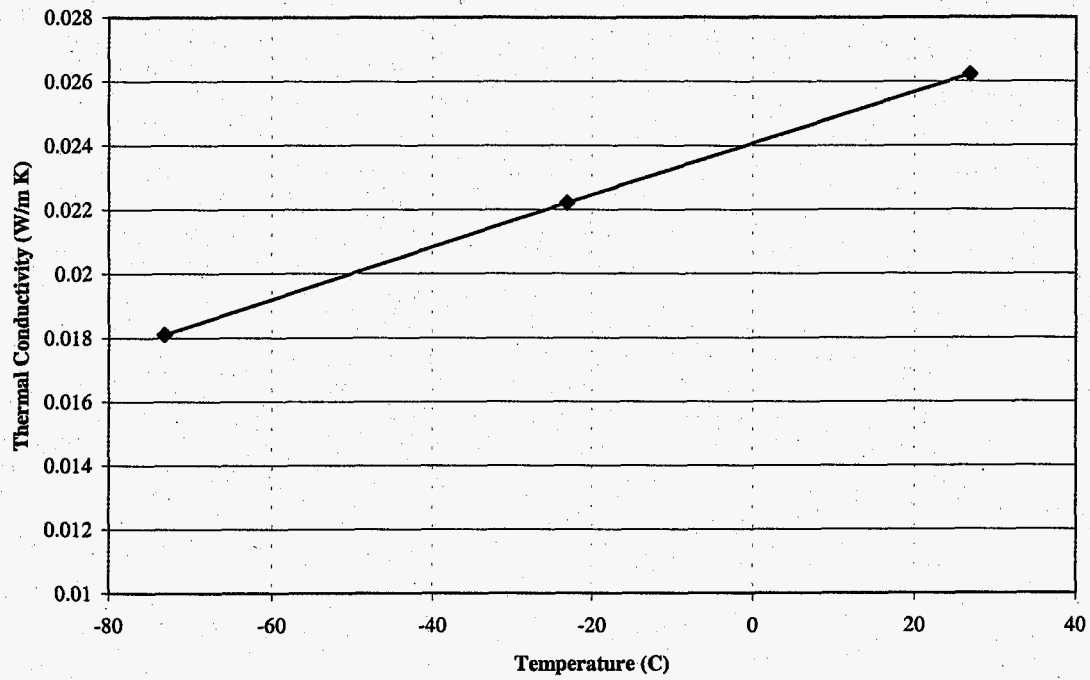
FIGURE 5.11 Variation of thermal conductivity of air with temperature

5.4 HEAT TRANSFER CALCULATIONS

To determine the heat transfer and fluid dynamic effects on the fluids flowing through the Trans-Alaska Pipeline System (TAPS), a computer program has been developed in Microsoft Visual Basic 6.0, giving links to Microsoft Access (for storing database) and Excel (spreadsheets) as the back ends providing a user friendly interface.

The design requirement of this algorithm is to calculate the required heat transfer and fluid dynamic parameters for different types of fluids (crude oil, pure GTL, commingled flow with various blends). This design is based on the concept that, some portion of TAPS is above ground and other is below ground. The total length of TAPS is 800.302 miles of which 420 miles of the pipe is above ground and 380 miles of the pipe is buried.

The program basically works on Microsoft Access and Excel, and works continuously unless the user manually quits the program and deletes the required files. The back end automatically opens the file and reads the data coming from the front end and the received data is stored as records in the form of tables in the memory of the program. The received data before storing in the records is converted into required data formats. The back end program keeps working till the front end quits.

The front end MS Visual Basic is a user friendly interface and works only on the commands of the user. The program stops working when there is no user entry or by any quit commands from the users. Depending upon the commands of the user it performs various operations and gives the required results.

5.5 RESULTS AND DISCUSSION

The heat transfer and fluid dynamic parameters are found by using the equations in Sections 5.1 and 5.2 for crude oil, GTL and commingled mixture . The results of these computations are summarized as follows.

5.5.1 Heat Transfer Parameters

The heat transfer parameters for unit length of the pipe are shown below. Three different types of fluids are considered, namely:

i)   100% Crude oil
ii)   100% GTL
iii)   Crude oil + GTL blend in 3:1 ratio

The results shown below are determined by assuming that 10 miles length of the pipe as buried and the adjacent 10 miles as above ground.

TABLE 5.3
Heat Transfer Parameters for Below Ground Pipeline

PARAMETERS	CRUDEOIL	GTL	COMMINGLED FLOW
Re_d (fluid)	350422	$1.6*10^6$	$4.12*10^5$
Pr (fluid)	68.75	13.95	57.42
Nu_d (fluid)	4326.16	8610.59	4632.45
h_i (W/m^2 k)	521.35	953.12	548.21
U_i (W/m^2 k)	0.91153	0.9122	0.91161
T_e (^{o}C)	45.236	45.020	45.188
q" (W/m^2)	68.14	68.09	68.12
q (kW) for 16.09 km	4119	4110	4115
q_1 (W/m)	255.95	255.39	255.70

TABLE 5.4
Air Parameters for below ground pipe

PARAMETERS	CRUDEOIL	GTL	COMMINGLED FLOW
Re_H (air)	$7.397*10^6$	$7.397*10^6$	$7.397*10^6$
Pr (air)	0.7271	0.7271	0.7271
Nu_H (air)	10430	10430	10430
h_o (W/m^2 k)	12.51	12.51	12.51

TABLE 5.5
Heat Transfer Parameters for above ground Pipeline

PARAMETERS	CRUDEOIL	GTL	COMMINGLED FLOW
U_i (W/m^2 k)	0.5543	0.5555	0.5544
T_e (^{o}C)	44.366	44.00	44.286
q" (W/m^2)	40.18	40.04	40.15
q (kW) for 16.09 km	2427.58	2419.19	2423.32
q_1 (W/m)	150.84	150.32	150.58

TABLE 5.6

Air Parameters for above ground Pipe

PARAMETERS	CRUDEOIL	GTL	COMMINGLED FLOW
Re_D (air)	$5.654*10^5$	$5.654*10^5$	$5.654*10^5$
Pr (air)	0.7314	0.7314	0.7314
Nu_D (air)	778.599	778.599	778.599
h_o (W/m^2 k)	12.217	12.217	12.217

5.5.2 Heat Loss from TAPS

The total heat loss from Trans-Alaska Pipeline System (TAPS) while transporting the three different types of fluid as mentioned above is determined for January, the coldest month of the year, and the results are tabulated as follows.

TABLE 5.7

Heat Loss from TAPS

Type of Fluid	Total Heat Loss from TAPS (kW)
Crude Oil	141793.51
GTL	132356.87
Commingled Mixture	141111.03

The above results are obtained for January with the temperature varying from –20°F to 10°F along the 800 miles length of the pipe. The wind velocity is taken as 10mph with a snow depth of 1ft on the ground. The variations in properties of the fluid and air are considered due to the changes in temperature along the length of the pipe. The results for all the three types of fluids are shown in a graphical form in Figure 5.12.

From Figure 5.12, it can be seen that for 100% Crude oil the cumulative heat loss from the pipeline is much more pronounced than in other cases. The density of GTL is much less than the density of Crude oil as the result of which the mass flow rate of GTL is less than that of Crude oil. The lower mass flow rate of GTL accounts for lesser heat loss from the fluid to the atmosphere. The commingled mixture is the combination of 75% of Crude oil and 25% GTL. The addition of 25% GTL to the Crude oil reduces the density of the mixture so results in the lower mass flow rate than Crude oil, which accounts for a relatively less heat loss from TAPS.

Cummulative Heat Loss From TAPS
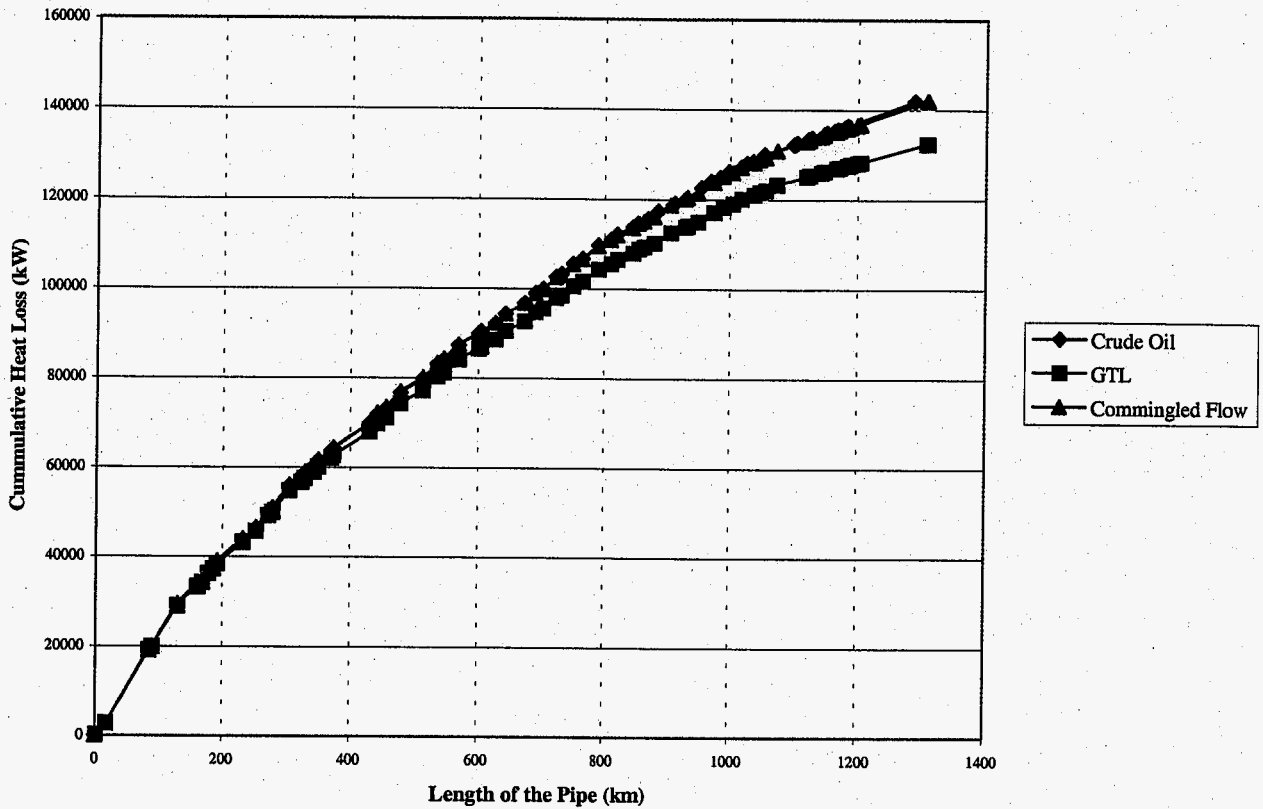
FIGURE 5.12 The Cumulative Heat Loss from TAPS from different fluids in January

The heat transfer from or to the body is given by

$$Q = \dot{m} c_p \Delta T$$

Where $\dot{m}$ is the mass flow rate

$\quad c_p$ is the specific heat of the medium

$\quad \Delta T \quad$ is the difference in temperature between two mediums

From the above equation it is clear that the mass flow rate is directly proportional to the heat transfer rate. The lower mass flow rate yields lower heat transfer rate and vice versa.
The specific heat is assumed to be same for the all the three fluids. So the lower heat loss from GTL is mainly because of lower mass flow rate.

5.5.3 Exit Temperature of the Fluid from TAPS

The exit temperature of the fluid at the terminal Valdez is determined for the all the three types of fluid and are tabulated below

TABLE 5.8 Exit Temperature of the Fluid from TAPS

Type of Fluid	Exit Temperature in °C
Crude Oil	0.877
GTL	-2.05
Commingled Mixture	-0.5

The above results are calculated for January with wind velocity of 10 mph and snow depth of 1ft. The exit temperature of the fluid leaving TAPS in the month of January is shown graphically in Figure 5.13.

Figure 5.13 shows that the exit temperature of GTL is less than that of the other two fluids. The lower temperature of GTL is because of its lower mass flow rate. Lower mass flow rate has lower heat content and cools down further. Lower mass flow rate is due to the lower density of GTL because of which the exit temperature of GTL is lower than the other two fluids. Since the density of crude oil is higher, the mass flow rate is higher and hence it has higher exit temperature than GTL and commingled mixture.

The mass flow rate is given by:

$$\overset{\bullet}{m} = volumertricflowrate * densityoffluid$$

Volumetric flow rate is taken as 1.1 Million barrels per day for all the fluids. As density of GTL is the minimum the mass flow rate is less for GTL.

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Exit Temperature of Oil Across TAPS
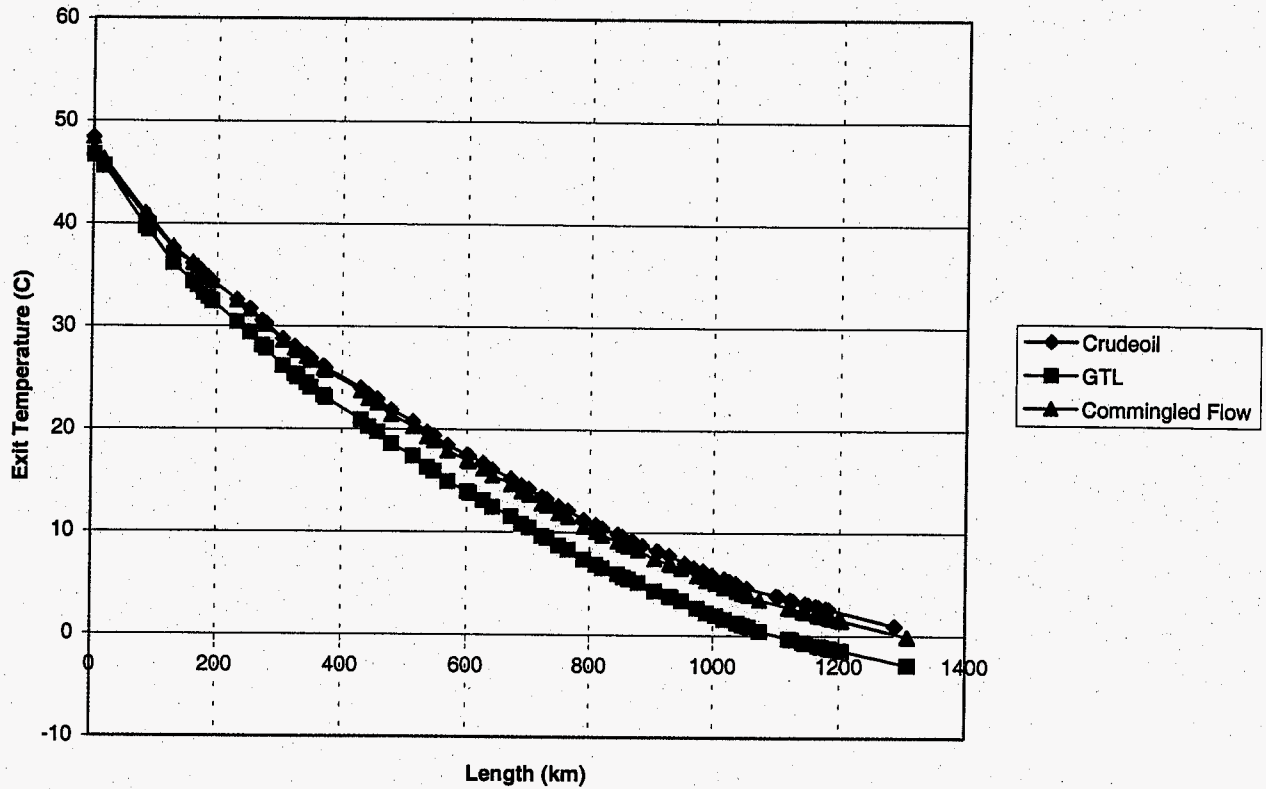
FIGURE 5.13 Exit Temperature of the fluid leaving TAPS in January

5.5.4 Exit Temperature of Fluid in Various Months

The exit temperature of the fluid leaving the TAPS is different in different months due to the variations in ambient temperatures. The exit temperatures of GTL and crude oil are calculated and shown in Table 5.9 for the four months of winter.

TABLE 5.9
The Exit Temperature of Fluid in Various Months

Month of the Year	Exit Temperature (°C)	
	Crude Oil	**GTL**
December	2.1	-1.4
January	0.877	-2.05
February	3.5	0.5
March	7.1	4.2

The above results are based on 10 MPH wind velocity and snow depth of 1ft. The exit temperatures of crude oil and GTL in the winter months are shown in Figures 5.14 and 5.15. Heat loss is maximum in January, which is due to the minimum air temperatures. Thus, because of the maximum temperature difference between the pipeline fluid and air in January, the exit temperature of the fluid from TAPS is minimum in January and increases towards March.
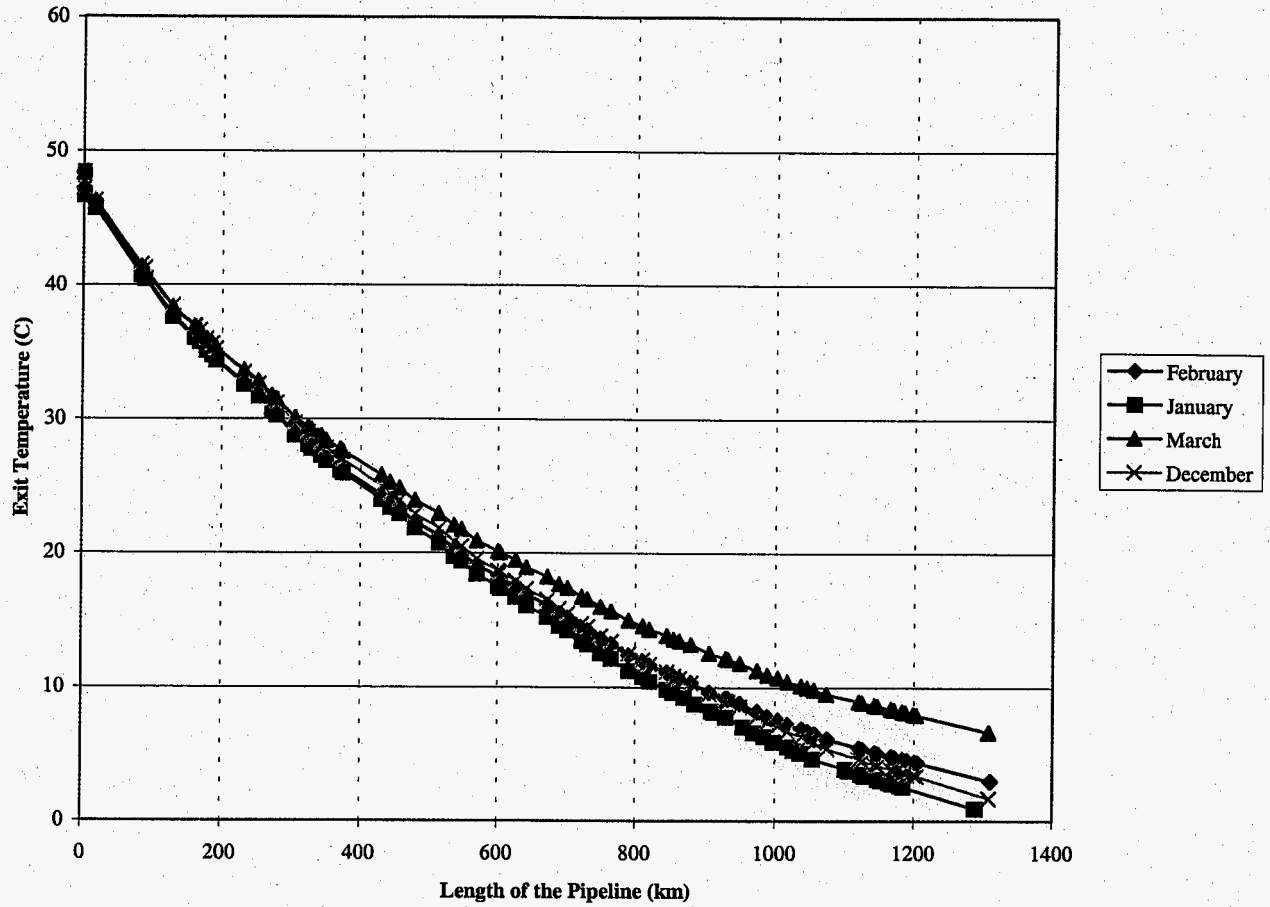
FIGURE 5.14 The exit temperature of the crude oil leaving TAPS in various months

Exit Temperature of GTL from TAPS
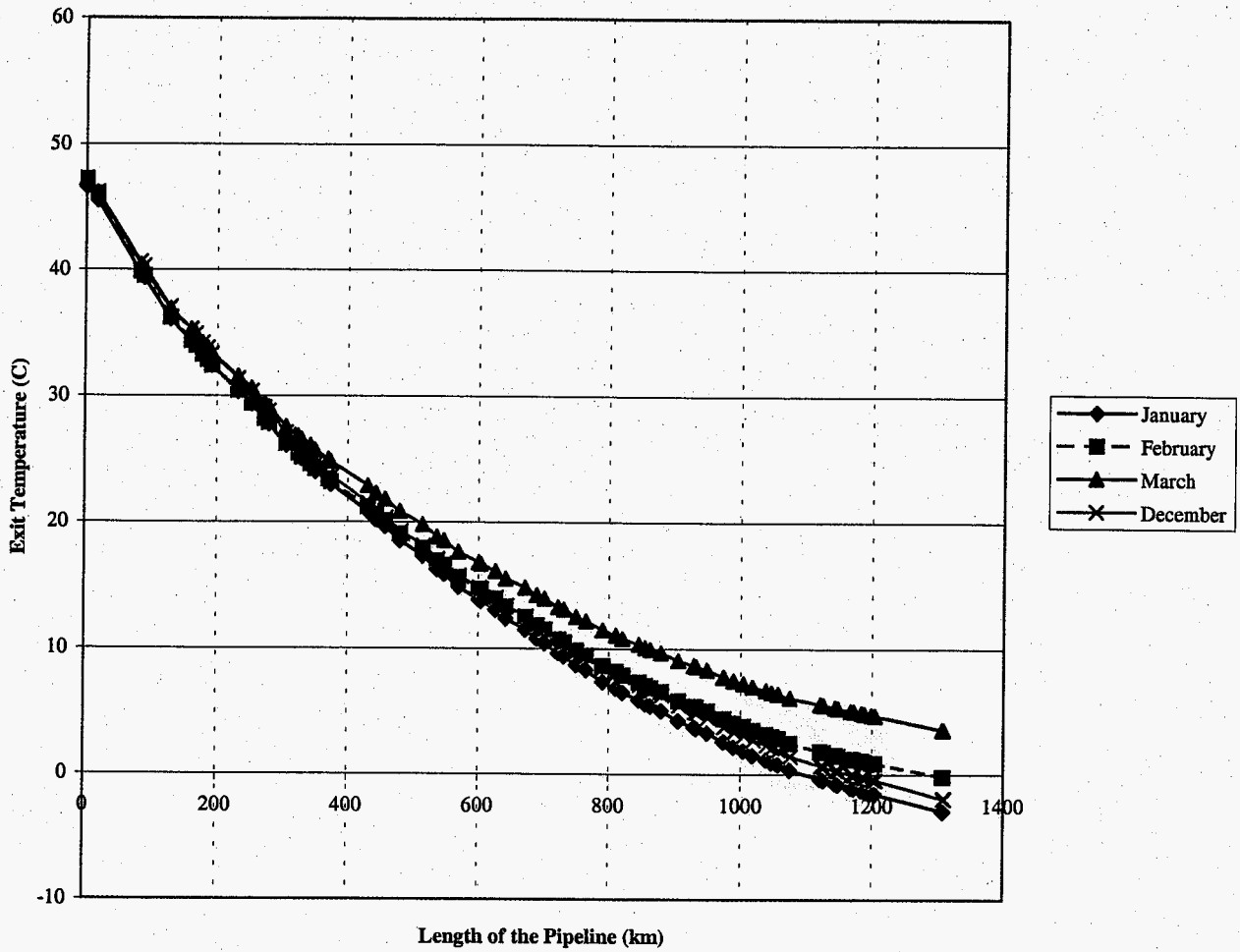
FIGURE 5.15 The exit temperature of GTL leaving TAPS in various months

5.5.5 Heat Loss from Aboveground and Belowground Sections of the Pipeline

The heat loss from the fluid to the ambient air for belowground sections of the pipe is given by

$$q = \frac{T_{iav} - T_\infty}{[1/(h_i 2\pi R_1 L)] + [\ln(R_2/R_1)/(2\pi k_p L)] + [1/(k_s S)] + [d_2/(k_{sn} LH)] + [1/(h_o LH)]}$$

The heat loss from the fluid to the ambient air for aboveground sections of the pipe is given by

$$q = \frac{T_{iav} - T_{\infty}}{[1/(h_i 2\pi R_1 L)] + [\ln(R_2 / R_1)/(2\pi k_p L)] + [\ln(R_3 / R_2)/(2\pi k_i L)] + [1/(h_0 2\pi R_3 L)]}$$

From the above equations we can see that the total resistance offered to the heat flow from belowground and aboveground sections of the pipe is different. Therefore the heat loss from belowground and aboveground sections will be different. The total resistance offered for the unit length of the pipe both for belowground and aboveground sections is calculated and shown in Table 5.10.

TABLE 5.10
Resistance Offered in Below Ground and Above Ground Section of the Pipe

Section of Pipe	Total Resistance Offered (m^2*k/W)
Belowground Pipe	1.1622
Aboveground Pipe	1.8316

The resistances are obtained for unit area and unit length of the pipe.

The resistance for the belowground pipe is less than the resistance for the aboveground pipe. So Heat Loss from the belowground pipe should be more than the heat loss from the above ground pipeline. The heat loss is determined for the unit length of the pipe for belowground and aboveground sections of the pipe for the month of January using wind velocity of 10 mph and is shown graphically in Figures 5.16 and Figure 5.17.

The heat loss from the entire 800 miles of the pipe of which 380 miles of the pipe is buried and 420 miles of pipe is elevated is determined. The amount of heat loss from the elevated and buried sections for the whole length of the pipe is determined and is shown in a tabular form in Table 5.11

TABLE 5.11
Heat Loss in Below Ground and Above Section of the Pipe

Section of Pipe	Heat Loss in kW
Belowground Pipeline	84292.74
Aboveground Pipeline	57500.76

The results in the above table are obtained with 10 MPH wind velocity, snow depth of 1ft and for January month. The heat loss in both the sections of the pipe is shown graphically in Figure 5.18.

Table 5.10 shows that the resistance offered to the heat flow for belowground section is much less than the resistance offered to the heat flow for aboveground pipeline. The resistance offered for the aboveground pipeline is nearly 36% more than that for the belowground section. The aboveground section of the pipe is 40 miles more than the belowground section. Due to the large difference in the resistance offered to the heat flow, the heat loss from the belowground section is higher than the heat loss from the aboveground section.

Heat Loss from Aboveground and Belowground Sections for Unit Length of the Pipe
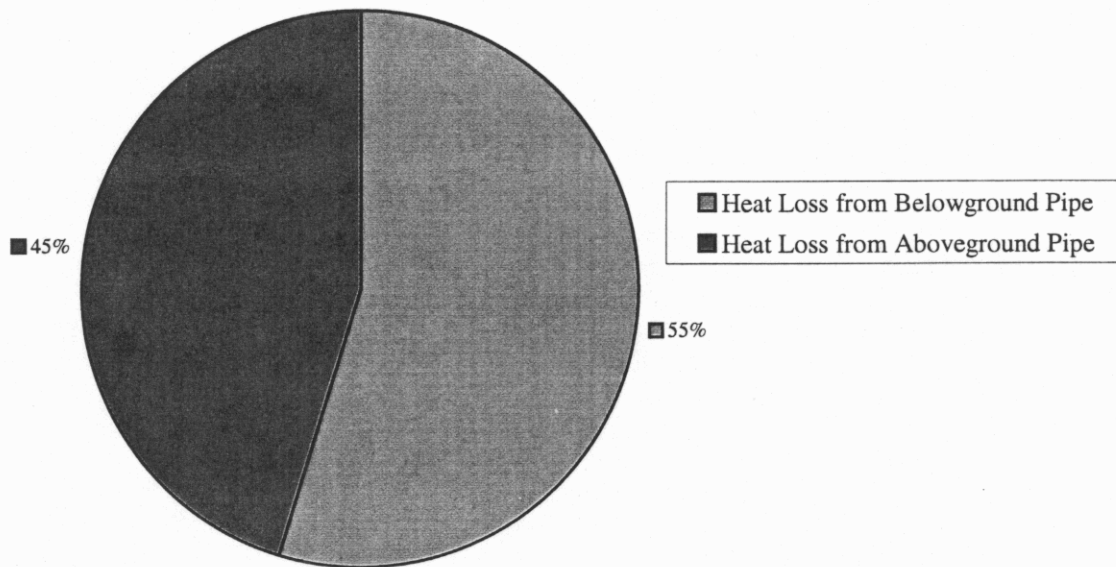
FIGURE 5.16 Heat Loss from Different Sections of the Pipe per Unit Length
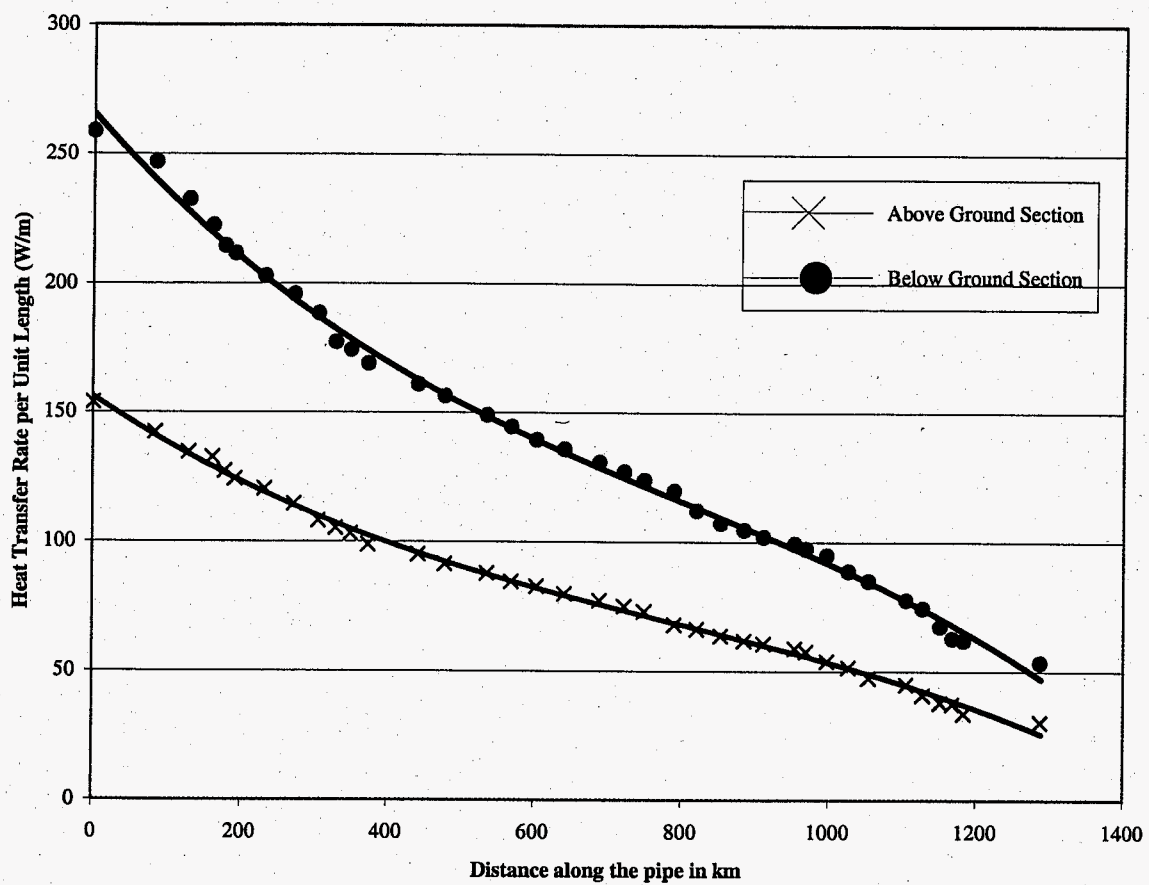
FIGURE 5.17   Heat Loss Rate from Above and Below Ground Sections of the Pipeline in January.

Heat loss in Belowground and Aboveground Portions for Crude oil in January
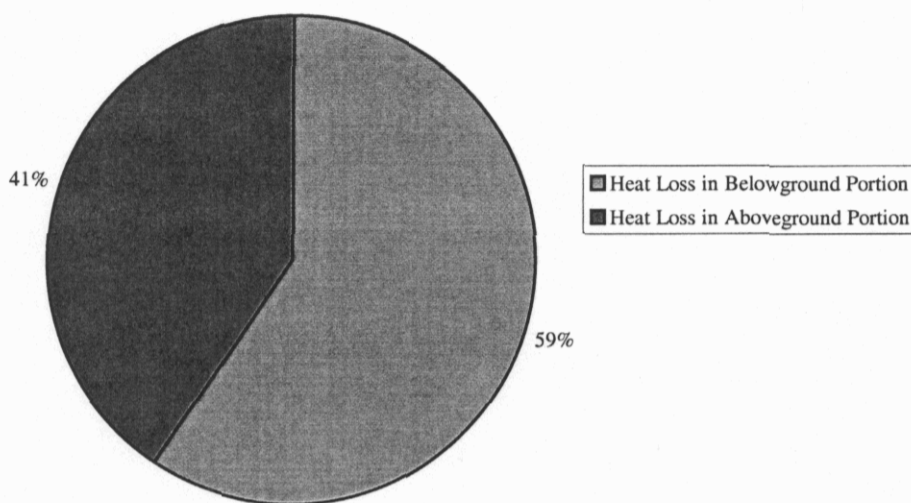
FIGURE 5.18
Heat Loss from Different Sections of the Pipe While Transporting Crude Oil

5.5.6 Comparison of Actual Data and Calculated Results

The 800 miles Trans Alaska Pipeline is currently under operation transporting crude oil from Prudhoe Bay to Valdez. The operating inlet and exit temperatures of the crude oil at different pump stations are known.

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The inlet and exit temperatures of the crude oil at working pump stations are shown in the Table 5.12 (Chrisman, 2000)

TABLE 5.12
Actual Temperature of Crude Oil at Various Pump Stations

Pump Station	Inlet Temperature	Exit Temperature
PS1		115.7
PS3	83.8	86.4
PS4	81.3	84.1
PS5	71.4	
PS7	66.5	70.3
PS9	72	74.9
PS12	64.4	64.7
Valdez	64.7	

The above temperatures are for a flow rate of 1.1 MBPD for the month of April.

By using the equations in Sections 5.1 and 5.2, and the Visual Basic program, the temperatures are determined and they are compared with the actual data. The comparison between the actual data and simulated data is shown graphically in Figure 5.19.

The simulated data are calculated for:

i)   1.1 Million Barrels per Day flow rate
ii)   10 MPH wind velocity
iii)   Snow depth of 1 ft
iv)   For the month April

The actual data graph shows an increase in the temperature of the fluid at around 400 miles, which is at pump station 7. The abrupt increase in the temperature of the fluid is due to the pumping problems in the pump station 7.

The simulated inlet and exit temperatures of the crude oil are shown in Table 5.13.

TABLE 5.13
Calculated Temperatures of the Crude Oil at Various Pump Stations

Pump Station	Temperature
PS1	115.7
PS3	98.99
PS4	94.32
PS5	80.99
PS7	71.99
PS9	64.78
PS12	57.16
Valdez	54.91

The differences in the actual and the simulated temperatures are due to:

i)   It is assumed that wind is blowing at a velocity of 10 MPH through the pipeline.

ii)   Snow depth of 1ft is taken as constant through the pipeline.

iii) The actual belowground and aboveground sections of the pipe are different from the simulated sections.

iv)   The variation in specific heat of crude oil is neglected with the change in temperature.

v)   Difference in the actual ambient temperatures and the simulated temperatures.

Comparision of Actual and Simulated Data
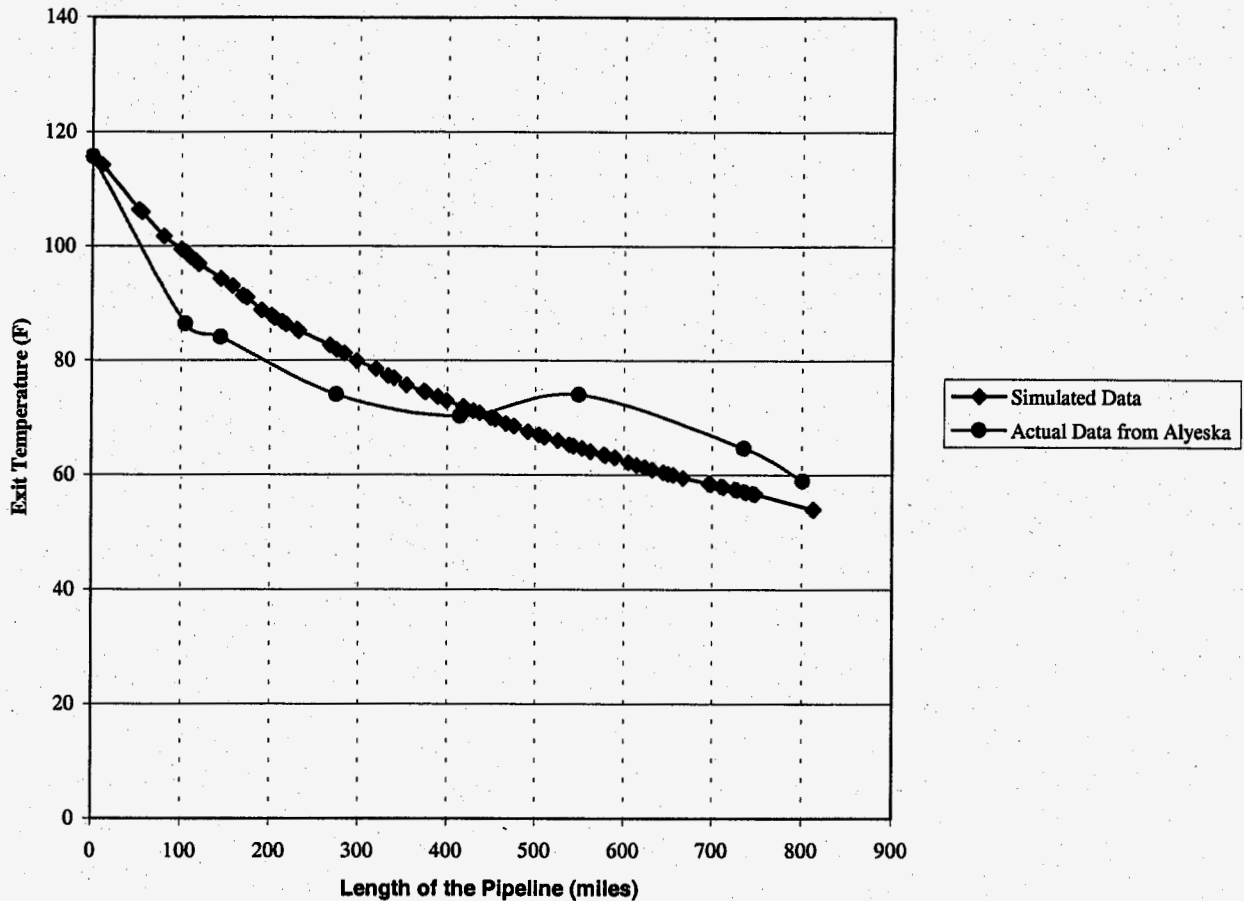
FIGURE 5.19 Comparison of Actual Temperatures with Calculated Results

5.6 CONCLUSIONS

The following conclusions are made based on this study.

1. Good agreement between calculated and measured oil temperatures is observed, proving the validity of our model.

2. Heat loss in below ground section is higher than heat loss in above ground section which is because of the absence of insulation in the below ground section.

3. Heat loss from GTL flow is less than heat loss from crude oil flow for both below ground and above ground pipeline. This is due to a reduction in mass flow rate for GTL.

4. Exit temperature of the GTL is slightly lower than the exit temperature of the crude oil. Lower mass flow rate has lower heat content and cools down further.

5. The heat loss is more in January than in March both for crude oil and GTL. The increased heat loss in January is because of the low ambient temperatures. The temperature difference between the fluid and the ambient air is greater which results in more heat loss.

6. The temperature of oil arriving at Valdez is 57°F for crude oil from the calculations. Temperature reported by Alyeska Pipeline Service Comapny is 60°F (Chrisman, 2001). Difference may be due to many assumptions made in the calculations, which may deviate from actual conditions.

NOMENCLATURE

A_i	Inside surface area of the pipe, m^2
c_p	Specific heat of the fluid, J/kg K
d	Buried depth of the pipeline below the ground, m
d_1	Inner diameter of the pipe, m
d_2	Thickness of the snow layer, m
f	Friction factor
h_f	Frictional head loss, m
h_i	Inside convective heat transfer coefficient, W/m^2 K
h_m	Head loss in fittings, m
h_o	Outside convective heat transfer coefficient, W/m^2 K
H	Width over which the heat is transmitted, m
k_a	Thermal conductivity of air, W/m k
k_f	Thermal conductivity of fluid, W/m K
k_i	Thermal conductivity of insulation, W/m K
k_p	Thermal conductivity of pipe, W/m K
k_s	Thermal conductivity of soil, W/m K
k_{sn}	Thermal conductivity of snow, W/m K
L	Length of the pipe segment. m
$\dot{m}$	mass flow rate of the fluid, kg/s
Nu_d	Nusselt number based on inner diameter
Nu_D	Nusselt number based on outside diamter
Nu_H	Nusselt number based on width
P	Power, kW (hp)
Pr	Prandtl number
Δp	Pressure drop due to friction, Pa
Δp_h	Pressure difference due to hydrostatic head, Pa
Δp_m	Pressure loss in fittings, Pa

q	Heat flow rate, W
q_1	Heat flow rate per meter, W/m
q''	Heat flux, W/m^2
Re_d	Reynolds number based on the inner diameter
Re_D	Reynolds number based on the outside diameter
Re_H	Reynolds number based on the width
R_1	Inner radius of the pipe, m
R_2	Outer radius of the pipe, m
R_3	Radius of the pipe with the insulation, m
S	Conduction shape factor for ground, m
T_{iav}	Average of inlet and outlet temperatures of fluid, °C
T_{s1}	Inside temperature of pipe wall below ground, °C
T_{s2}	Outside temperature of the pipe wall below ground, °C
T_{s3}	Temperature of the ground surface, °C
T_{s4}	Temperature of the snow surface
T_{s1}'	Inside temperature of pipe wall above ground, °C
T_{s2}'	Outside temperature of pipe wall above ground, °C
T_{s3}'	Outside temperature of the insulation above ground, °C
T_i	Inlet temperature of the fluid, °C
T_e	Outlet temperature of the fluid, °C
T_∞	Ambient air temperature, °C
U_i	Overall heat transfer coefficient with respect to pipe inner surface, W/m^2 K
V	Velocity of fluid flowing through the pipe, m/s
V_∞	Wind velocity across the pipeline, m/s
z_1	Elevation of the pump station 1, m
z_2	Elevation of the pump station 3, m
μ	Coefficient of dynamic viscosity of the fluid, kg/m s
ν	Coefficient of dynamic viscosity of the fluid, m^2/s
ρ	Density of the fluid, kg/m^3
ρ_a	Density of air, kg/m^3
ε	Roughness of the pipe, mm

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CHAPTER 6

ECONOMICS OF GTL TRANSPORTATION THROUGH TAPS

6.1 INTRODUCTION

One of the greatest potential options for using the Gas-To-Liquid (GTL) technology is the possibility of harnessing and utilizing the abundant natural gas resources on the Alaskan North Slope (ANS). The advantage of using ANS gas is that it is possible to transport liquids converted from gas by the GTL process along with the crude oil produced from the North Slope through the existing Trans Alaska Pipeline System (TAPS). Many operational challenges are expected in order to implement the project since the GTL products will be transported through the arctic environment from the North Slope to Valdez on Alaska south coast. A very high quality, contaminant free, product is produced by the GTL synthesis process. At the same time, the product can contain long chain paraffin, which are desirable for a diesel fuel but can pose potential flow problems in a cold environment.

The real challenges are: i) choose the GTL processing option to produce a transportable product, ii) get these products down through TAPS with minimal contamination from the crude oil, iii) simply blend the product with crude oil and transport through the TAPS. Each transportation option available has different economic impacts on the overall economics of the project depending on how much investment is made to keep the fuel as clean as possible at the terminal.

The two transportation options considered in this study are either to batch alternate slugs of the products or to transport the crude oil and the GTL as commingled fluids through the TAPS. The main focus of this section of the study is the impact of the transportation method chosen on the overall project economics.

6.2 GTL TRANSPORTATION MODES

In transporting GTL products through the Trans Alaska Pipeline System (TAPS), two modes of transportation are evaluated in this study. The choice of the mode of transportation to adopt depends on the expected purity of the shipped product and a trade-off between loss in product value due to contamination and cost of keeping the product pure at the terminal.

The first method considered involves blending the ANS crude oil and the GTL products. This method is termed the commingled mode of transportation. The Second method is to pump alternate slugs of the GTL products and crude oil through the TAPS, a batch mode. Batching of the product could be achieved in three different ways namely; the traditional batching technique called the batch mode A in this study, batching with physical barriers such as pigs and some other spacers called batch mode B, and a third technique which uses modern batching technology called batch mode C.

6.2.1 Commingled Mode

To discuss this mode of transportation, it is necessary to take a close look at the physical properties of each of the products to be blended. Generally, the crude oil blend from the North Slope of Alaska is a dark brown medium crude with an API gravity of about 32°, viscosity of about 17 cp at standard conditions with wax deposition tendencies at standard condition of temperature and pressure. Samples of GTL products from pilot plants show that they lie in the boiling range of middle distillates found in a typical crude oil. The GTL product has a viscosity of about 1.5 cp at standard conditions, typically diesel and naphtha based product with API ranging from about 62° for 354°C distillate to 66° for the 254°C distillate. The blending proportion of crude oil and GTL product on the North Slope is assumed to be a matter of availability of each of the product at any particular time rather than an intended ratio. However, with the commencement of the GTL project, the ratio is expected to continue changing depending on the amount of crude throughput available for blending assuming the GTL production remains constant. From the operational perspective, blends such as 3:1 crude oil to GTL with a resultant API of about 39.9°, 1:1 of crude oil and GTL with a resultant API of about 47° have been studied.

The flexibility of using existing infrastructure to the fullest advantage with minimal addition to capital cost for transportation is the most attractive aspect of this mode of transportation. This includes the use of the present holding tanks at the North Slope and storage tanks at the Valdez Marine Terminal, elimination of extra piping to the respective tanks at the Valdez Marine Terminal and minimal logistic concerns. At first glance therefore, it would be intuitive to tag this method as the most cost-effective. In the pump stations, pressure relief tanks are required for emergency operations. They are expected to come in as temporary storage in case of any unforeseen valve or process malfunction to cushion any pressure build up in the pipeline. The commingled transportation does not require this additional facility because the present relief tanks system is capable of handling the crude – GTL blend together.

The GTL economic model analyzes the effect of these initial savings on the entire project economics.

6.2.2 Batch Mode of Transportation

In the batch mode of transportation, the GTL products could be batched in three different ways:

> Uncontrolled batching of Products termed batch mode 'A'
> Controlled batching using pigs and spacers termed as batch mode 'B'.
> Controlled batching using modern batching techniques termed batch mode 'C'.

6.2.2.1 Batch Mode 'A'

The batch mode A or the as-is batching is considered the easiest of the batch modes of transportation. This mode of transportation requires minimal additions to capital and labor costs. Typically, any batch operation requires that there is segregated tankage for the GTL at NS and Valdez and clean tankers. Basically, the physics of the flowing liquid products (Crude

Oil and GTL) controls the behavior of the products while in the pipeline. This uncontrolled batching technique results in the creation of an interface zone in between the two phases (Crude Oil and GTL mixture). The length of this interface is a function of the viscosity, velocity and density difference between the two products, pipeline diameter and distance. Loss of product value due to contamination of the GTL products is at it's maximum in this mode when compared to other batching techniques. However, this is the common practice for refined products batching in the US. The interface generally gets downgraded to crude oil.
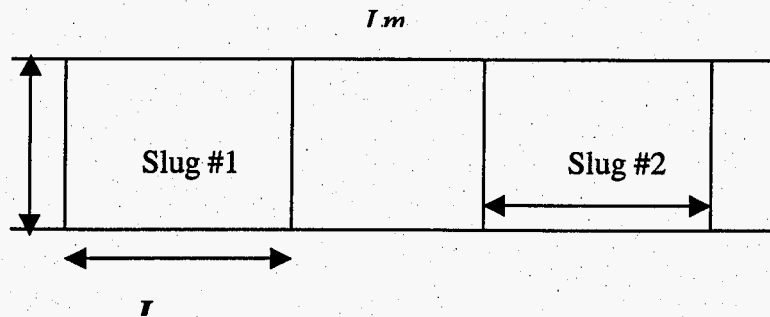
Figure 6.1: Typical Batch in TAPS

Equations have been derived for calculating the minimum slug length (please refer to Chapter 4), which can be translated to volumes for effective batching and minimal contamination for large diameter pipes. However, from the economic view point, it is pointed out that as slug length increases, segregated tankage requirements also increase. Further discussion on these is in the hydraulics section of this study (Chapter 4). In the case of Batch Mode "A" operations or any other batching technique, it is assumed that a special berth will be dedicated to GTL products at the Valdez Marine Terminal and special tankers will be used to carry only GTL products. This ensures that further secondary contamination does not take place beyond the Trans Alaska Pipeline System. This method of transportation is very similar to mode 'C' with the major difference coming from the employment of available technologies in the later to enhance purity of the transported products before, during and after transportation. The economics of this mode therefore forms a base case for the modern batching technique.

6.2.2.2 Batch Mode B

In this method, pipeline pigs are used to achieve the objective of phase or slug separation. It is expected that these pigs will effectively prevent mixing of the alternate slugs of GTL products and crude oil. This method requires the entire basic infrastructure that is used in the base case batch mode plus some additional capital. The GTL products will be stored in separate tanks both at the North Slope and in Valdez Marine Terminal. Transporting the products through the Trans Alaska Pipeline System would not be left to fluid dynamics to govern their movement in the pipeline since the pigs would keep the oil and GTL separate. The number of slugs expected to be in the pipeline at any time would determine the number of pigs required. Intelligent pigs with sensors attached to them are commonly available with capabilities of detecting product

movement. Detecting product movement can minimize the time needed to divert flow from crude oil tank to GTL storage tanks.

A major operational challenge arises here. The pigs need to be diverted at every pump station along the TAPS where flow is diverted accordingly for the fluid to pass through the pumps. Additional labor is required at each pump station to carry out this task on a continuous basis.

Most operators are of the view that the use of pigs to batch these products would reduce or completely prevent the mixing of the products. Some other operators believe that batching with pigs would not reduce the mixing but rather increase the mixing due to increased turbulence. This task is currently under study. In this analysis, the assumption is that the pigs would help maintain product purity. Possibilities exist for improvement in pigging technology as deemed necessary. For instance, it is possible to attach sensors to the pigs that would enable automatic diversion of flow at the pump stations. The opening and closing of valves can fully be automated at the pump stations with good instrumentation and controls. Pigs are also available that are specifically designed for batching of petroleum products.

6.2.2.3 Batch Mode C

The modern batching technique, identified here as batch mode 'C' entails pumping alternate slugs of GTL and Crude oil while having fluid movement monitored by interface detection devices to minimize loss of product value. Available interface detection technologies include densitometers, sound-velocity interface detectors, colorimeters, pipeline interface detectors and photo detectors (Baum et al., 1998).

A densitometer measures online the specific gravity of the product in the pipeline, and can detect even small changes in product density. In terms of accuracy, they can distinguish between premium and regular gasoline.

The sound velocity interface detector employs changes in the sound velocity rather than changes in density to detect different liquids.

A colorimeter detects color changes in the contents of the pipeline. It measures color quality with a dual wavelength, dual detector optical system. At the receiving terminal, which could be at a refinery or tank farm, a dynamic hydraulic model for optimizing control of product movement and a Distributed Control System (DCS) can optimize control of product movement. A DCS allocates the crude oil and GTL to their respective tanks at the optimal time reducing error in valve opening and closing and this is synchronized with the pumping at the plant end, employing already calculated optimum slug length from batch mode 'A', length of interface, the interface and slug velocities. This method would require additional storage tanks and shipping facility to ensure that purity of the products is maintained.

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6.3 ECONOMIC PARAMETERS

In order to conduct the economic analysis of GTL transportation through TAPS, the following economic parameters are considered in this study:

- A large-scale GTL project consisting of three trains each capable of producing about 100,000 barrels of GTL products per day.
- Pipeline Tariffs obtained from available forecasts and charged based on throughput and is expected to pay for the pipeline, pipeline maintenance and storage cost at the terminal and some return on investment.
- A salvage value of zero.
- Each mode of GTL transportation has an associated capital cost which varies from minimal capital investments for the commingled mode to huge capital costs for the modern batching approach.
- Construction starts in 3 years by 2005, lasts 4 years through 2009 and production begins same year.
- Train 2 construction commences after train one has started production and train 3 commenced two years after train 2 is started to spread out the investment.
- All transportation costs rely on the existing infrastructure of the oil pipeline operation and maintenance, therefore; only additional capital costs specific to GTL will increase the cost.
- Discount rate of 10% is used for the capital costs
- Depreciation of property is by Modified Accelerated Capital Recovery System (MACRS).

6.3.1 Rate Of Return Analysis

Rate of return analysis was used in evaluating the various transportation modes. To conduct rate of return analysis, an accurate estimate of the capital and operational costs involved in the project was necessary as well as an estimate of expected product price and revenue. The project life was estimated to be twenty years. Based on construction costs, the construction schedule, the timing of product sales, and the expected revenue a rate of return was estimated. It is assumed that 100% equity financing is used which is typical for oil and gas firms when comparing different projects.

6.4 IDENTIFYING CAPITAL AND OPERATING COSTS

The capital costs include all costs from the GTL plant to the delivery of product onto the GTL tankers in Valdez.

The first common cost to all modes of transportation is the contingency plan capital. No production of GTL can begin until this capital is in place. Contingency plan capital refers to the capital that must be set aside to help handle emergency situations that might lead to shutting down the pipeline and ensure quick restart of operations. Laboratory measurements show that in the arctic environment a window of about thirty (30) days is allowed to restore operation in case of any shutdown during the winter season or risk shutting down operation once the crude oil in the pipeline gels due to very cold temperatures. The window for cold restarts in the winter

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for the pipeline when GTL is in the pipeline for either the commingled mode or the batch mode is estimated to be smaller compared to when only crude oil flows through the pipeline. This is given adequate treatment in the gel strength prediction section of this report for both a fast and slow ramp cooling process.

Another common cost to all modes of transportation is the cost of building a pipeline from the GTL plant to Pump Station 1. It is assumed that the GTL plant would be situated not more than one mile for pump station one.

The piping cost required to transporting the gas from the production wells and stations are not included in the GTL project cost. The drilling and completion cost of the gas wells are also not part of the GTL cost here but are assumed to be a part of the gas purchase cost. The GTL plant is assumed to come with a conditioning unit for removal of acid gases such as CO_2 and H_2S and are therefore not considered separately.

On the distinctive capital costs, batch mode 'A' does not incur any extra costs apart from that outlined above. Batch mode B incurs additional capital costs in purchasing pigs and labor to handle the pigs on a continuous basis at different pump stations.

Batch Mode 'C' requires additional investment costs from those outlined above including interface detection devices for minimizing impurities associated with mixing, product movement control devices that use the Distributed Control System (DCS), densitometers, colorimeters, and other complex instrumentation. This technology has been proven effective and has been used extensively by the petroleum products transportation industry in pipelines. Product movement control has two main components. The first is the dynamic hydraulic model and the second is a Distributed Control System (DCS). This system is complex and expensive.

6.4.1 Plant Cost

The capital cost of a GTL plant is estimated at between $25,000 per daily barrel (DBL) capacity and $35,000/DBL (Thomas et al., 1996). Current industry average for a US gulf coast plant puts the capital cost at about $24,000 / DBL. Most of the plants from which these cost estimates were derived are small-scale GTL plants with design capacity of between 30,000 to 50,000 barrels per day (bpd). As technology advances, these costs are expected to come down significantly. One such significant leap is the reduction in the size of the steam-reforming unit to about forty times less than the conventional size of the steam reformer. This is projected to result in a significant change in the capital cost for GTL plants. This is estimated to put the capital cost at about $20,000/ DBL for a commercial scale plant in the Gulf Coast. This compact reformer technology is currently being tested with a pilot plant in Nikiski, AK by BP Exploration (Alaska) Inc. The reformers come in compact units built to commercial scale. To increase output, additional whole compact reformer units are added to operate in parallel with existing ones and minor modifications made to other units in the plant to increase plant output capacity.

The Alaskan North slope is assumed to have a cost scaling up factor of about 1.3-1.5 times the cost of building the same plant in the Gulf of Mexico. If the compact reformer technology

passes the test to commercial status, then the capital cost of the plant on the North Slope is anticipated to be at about $28,000 /DBL capacity assuming the same plant is built at a cost of about $20,000 /DBL in the Gulf Coast. However, this study evaluated a wide range of capital costs of GTL plants from $35,000 /DBL down to $20,000 /DBL.

Application of the learning curve as presented by Robertson et al (1999) was not employed in this study. Cost improvement based on a learning curve or progress curve plays a crucial role in the competitiveness of the chemical and petrochemical industry. It has been observed that more rapid cost improvement for a product results in expanding market share and profits. Though initial or pilot projects may be economically marginal, expectations of rapid cost improvement based on a learning curve is often the motivator to invest in such projects. As GTL technology unfolds and operators gain experience from building and operating earlier trains, a rapid cost improvement is expected. This is usually represented by a common rule of thumb based on observations from petrochemical plants as;

$$C_n = C_1 n^b$$

Where,

C_n = Cost of the nth unit,
C_1 = Cost of the first unit,

n = number of unit being estimated and
b = exponent equal to the improvement – curve rate divided by the log of 2

Cost improvement rate for organic chemical production was found to be 73.8 percent on the average. GTL plant falling under the same industry, would have the 'b' exponent given by:

$$b = \frac{\ln 0.738}{\ln 2.0} = -0.4383$$

In the learning curve advantage as presented, one or combinations of factors presented below are expected to play important roles in driving down cost of subsequent trains:

Learning by plant operators and designers
Technical improvement
Economies of scale
Probable decrease in cost of raw (feedstock) material

The scenarios presented assume that the capital cost remains the same in all the trains and this is the worst case possible since capital cost improvement would be significant in the second and third trains. As noted above, the Prudhoe Bay gas has a high carbon dioxide content and needs to be conditioned before it is fed to the GTL plant. The above cost is expected to cover the gas conditioning.

6.4.2 Storage, Product Separation and Other Costs

For the batch mode of transportation, it is assumed that three new holding tanks (APSC 2002) would be built on the North Slope. To arrive at this, the present tanks have a holding capacity per foot of 4,400 barrels. For an estimated 300,000 barrels per day of Gas-To-Liquid product conversion plant, the footage of temporary storage required would be given by:

$$\frac{300,000}{4,400} = 68.18\,ft$$

Maximum allowable height by OCC (Operational Command Center) is approximately 32 ft and an 8ft minimum level maintained, leaving out only 24 ft (APSC April 2002).

$$\frac{68.18\,ft}{24\,ft} = 3\,StorageTanks$$

Each of the tanks is estimated to cost about $50 million. This estimate includes fittings, accessories, piping and refrigerated foundation.

At the Marine Terminal in Valdez, a first case where new tanks are built for storage of GTL is considered first. This represents the worst-case scenario. For a one-week storage capacity, four new tanks are required at the Valdez Marine Terminal where the tanks are 500,000 barrels capacity each. The cost of these four tanks is estimated at approximately $270 million or $65 million each. This cost is expected to cover some fittings and appurtenances such as; pressure relief valves, emergency relief vents, tank piping, mixers, internal heaters, water draw-off valves, tank instrumentation, tank insulation, thief hatch, corrosion control. Another option is to recondition and reconfigure four of the 18 existing tanks for GTL storage. This is an optimistic assumption. The cost of reconditioning and reconfiguring each of the tanks is put at approximately $5 million dollars so about 20 million dollars is estimated to recondition the four tanks.

Emergency relief tanks for GTL at pump stations 3, 4, 5, 7, 9, and 12 are required. Building these tanks is another major cost in the transportation model. These emergency relief tanks are 55,000-barrel capacity tanks. To maintain GTL product purity, each of the pump stations may require a separate emergency relief tank for GTL products. Each of these tanks is estimated to cost about $16 million bringing the cost for all the pump stations mentioned above to $96 million. In the second and optimistic scenario, it is assumed that the emergency relief tanks will not be required since such emergency operations are only very occasional. The present emergency tanks are therefore assumed sufficient to handle the situations as long as they are kept clean and ready to receive any products in case of emergency.

For batching of products with pigs, by applying the optimum length of slug for batching of product, the number of pigs required is obtained and the cost added to the cost of batching with pigs. Labor is required to handle these pigs at the pump stations. This is also accounted for in the economic analysis of this mode of transportation.

Vapor pressure estimates from the laboratory show that the vapor pressure of GTL products is within the acceptable limits and can be handled by the existing vapor pressure recovery system. Further study of the vapor pressure of GTL products are also in progress. The vapor pressures are required for live GTL products from the plants under pipeline conditions of pressure and temperature to obtain the true behavior of the GTL products in pipeline conditions. However, some piping modification will need to be done and together with all other piping jobs to the tanks, an estimate of $10 million dollars might be required.

Table 6.1
Capital Cost Schedule For the Various Modes of Transportation

SUMMARY OF CAPITAL COST ESTIMATES FOR DIFFERENT MODES OF TRANSPORTATION						
No	Item	Estimated Cost each ($mm)	Batch Mode A	Batch Mode B	Batch Mode C	Commingled
4	Tanks @ Valdez	65	260	260	260	0
3	Tanks @ Slope	65	195	195	195	0
6	Pressure Relief Tanks	16	96	96	96	0
1	Contingency Plan Capital	20	20	20	20	0
1	Additional Piping	10	10	10	10	10
	Labor Cost/ yr	2.72	0	2.72	0	0
	Cost Of Pigs	5	0	5	0	0
	Cost of DCS and Accessories	20	0	0	20	0
	Total ($mm)		581	589	601	10

6.4.3 Energy Cost

The Products from the North Slope to Valdez pass through several pump stations at the moment. These stations are booster stations and consume fuel for running the pumps and power generators. The first four pump stations are run on gas fuel. Currently the gas is supplied to the

pump stations at no extra cost from the North Slope. The other stations are run on diesel fuel. The fuel cost is a function of throughput of the TAPS. A plot of throughput versus energy cost at various oil prices is presented in Figure 6.2. The gas is assumed to sell at the same price as the gas supplied to the GTL plant and half (for simplicity sake) of the calculated cost for running these stations tied to the GTL process. Equations were fitted through the gas consumption curve to determine what the gas consumption would be at rates that have not been transported through the TAPS and which are anticipated in the future as throughput continues to decline.

6.4.4 Cost of Upstream Natural Gas

Typically natural gas supplied to the GTL plant will be sold by gas producers. The gas producers will price the feed gas high enough to pay for their costs of extraction however they could also add a premium to account for other opportunities to sell the gas. In this analysis, we only consider the minimum cost of feed gas to pay for extraction costs and a profit margin.

To determine the amount of gas needed as feedstock to produce a barrel of GTL, it is necessary to relate the energy content of the produced liquid fuel to the gas used in a common energy unit usually in BTU.

The energy content of a typical barrel of oil is estimated to be 5.8 MMBTU. The energy content of GTL is assumed to be the same with that of a typical barrel of oil. For natural gas, the energy content is about 1 MMBTU per MCF. Solving for the gas energy required per barrel as below:

$$\text{Gas energy per barrel of GTL} = \frac{5.8 \dfrac{MMBTU}{BBL}}{1.00 \dfrac{MMBTU}{MCF}} = 5.8 \frac{MCF}{BBL}$$

At 60% conversion efficiency, the feedstock needed to produce a barrel of GTL is:

$$\text{Gas to GTL conversion} = \frac{5.8}{0.6} = 8.33 \frac{MCF}{BBL}$$

$$\text{North Slope gas price} = \frac{(\text{North Slope GTL Price}) \text{ X } (\text{gas Product net back})}{\text{Gas to GTL Conversion}}$$

Where the North Slope GTL price is also known as the wellhead price.

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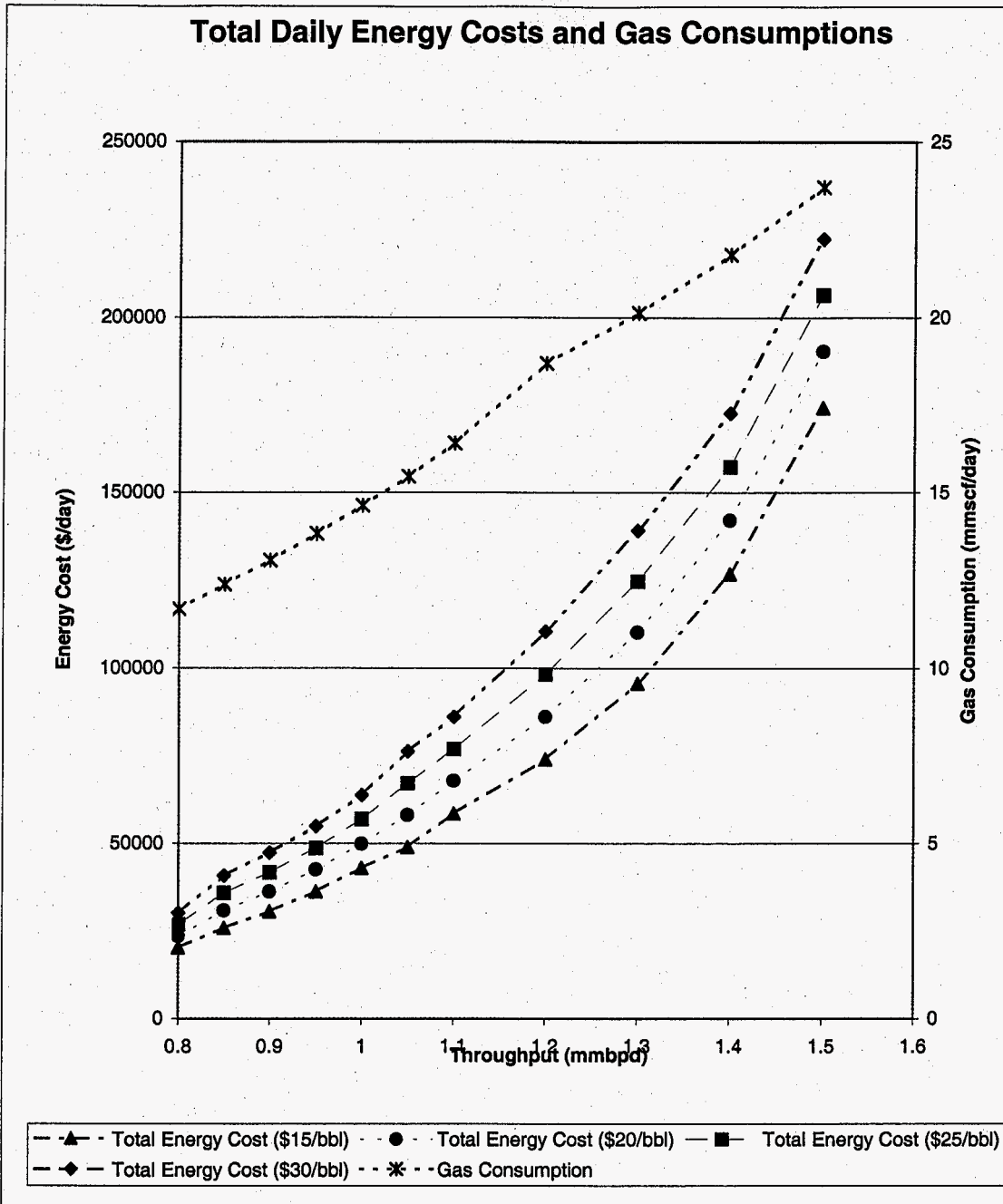
Figure 6.2 Energy cost as a function of throughput. (APSC 2001)

The North Slope gas price is often known also as the gas transfer price and is the cost of the gas feed stock to the buyers which in this case is the GTL operator. The term gas product 'net back' refers to the net fraction of the gas sales as GTL that goes to the owner of the gas. It is usually determined based on agreement on a return on investment expected by the gas owners. As an example, if the price of GTL is $28 and a net back of 10% is used, then the gas transfer price would be approximately $0.34 per MCF. The daily cost of natural gas is estimated at over $1 million. This number is arrived at as follows.

6.4.5 The TAPS Tariff

The TAPS tariff is the most significant cost item in the economics of the transportation of the GTL products through the pipeline. Six independent companies own the Trans Alaska Pipeline System (TAPS). Each of the owners charges their own tariff per barrel of product transported through the pipeline. The tariff is expected to cover the cost of operation and maintenance of the pipeline, the cost of storage, cost of dismantling and demobilizing the TAPS at the end of its operations and in addition to the above yield some return on investment for the owner companies. Operating the pump stations with GTL and Crude oil passing through the TAPS would require an increase in the cost of diesel fuel to run the pump stations. This energy cost is a function of both the throughput and the world spot oil price (Figure 6.2). Presently, pump stations one to four have gas turbines and the gas is supplied at no cost to the pipeline company. When the GTL project commences, the gas is expected to attract extra cost. This is because the gas for the pump stations operations will be an added cost, purchased at the going price of natural gas on the North Slope. The amount of gas required to run the pump stations is also a function of the TAPS throughput. A plot of the amount of gas required to run stations versus throughput is also presented in Figure 6.2.
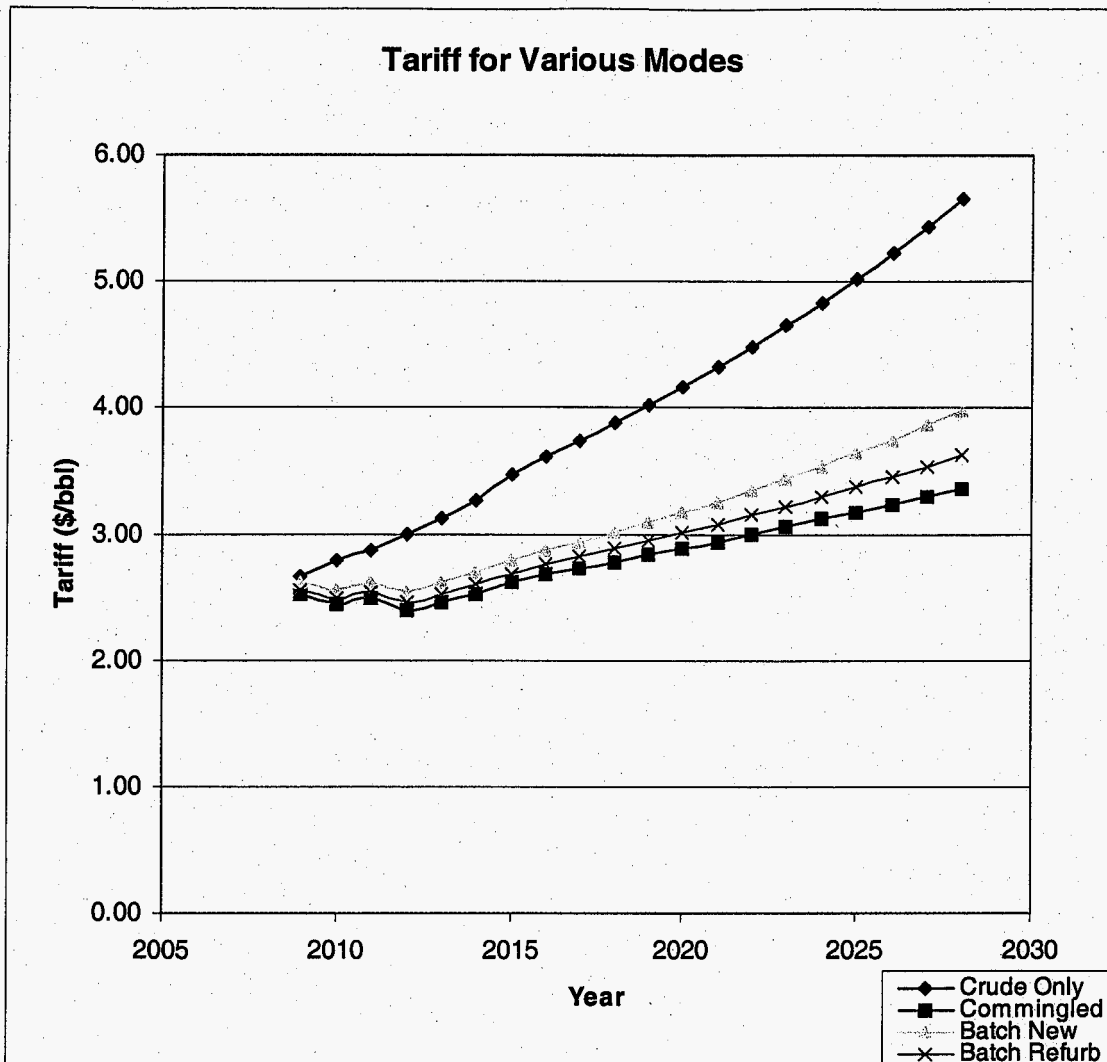
Figure 6.3 Tariff estimate for different scenarios of fluid through pipeline

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Table 6.2 TAPS Tariff Estimate for Various Modes

TAPS TARIFF ESTIMATE FOR DIFFERENT MODES							
No of Periods				20			
Discount Rate				15%			
Capital Inv				601000000		260000000	
Period	Year	Crude Only	Commingled	Batch New ($/bbl)		Batch Refurb ($/bbl)	
1	2009	2.66	2.51	$0.06	$2.57	$0.02	$2.54
2	2010	2.78	2.43	$0.06	$2.50	$0.03	$2.46
3	2011	2.86	2.48	$0.07	$2.56	$0.03	$2.52
4	2012	2.99	2.40	$0.09	$2.48	$0.04	$2.43
5	2013	3.12	2.46	$0.10	$2.56	$0.04	$2.50
6	2014	3.26	2.52	$0.11	$2.63	$0.05	$2.57
7	2015	3.46	2.61	$0.13	$2.74	$0.06	$2.66
8	2016	3.62	2.67	$0.15	$2.82	$0.06	$2.73
9	2017	3.74	2.72	$0.17	$2.89	$0.07	$2.79
10	2018	3.88	2.77	$0.20	$2.97	$0.09	$2.86
11	2019	4.02	2.83	$0.23	$3.05	$0.10	$2.92
12	2020	4.16	2.88	$0.26	$3.14	$0.11	$2.99
13	2021	4.32	2.94	$0.30	$3.24	$0.13	$3.07
14	2022	4.48	2.99	$0.35	$3.34	$0.15	$3.14
15	2023	4.65	3.05	$0.40	$3.45	$0.17	$3.22
16	2024	4.83	3.11	$0.46	$3.57	$0.20	$3.31
17	2025	5.02	3.17	$0.53	$3.70	$0.23	$3.40
18	2026	5.22	3.23	$0.61	$3.84	$0.26	$3.50
19	2027	5.43	3.30	$0.70	$3.99	$0.30	$3.60
20	2028	5.65	3.36	$0.80	$4.16	$0.35	$3.71

The TAPS tariff as noted above incorporates some return on investments for the owners of the pipeline after the operation and maintenance cost. The six owners of the pipeline charge different rates for their 'space' in the pipeline. Therefore, it is not very correct to generalize and assume one particular discount rate for all the companies though they all fall within a range. The discount rate charged by each company depends on the company's view of rate of return and their perception of 'risk'.

The tariff estimates for the next two decades are obtained from the Alaska Department of Revenue (Table 6.2). The estimates give separate numbers for a case where GTL is transported through the TAPS with the crude oil and the tariff if crude oil alone continues to be transported through the pipeline. The tariff for the crude oil and GTL represents the commingled mode, which does not account for the extra capital investment required for batching of the products. For the batch modes, the huge capital costs required to keep the products as clean as possible is factored into the tariff. The tariff for the batch modes is therefore different for the various modes of transportation (Figure 6.3). This illustrates one of the arguments for GTL, that it

provides added liquid fill for the pipeline as crude oil production decreases. That is, it gives a longer life and makes it economic to transport the lower volume of crude oil. This is because it will get so expensive on a per barrel basis that crude oil transportation would have to be shut down were it not for GTL.

For the batch modes, the additional capital investment is allocated on a per barrel basis and a 10% discount rate.

6.4.6 Taxes

6.4.6.1 Property Tax (Ad Valorem)

Each of the three trains is depreciated depending on the number years it is expected to operate within in the 20-year period. Train one, is depreciated over twenty years, train two is depreciated over a 15-year period and train three is depreciated over a 13-year period. The tax base is computed and the property tax derived. The property tax rate is 2%.

6.4.6.2. State Corporate Income Tax

The state corporate income is given by; (income before State and Federal taxes – State Income Tax depreciation) X State Income Tax Rate.

The income tax depreciation is calculated using the MACRS depreciation method. The State Income tax rate used is 9.40 % based on recent values of this rate from the State Department of Revenue (DOR).

6.4.6.3 Severance Tax

The State gas severance tax is assumed to be zero for the GTL project. This assumption is based on the DOR's tax model for the gas projects and would serve as some tax relief to encourage the take off of the gas utilization project.

6.4.6.4 Federal Corporate Income Tax

This is calculated using the income before state and federal income taxes, less the depreciation and multiplied by the Federal Income Tax rate. The Federal Income Tax rate used here is 35% based on current values of this tax.

6.5 GTL PRODUCT PREMIUM

GTL products are expected to receive some price premium compared to conventional crude oil products to reflect their high quality and environmental attractiveness as a fossil fuel. This premium is dependent on the marketing strategies of the GTL. It is expected to follow the world crude oil and oil product pricing system closely. An important crude oil marker grade is the Brent crude oil produced in the North Sea. It is traded internationally on the Internal

Petroleum Exchange (IPE) and the futures market, a rapidly growing trend in world crude oil marketing. The price of crude oils have continued to fluctuate over the past decade and future trends difficult to predict. For example, at low point, Brent sold for $10 per barrel in 1998, but rose to about $33 per barrel in September 2000. In the last decade, the average Brent price was about $19 per barrel and projections put the average at over $22 per barrel in the next five years. Typical GTL yield assessment like 20% naphtha and 80% diesel is assumed reasonable. The GTL diesel is superior to the conventional crude oil refined diesel with regards to sulfur, cetane number, aromatic content and density. However it has relatively poor cold flow properties. Typical GTL diesel has a cetane number greater than 70, compared to a usual diesel product end specification of 50 (Duckler and Hubbard, 1975). This means that opportunities exist for utilizing GTL diesel as a blend stock to upgrade refinery middle distillates products. The zero aromatics content of GTL diesel gives it another advantage for blending with conventional distillates where aromatic content specification becomes a limiting factor. Various numbers have been advanced for GTL product premium. Generally, the GTL diesel product is predicted to have between $2 and $2.5 per barrel premium over conventional diesel. In the model used, a premium of 1.3 times the world crude oil spot price is used. The choice of relating the product premium and price, as a function of the world crude oil price is an obvious one taking into account that price refined products follow the trend of crude oil prices.

Table 6.3
Economic Assumptions

ECONOMIC ASSUMPTIONS	
Conversion @ 60% efficiency	8.33 MScf / Bbl
Plant Uptime Efficiency	95%
Project Life	20 years
Plant Capacity	100 MBPD
Taxes	
State Income Tax	9.4%
Federal CIT	35.0%
Property Tax	2%
Depreciation	Modified Accelerated Capital Recovery Scheme

The assumptions made in Tables 6.3 and 6.4 are part of the major input parameters in the economic model and are used to show the effect of each mode of transportation on the entire project economics.

Table 6.4
Model Parameters

> **MODEL PARAMETERS FOR ROR**
> Cost Estimates
> Plant Cost ranging from $20,000/BPD to $35,000
> Gas Cost based on net back of 10%
> Annual Operating and Maintenance cost of 5.6% of Plant Cost
> Transportation and storage estimated with Tariff estimates.
> Capital investment are amortized over the project life and worked
> out per barrel of product.
>
>
> Revenue Estimates
> ROR calculation based on $21.00 per barrel crude price.
> GTL products given a premium of 1.3 times Spot Oil price
> Batch Transportation efficiency of 95%

6.6 SENSITIVITY ANALYSIS

Key parameters in the rate of return analysis were modified to identify those with the greatest influence on the results. The parameters include:

Capital Expenditure was varied between $20,000 per daily barrel and $35,000 per daily barrel to accommodate speculated range of plant costs and possible North Slope scale up factor.
The crude oil price was varied between $21.00 per barrel and $35.00 per barrel
For the batching operation, installing new storage and relief tanks at the terminal and pump stations respectively versus refurbishing some old tanks to accommodate GTL production and storage.

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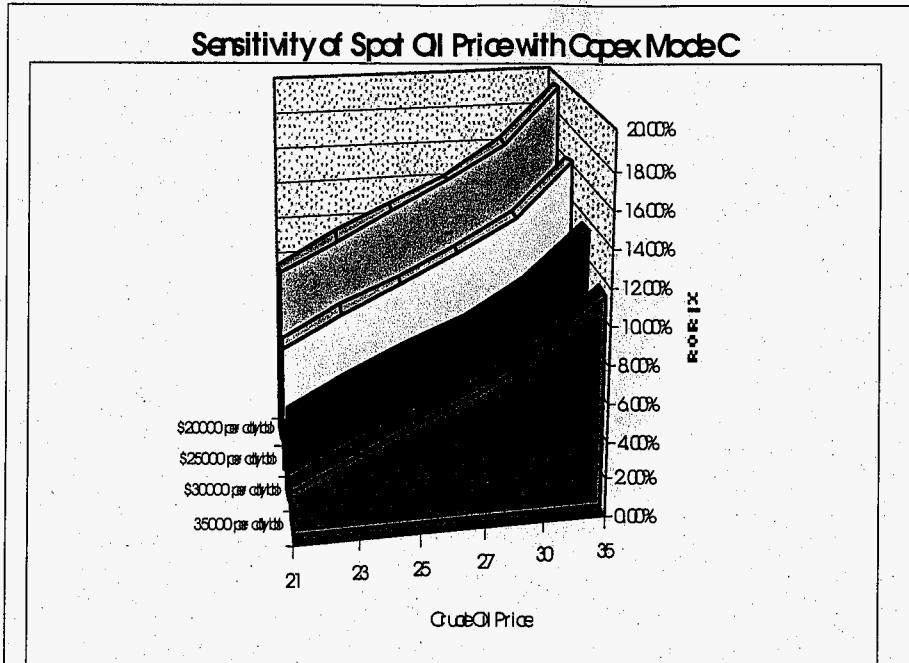
Figure 6.4 Sensitivity Analysis with Various World Oil Prices and Various Capex

Table 6.5
Summary of Results

Summary of Sensitivity Using Batch Mode C				
Crude Price/ Capex $/DBL	35000	30000	25000	20000
21	1.03%	3.30%	5.91%	9.05%
23	2.68%	4.93%	7.54%	10.70%
25	4.16%	6.40%	9.02%	12.22%
27	5.50%	7.75%	10.40%	13.64%
30	7.34%	9.60%	12.29%	15.61%
35	10.02%	12.34%	15.12%	18.57%

6.7 METHOD OF EVALUATION

The capital investment required for transportation of GTL products was amortized and will be paid back through the twenty years of the project life at a discount rate of 15%. The yearly amortization was divided by the throughput to arrive at the extra cost in $/bbl of batching GTL product either by purchase of new infrastructure or refurbishing of existing infrastructure.

6.7.1 Investment Pattern

Construction is assumed to start in year 2005 and last till 2008 for the first train. The capital cost is varied between $20,000 /DBL and $35,000 / DBL invested equally between the four years. The second train is assumed to commence immediately the first is completed and put on production and the third after two years of commencing of the construction train 2. Operating and maintenance cost for each of the trains commenced comes in the same year with production for each of the three trains.

The property tax is calculated from a tax base obtained after depreciating the capital cost using the MACRS and using the taxation formula obtained from the state's department of revenue to calculate property tax base and finally obtain the tax, which is 2% of the tax base. A cash flow model was set up to analyze the same. For the different modes of transportation, the associated capital cost was included under the tariff and comes as cost per year. The cost of gas both as raw material for the GTL plant and the cost of gas for running the first four pump stations are all included in the cost section. The revenue was obtained as a product of the expected product sale price and the total product transported. The taxation was then applied appropriately to calculate net revenue and profit.

6.8 RESULTS AND DISCUSSION

The rate of return analysis result for the various transportation modes are presented below (Figures 6.5-6.7).
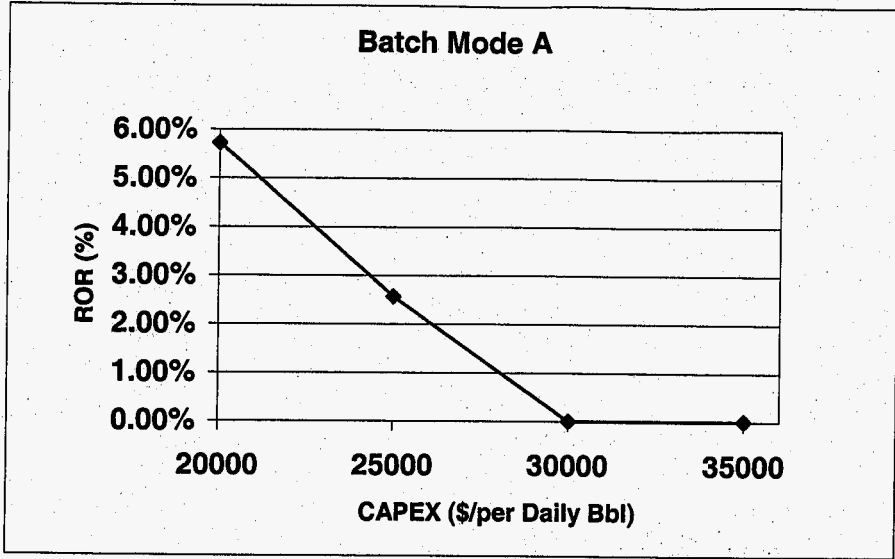
Figure 6.5 ROR analysis for Batch Mode A
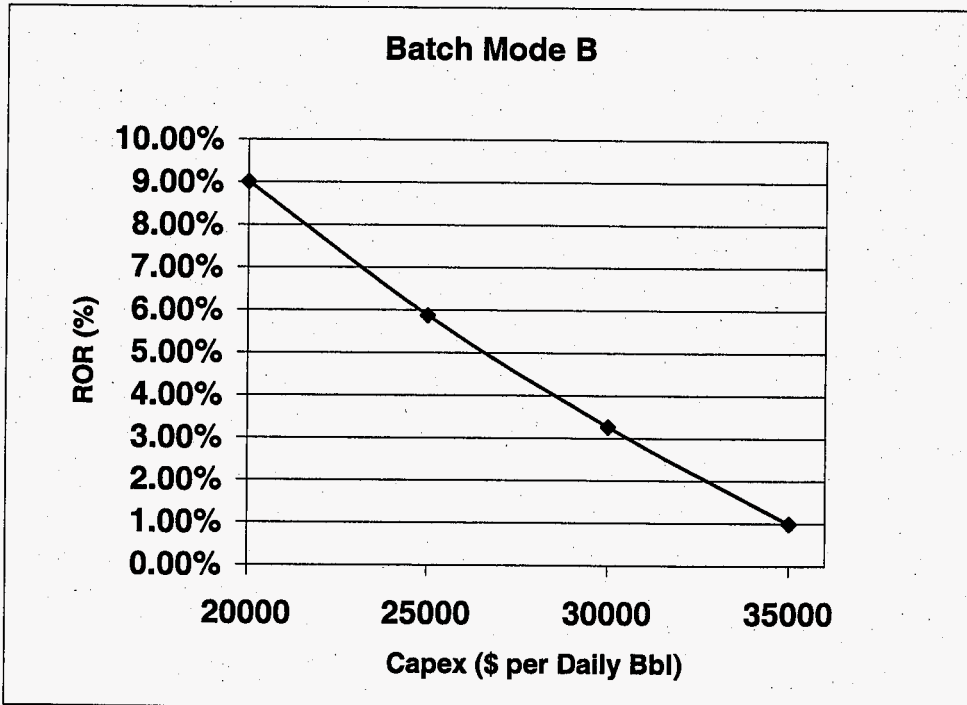
Figure 6.6: ROR analysis for Batch Mode B

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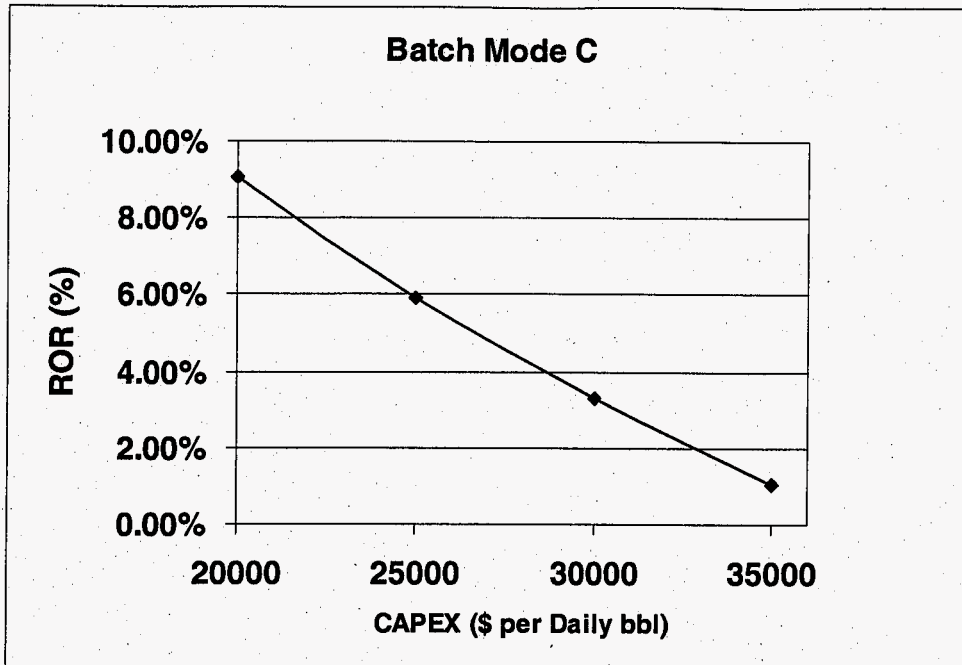
Batch Mode C

Figure 6.7 ROR analysis for Batch Mode C

Batch mode C had the highest return on investment among all the transportation modes and for all plant capital investments scenarios studied. The recovery efficiency of pure GTL product here was put at 95%. This assumes that 5% of the GTL mixes with the lead crude to form an interface and is expected to clean the pipeline for the pure GTL product as the middle fluid followed by another interface of GTL crude oil mixture. Experience of operators that carry out similar operations show that typically, the length of the interface does not depend on the volume pumped but rather on the difference in the physical properties such as density and viscosity of the leading and tailing product as well as the velocity of the fluids in the pipeline. This implies that holding capacity at the North Slope may play a significant role in the optimization process to help minimize the number of slugs to be pumped through in a day.

The GTL premium used in this calculation is 1.3 times the world spot oil price. To arrive at this number, a survey carried out showed that conventional diesel products over the years sold for about 1.42 times the price of crude on the average. A typical GTL plant in this study assumed a product with an 80% yield of Fischer Tropsch (FT) diesel and 20% yield of Naphtha products. Naphtha was given a number of about 1.19 times the price of oil from the historical survey. Combining these two in their ratio of yield and price will give the combined GTL product a value of about 1.37 times the price of crude oil. However, to adopt a conservative approach, 1.3 times the world spot oil price was taken to perform the evaluation. Many authors in the subject are also of the opinion that the GTL diesel should sell at a higher price than the conventional diesel product from typical crude oil distillation process considering its environmental superiority as discussed above. This edge for the GTL diesel was not taken into account in the study. Figure 6.8 shows various rates of return for different GTL product premium prices.
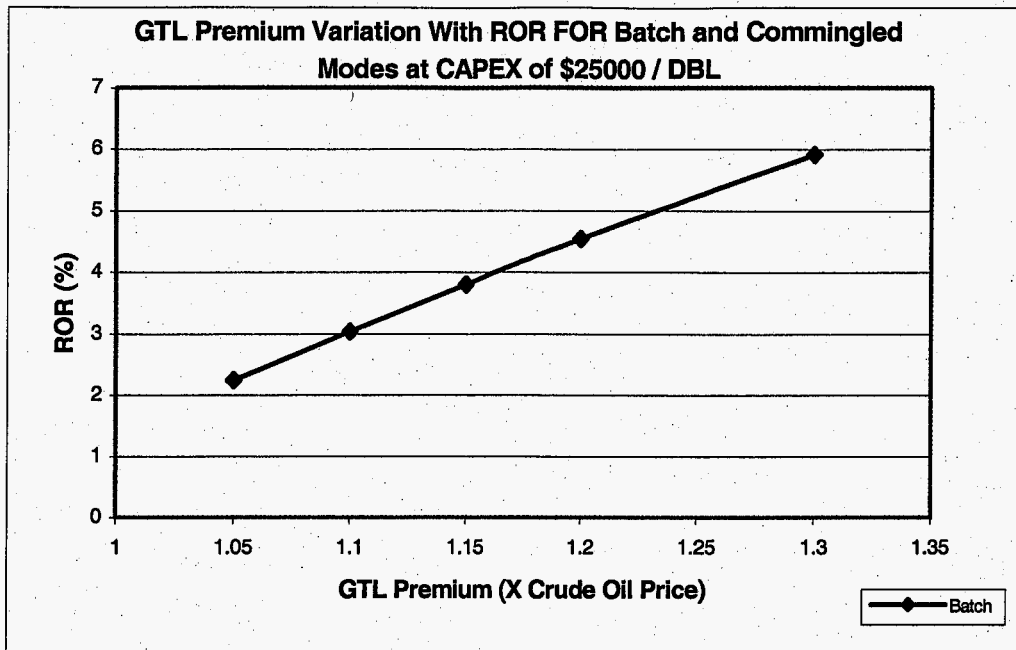
Figure 6.8 GTL Premium Variation with Rate of Return

Another interesting comparison that was conducted out for the adopted batch mode was to compare the difference in the rate of returns between building new tanks at the terminal and reconfiguring existing ones.

Table 6.1 and Figures 6.5-6.7 show the projected difference in capital cost and rate of return respectively.

Table 6.6
Effect of Reconfiguring versus Building New Tanks

CAPEX ($/DBL)	Reconfigure Old Tanks	Build New Tanks
20,000	9.16%	9.05%
25000	6.03%	5.91%
30000	3.43%	3.30%
35000	1.17%	1.03%

Reconfiguring Old Tanks Vs New Tanks
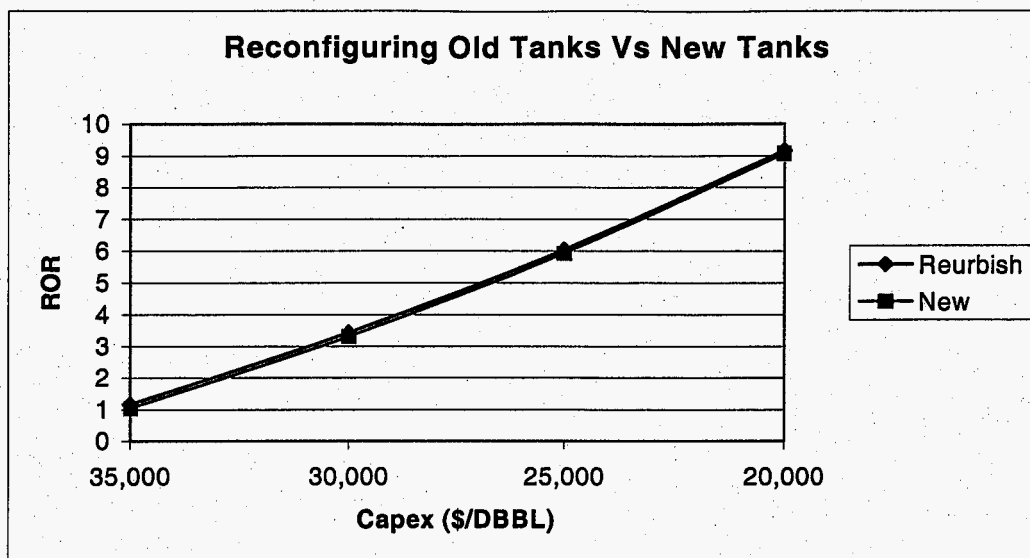
Figure 6.9 Effect of Building new Tanks versus Reconfiguring existing tanks at terminal.

Based on the above, it may appear that there is no major benefit of reconditioning and reconfiguring existing tanks at the terminal for batching operations. However, it is important to take into account the huge capital investment in this project, which means that a slight difference in the rate of returns represents a significant sum of money.

Next to batch mode C on the return on investment analysis is the batch mode B. Product purity is assumed maintained in this mode of transportation with the 95% pure GTL recovery. The labor cost for transitioning the pigs at the pump stations on a continuous basis is the major drawback of this method of batching. Another set back here is that the running such rigorous operation on a continuous basis may introduce a lot of human error factors. Pipeline Pig manufacturers believe the pigging technology can be adapted to suit the TAPS operation and reduce the labor required for this operation. However under the current assumptions, the return on investment for mode B is just slightly less than the batch mode C. The cost of obtaining the extra technology for batch mode C is offset by the high pure product recovery.

The batch mode A is just a base case which is similar to mode C except that no investment is made towards pure product recovery and therefore expected high mixing of the Gas-to-Liquids products and Crude oil is expected in this case.

The commingled mode shows the least returns on the investment. This mode assumes that minimal investment is made to the transportation of the products. The piping from the GTL plant to pump station one on the North Slope is the only major capital investment made on this mode of transportation. The commingled product was initially assumed to sell at same price as the spot crude oil price. When the improvement in the API gravity of the crude oil was put into consideration and assuming that the commingled product will have a higher yield of middle distillate when compared to the original crude oil, a one-dollar raise in product value was

assigned and the model run again. The batch mode gave the higher return on investment. The difference in the return on investment between the batch and the commingled mode is very significant considering the fact that any minute difference in ROR figure of even less than 1% for such capital intensive project runs into hundreds of millions of dollars.
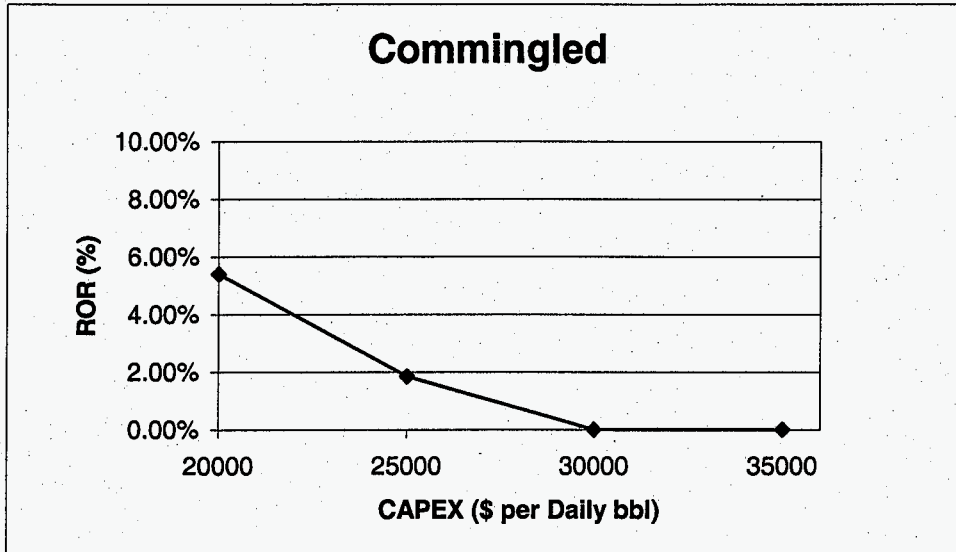
Commingled

Figure 6.10 ROR analysis for Commingled Mode

Summary of ROR @ $25,000 and $21/bbl
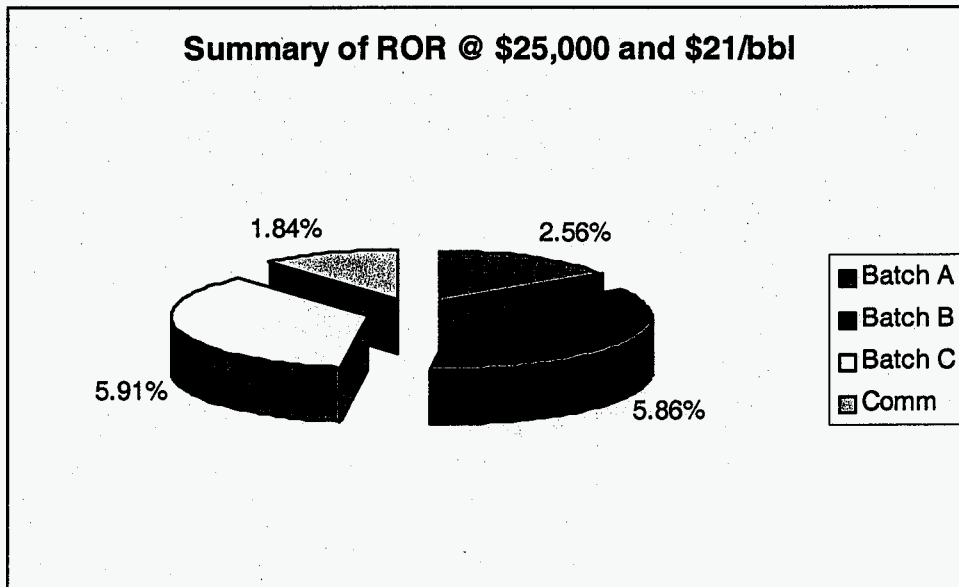
Figure 6.11 Summary of ROR Analysis For all Modes of Transportation

6.9 SUMMARY AND CONCLUSIONS

The modern batching approach consistently gave the highest return on investment and is recommended for transporting the Gas-To-Liquid products from the North Slope of Alaska to Valdez. The major concern with batching is the length of the mixing zone or interface and the purity of the GTL products as they arrive the Marine Terminal in Valdez. Since experience shows that the length of this interface is independent on volume pumped, it becomes an optimization issue to find the optimum holding capacity on the North Slope that can give the minimum number of batches at any given production period. The optimum fluid velocity in the pipeline should be determined with reasonable accuracy based on the density and viscosity difference of the two products to be transported to ensure minimum interface.

One reason for the low numbers in the rate of return analysis is that the project life assumed is not long enough to enable the project make adequate profits after pay out. Considering the investment pattern, train 2 and 3 for example barely had enough time to pay out and start making profit. Typical pay out times are shown in the figure 6.11 below:
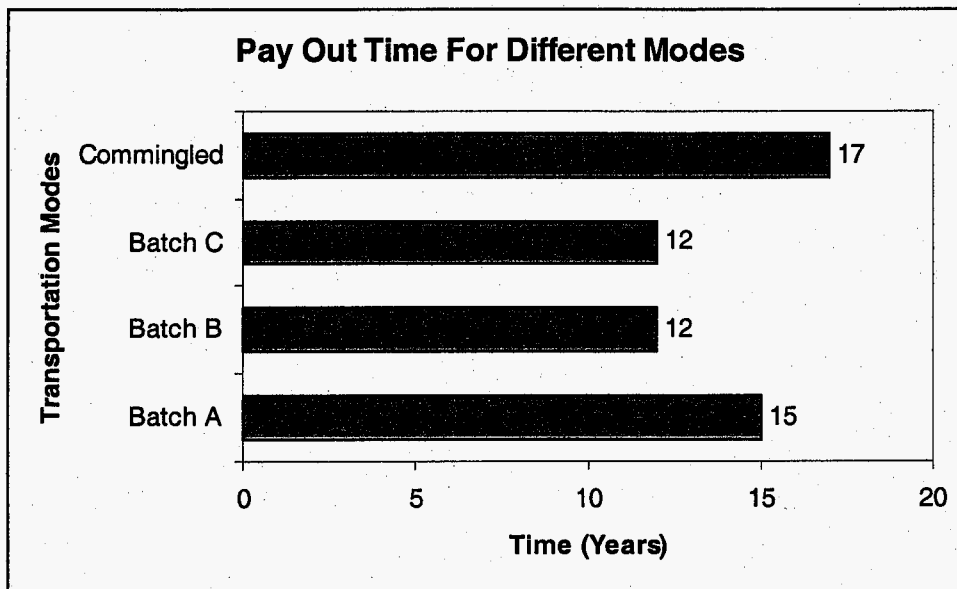
Figure 6.12 Summary of Payout time for Capex $25,000 / DBL and Crude price of $21/bbl

From Figure 6.11 above, it is evident that the project has not had enough time to make sufficient profit for the 20-year evaluation. One quick way to make projection after the twenty years period is to make a plot of the ROR progress over the observed years and make forecasts. Figure 6.12 shows these results.

From Figure 6.12, the projection on the ROR curve shows that the project still has about 8 years before the project life chosen would not matter anymore. Since this study is focused on a

comparative analysis of the transportation modes, the magnitude of the rate of return is not the key concern.
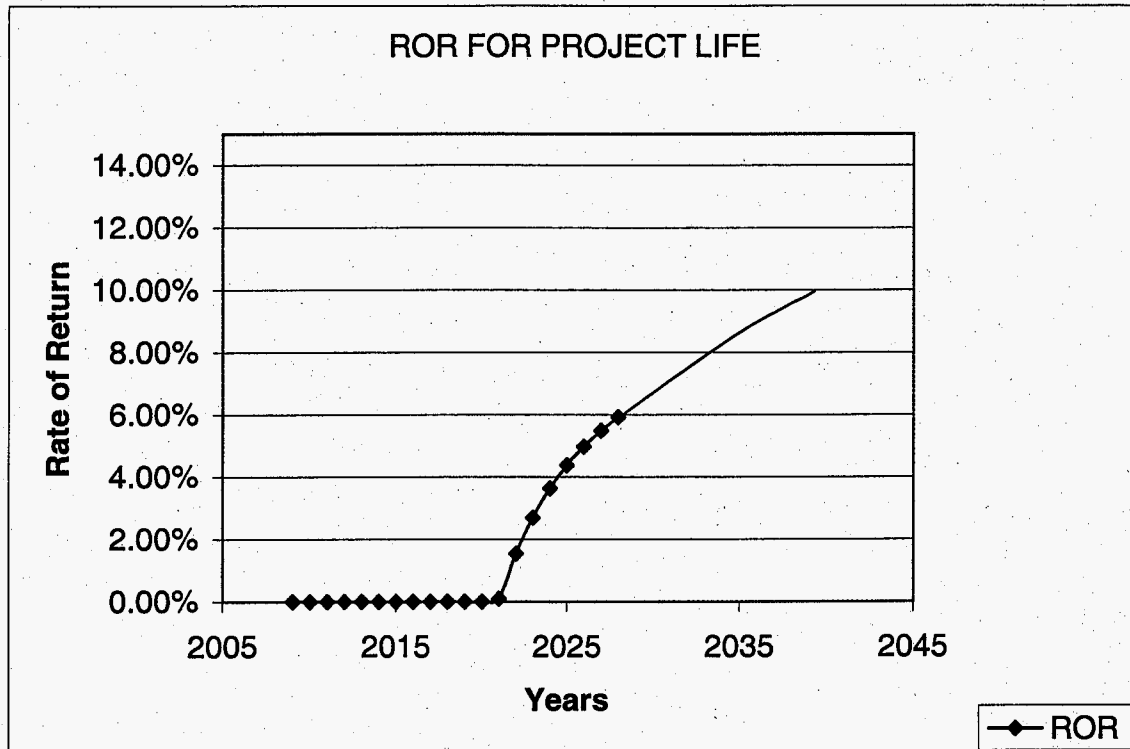
Figure 6.13 Project Life evaluation time.

NOMENCLATURE

ANS	Alaska North Slope
APSC	Alyeska Pipeline Service Company
DBL	Daily Barrel Liquid
DCS	Distributed Control System
DOE	Department of Energy
EIA	Energy Information Administration
GTL	Gas-to-Liquids
LNG	Liquefied Natural Gas
ROR	Rate of return
TAPS	Trans-Alaska Pipeline System
CAPEX	Capital Expenditure
L_m	Length of Mixing Zone, m [ft]
L_s	Slug Length, m [ft]

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CHAPTER 7

CONCLUSIONS AND RECOMMENDATIONS

7.1 CONCLUSIONS

Based on the experimental and analytical study carried out at UAF on technical and economic feasibility of transporting GTL products through TAPS, the following main conclusions are drawn.

Cold restart of TAPS following an extended winter shutdown is the main transportation issue that will govern the technical feasibility of moving GTL products through the pipeline.

It was found that GTL samples and crude oil readily developed miscibility when mixed at ambient temperature and pressure conditions.

The trend of both density as well as viscosity reduction as a function of increasing temperature is clearly evident for all the tested samples.

The dilution of crude oil by GTL causes a major reduction in both density as well as viscosity.

The measured density and viscosity data can be correlated using non-linear regression techniques. It is demonstrated that the developed correlations are capable of reproducing the measured data with reasonable accuracy and reliability.

A trend of increasing yield stress with increasing wax content was clearly observed. Gel strength tests at $-20°F$ for the GTL 1 and TAPS crude oil blends had to be abandoned owing to very high yield stress (beyond measurable limits of the apparatus) already encountered at temperatures higher than $-20°F$.

The results indicate that light GTL samples had measurable gel strength whereas significant gel strength problems were observed for heavier GTL and crude oil blends.

The GTL 2 sample (FT diesel) produced high gel strength, but when it was blended with TAPS crude oil, a significant reduction in gel strength was observed.

No gel strength measurements could be reported at temperatures of $-20°F$ for the GTL 2 and TAPS crude oil blends due to very high yield stress (beyond measurable limits of the apparatus). The high gel strength can be attributed to the chemical composition of the GTL 2, which is highly paraffinic in nature.

The flow model equations developed in this study was analytically solved for predicting the pressure gradients encountered in the batch and commingled flow modes of transporting GTL products and Crude Oil through the Trans-Alaskan Pipeline System (TAPS).

The derived flow equations presented here can be modified under specified operating conditions or constraints of the Trans-Alaskan Pipeline System (TAPS), using live GTL or Crude Oil data.

Mixing at the Oil-GTL interface in the case of batch mode transportation poses flow modeling and simulation difficulties.

The pressure gradients obtained from the batch flow calculations are higher than those obtained from that of commingled flow.

Because GTL products have higher gel strength than crude oil or GTL-crude oil blends, feasibility of batch mode of transportation will be limited by TAPS cold restart requirements.

The thermodynamic model developed in this study shows good agreement between calculated and measured oil temperatures, proving validity of the model.

Heat loss in the below ground section of TAPS is higher than heat loss in the above ground section, which is because of the absence of insulation in the below ground section.

Heat loss from GTL flow is less than heat loss from crude oil flow for both below ground and above ground pipeline. This is due to a reduction in mass flow rate for GTL.

Exit temperature of the GTL is slightly lower than the exit temperature of the crude oil. Lower mass flow rate has lower heat content and cools down further.

The heat loss is more in January than in March both for crude oil and GTL. The increased heat loss in January is because of the low ambient temperatures. The temperature difference between the fluid and the ambient air is greater which results in more heat loss.

The temperature of oil arriving at Valdez is 57°F for crude oil from the calculations. Temperature reported by Alyeska Pipeline Service Comapny is 60°F. This minor difference may be due to the assumptions made in the calculations.

The economic analysis shows that the modern batching approach consistently gave the highest return on investment and is recommended for transporting the GTL products from the North Slope of Alaska to Valdez.

The major concern with batching is the length of the mixing zone or interface and the purity of the GTL products as they arrive the Marine Terminal in Valdez. Since experience shows that the length of this interface is independent on volume pumped, it becomes an optimization issue to find the optimum holding capacity on the North Slope that can give the minimum number of batches at any given production period. The optimum fluid velocity in the pipeline should be determined with reasonable accuracy based on the density and viscosity difference of the two products to be transported to ensure minimum interface.

One reason for the low numbers in the rate of return analysis is that the project life assumed is not long enough to enable the project make adequate profits after pay out. Considering the investment pattern, train 2 and 3 for example barely had enough time to pay out and start making profit. Typical payout times are shown in the Figure 7.1 below:

Pay Out Time For Different Modes
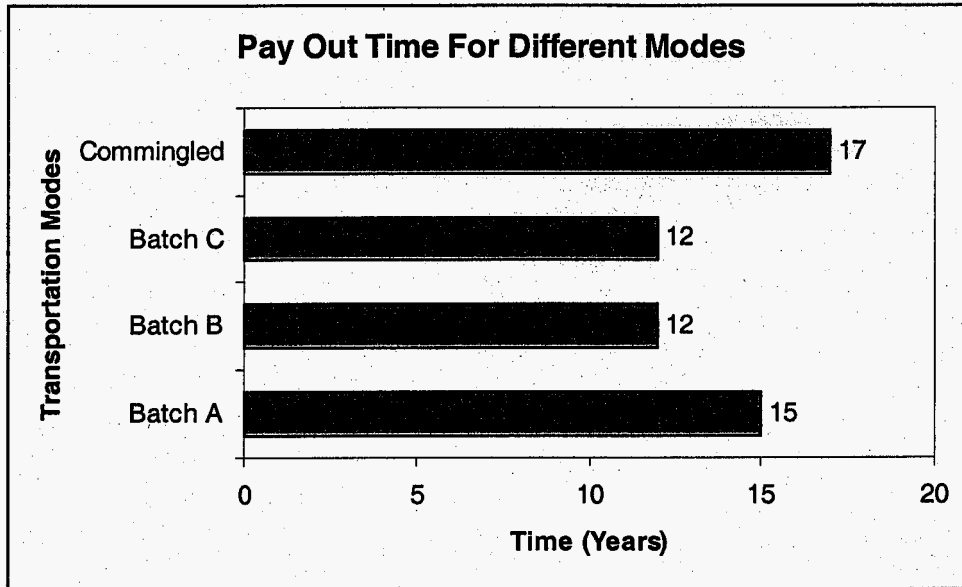
FIGURE 7.1 Summary of Payout Time (Capex $25,000/DBL, Oil $21/bbl)

From Figure 7.1 above, it is evident that the project has not had enough time to make sufficient profit for the 20-year evaluation. One quick way to make projection after the twenty years period is to make a plot of the ROR progress over the observed years and make forecasts. Figure 7.2 shows these results.

From Figure 7.2, the projection on the ROR curve shows that the project still has about 8 years before the project life chosen would not matter anymore. Since this study is focused on a comparative analysis of the transportation modes, the magnitude of the rate of return is not the key concern.

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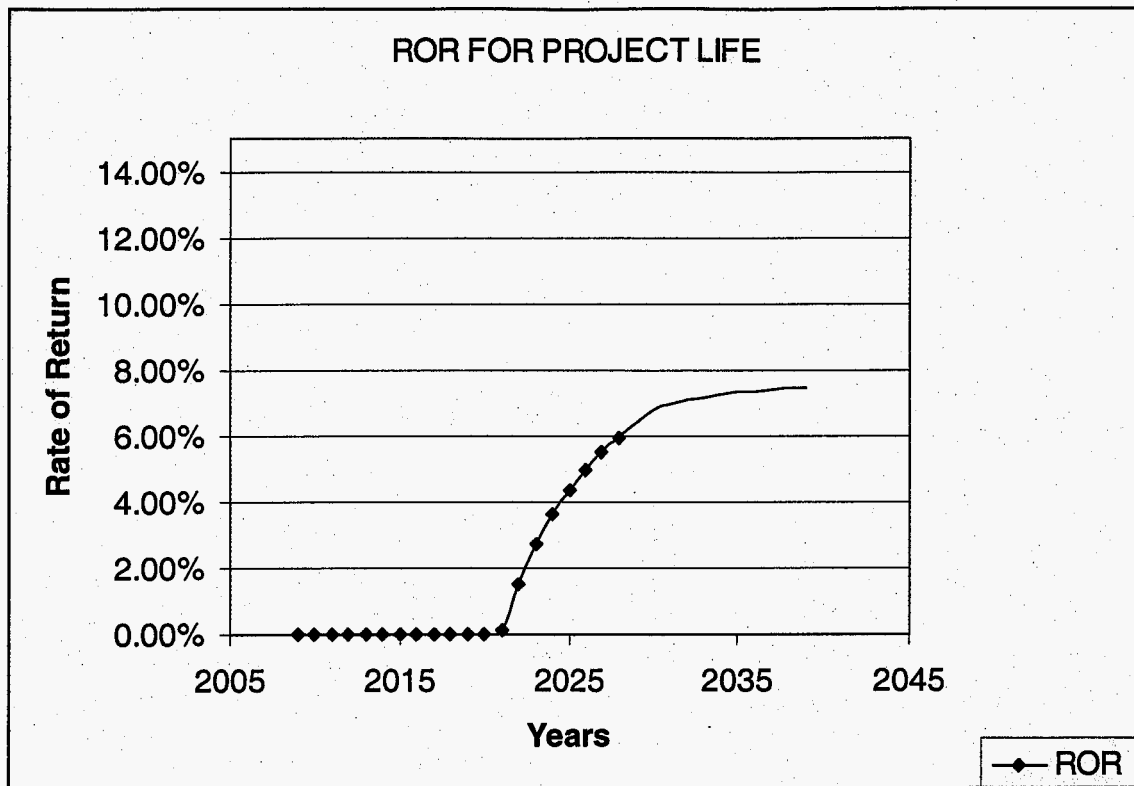
FIGURE 7.2 Project Life Evaluation Time.

7.2 RECOMMENDATIONS

It is evident from the results of this study that the logistics of transporting GTL products from the Alaskan North Slope to the markets through TAPS is extremely complicated. This study by no means should be considered as the final word on this issue. Many transportation issues were identified, which pose unique operational challenges in GTL transportation through TAPS and need to be examined. The following recommendations are made for continuing work in this area.

This study indicates that even the light GTL (LaPorte type) or FT diesel can pose problems for transportation through TAPS from the cold restart point of view. Rigorous studies are needed to identify the upper limit on the quantity and nature of paraffins in GTL that can be accepted for transportation through TAPS.

Since high molecular weight paraffins are not likely to be acceptable, it may be necessary to include a hydrocracking unit in the GTL plant considerations for the North Slope. Studies are necessary to recommend the composition and upgrading options for GTL to be produced in the North Slope.

Blending of GTL and crude oil may be effective in lowering the ultimate yield stress values. However, it will be worthwhile to determine if the effect of blending is reproducible with varying crude oil composition.

Along with gel strength, Reid vapor pressure (RVP) is also an important parameter that will determine transportability of any fluid through TAPS. Therefore, RVP measurement studies on GTL products and their blends with crude oil need to be performed.

Although modern batching technique appears to be the transportation mode of choice at this time, batching GTL products through the same pipeline that carries crude oil is likely to create significant problems of GTL product contamination. Wax, sulfur, asphaltene and other assorted solids deposited on the inside of the pipeline can potentially re-dissolve in the slug of pure GTL. Since GTL is a clean, zero sulfur fuel, this type of contamination could defeat the very purpose of gas to liquid conversion. Further studies are necessary to investigate the effect of GTL contamination from the pipe-wall residue.

After studying the operational issues, it will be necessary to re-visit the economics of GTL transportation. For example, the economics of batching mode could potentially include an additional cost of purifying contaminated GTL products. The blending mode, on the other hand, may make it feasible to have a cheap GTL plant producing low grade GTL, thus reducing capital expenditure.

APPENDIX A

QUALITY ASSURANCE PROJECT PLAN

This Quality Assurance Project Plan (QAPjP) has been prepared in connection with potential collaborative project work between APSC Pipeline Service Company (APSC) and the University of Alaska Fairbanks (UAF). The collaborative work is envisioned to include various aspects of transporting crude oil and/or oil-GTL blends. Studies of wax characterization, solids precipitation, fluid properties and rheological measurements are being planned. Such work would require laboratory measurement of fluid and solid properties and their characterization. This QAPjP outlines the general provisions for ensuring quality and integrity of laboratory data generated by UAF.

A.1 QUALITY OBJECTIVES AND CRITERIA FOR EXPERIMENTAL DATA

The data quality objective for this project is to provide valid data of known, acceptable and documented quality for all samples submitted for testing. The data quality indicators to be measured are identified below.

A.1.1 Accuracy and Precision

The goals for analytical testing accuracy and precision are defined by the "bias" and single-analyst 'repeatability' statements for the associated the American Society for Testing and Materials (ASTM) method. These ASTM data quality measures will be met as acceptance criteria standards for data generated, unless otherwise specifically agreed in writing by APSC. If no quantitative measure is defined by ASTM, UAF acceptance criteria will be generated as described below.

When an ASTM method (or other industry standardized method) is not used, then acceptance criteria may be approved by APSC when documented by UAF statistical analysis of recent test data (acquired from testing like/similar matrices), or when those acceptance criteria are documented in the associated UAF procedure document.

A.1.2 Comparability and Representativeness

Representativeness will be addressed through the controlled collection of samples, as governed by the standard sampling and shipping procedures used by UAF and the associated provisions of the APSC Work Order document initiating the activity. Comparability will be addressed through the consistent use of established procedures for sample collection, laboratory analysis, data review, validation and reporting.

A.1.3 Completeness

A completeness goal of 90% is deemed necessary. Valid data are required for each sample type in order to complete accurate data interpretation and to complete effective pipeline modeling. Completeness will be calculated and reported in the Quality Assurance (QA) / Quality Control (QC) reports.

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A.1.4 Special Training Requirements/Certification

UAF personnel used in the technical performance of this project will have demonstrated and documented capabilities for the analytical, professional and quality activities they complete. UAF personnel training records, applicable certificates, accreditations and the like will be reviewed and accepted for adequacy and applicability to the project by APSC prior to those staff being assigned to work on a project. Training records of all UAF staff and students working on the project will be kept within the project files at UAF.

Alyeska Pipeline Service Company (APSC) will approve all personnel, in writing, that work on the project. These sign-off documents will become part of the project documentation retained by UAF.

A.1.5 Documentation and Records
Laboratory Records
The following list of the laboratory-specific records will be complied by UAF.

A.1.5.1 Sample Management Records

Sample management records document sample receipt, handling and storage, and scheduling of analyses. The records verify that the chain-of-custody and proper pre-treatments were completed. These records reflect any anomalies in the samples (such as receipt of damaged samples), note proper log-in of samples into the laboratory, and address procedures used to ensure that analytical requirements were met (e.g. handling to prevent light ends loss).

A.1.5.2 Test Methods

Analyses are expected to be performed exactly as prescribed by the documented Standard Operating Procedures (SOPs). If not, test methods documentation will describe how the analyses were actually carried out in the laboratory. This topic includes sample preparation and analysis, instrument standardization and calibration, detection and reporting limits, and test-specific QC criteria. Documentation shall be included that demonstrates laboratory proficiency and compliance in each of these methods.

A.1.5.3 Sample Data

These records contain raw and finished data as produced by technicians and analysts for laboratory testing, and will be maintained in the respective job file folder. These records also include: the overall number of samples, sample location information, any deviations from the SOPs, date analyzed and person performing the work. Corrective actions taken to replace sample data violating an approved protocol must also be noted.

A.1.5.4 QA/QC Reports

These reports will include where applicable the general QC records (e.g. initial demonstration of capability), instrument calibration, routine monitoring of analytical performance and calibration verification. Project-specific information from the QA/QC checks such as calibration check samples (zero check, span check, and mid-range check), field and laboratory replicates, sample splits, and the like will be included where applicable in these reports to facilitate data quality analysis.

A.1.5.5 Data Handling Records

These records document protocols used in data reduction, verification, and validation. Data reduction addresses data transformation operations such as converting raw data into reportable quantities and units, use of significant figures, recording of extreme values, blank corrections, etc.

Data verification ensures the accuracy of data transcription and calculations and, if necessary, by checking a set of computer calculations manually. Data validation ensures that QC criteria have been met.

Each sample received in the laboratory is assigned a unique identification number. Data generated for each sample is referenced to this number. Data generated may be recorded by hand, or through electronic means such as spreadsheets.

A.1.5.6 Data Reporting Package Format and Documentation Control

All individual records that represent actions taken by UAF to achieve data quality and the performance of specific QA functions are potential components of the final data-reporting package. A Data Package Checklist (Table A.1) will be utilized to help ensure that the reporting package is complete and meets the specified requirements.

Interim, draft and final reports, data tables and other deliverables will be provided by UAF to APSC as requested. Hard copy reports will be faxed or courier delivered when requested by APSC. Electronic data deliverables will be provided by UAF using Microsoft Office (Professional) component applications (latest version available to UAF will be used), unless otherwise approved or requested by APSC in writing.

Data passing the second level quality review will be forwarded in electronic data format (MS Excel spreadsheet), with headers/titles as needed to specify date and time of sampling, date/time of sample analysis, name of the person performing the test, test results (with appropriate significant figures), test accuracy and precision (per applicable SOP or published methodology) and comment fields (as required to provide needed explanatory information about the test results, e.g. unusual sample properties, observations, etc.). This electronic submission of reviewed data to the Crude Oil Study (COS) project will be sent promptly after work completion, but not later than ten (10) working days following completion of second level quality review.

All data for all tests will also be collated and reported formally in both electronic and hardcopy formats. The report will follow UAF's standard data and QA/QC report or mutually agreeable format. Electronic submission will consist of 1) MS Word for report text and discussion, and 2) MS Excel for data tables and graphical presentation of test results or other numeric data.

The final draft report will be reviewed by UAF to ensure work results of appropriate quality (completeness, accuracy, precision, etc.). Report release will be authorized by the UAF Principal Investigator's signature (on hardtop) attesting to its acceptability and compliance of the work reported with the provisions of this QAPjP.

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A.1.5.7 Data Reporting Package Archiving and Retrieval

All electronic and hardcopy records associated with this UAF-APSC collaborative project produced by UAF will be retained in original form and format for a minimum of 5 years. These records include all logbooks, procedures, analyst notes, instrument calibration information, instrument output files (e.g. chromatograph output data files), raw and finished data, data validation and verification records, the approved QAPjP, personnel qualifications and training records, draft reports, chain of custody and QA/QC reports. Accordingly, UAF will retain the capability to re-examine and reprocess any raw data, (e.g. retain current chromatographic data processing software and its native hardware platform).

A.1.5.8 Electronic Data

Archiving of electronic data will be accomplished through the use of an 8 mm DAT data cartridge or floppy disk based archiving format. Data will be archived according to the source for ease of retrieval. Files will be identified by a name that will relate it to the job file number assigned by UAF during log in. Data generated or calculated using PC-based spreadsheets (MS Excel®, etc.) or other programs will be archived periodically as required. Correspondence or report text and discussion will also be archived.

A.1.5.9 Hardcopy Records

Hardcopy records will be maintained for a minimum of five years. Such records shall include, where applicable, sample test results, external and internal chain-of-custody records sample receipt checklists, data review checklists, data package checklists, test method documents, QA/QC reports, data handling records, final reports, data tables, or quality records.

A.2 MEASUREMENT/DATA ACQUISITION

A.2.1 Sampling Methods Requirements

All field sampling will be performed by qualified staff in a professional manner, consistent with established and documented Quanterra sampling procedures, as approved by APSC for the UAF-APSC collaborative work.

A.2.2 Sample Handling and Custody Requirements

All samples received by UAF will be initially inspected to identify damaged shipping containers, leaking containers or other sample problems. 'Condition upon receipt' records will be generated for all samples.

Documentation of Chain-of-Custody (CoC) will be provided for all samples by UAF upon receipt of the samples. CoC documentation will be maintained (or initiated internally) by UAF for all received samples. Proper sample custody minimizes accidents by assigning responsibility for all stages of sample handling and ensures that problems will be detected and documented if they occur. A sample is in custody if it is in actual physical possession or it is in a secured area that is restricted to authorized personnel.

Sample custody procedures are necessary to prove that the sample data correspond to the sample collected. Aliquots prepared from CoC samples must also have CoC records. These will be generated by UAF and show aliquot identities as associated with the parent CoC

sample. All samples, sub-samples, splits and aliquots will be identified and approximately labeled to show sample custody through its end use. Any unused material must remain in custody until the sample is disposed off or return is directed by APSC.

A.2.2.1 Receipt of Chain of Custody and Log-In
Log-in and receipt of samples will be conducted at UAF by authorized personnel or designee. At the time of receipt, the accompanying CoC will be completed. The receiving log to be used for TAPS/COS samples is shown in Table A.2. CoC forms may be supplied by UAF or other APSC approved forms can be used. Additionally, UAF supplied CoC seals or other acceptable seals will be used to ensure integrity of sample containers. A checklist will be completed for each shipment received and maintained in the job file folder. Samples received will be given a unique identification number. Tables A.3 – A.5 shows the forms to be used for sample log-in and identification purposes. All shipping documents and associated papers will be placed and maintained in the job file folder. If at the time of receipt, any non-conformity is noted with the samples, a representative of APSC will be notified.

A.2.2.2 In-House Laboratory Transfer and Chain of Custody
Samples received will be delivered to the designated sample manager for UAF. The sample manager will be responsible for in-house transfer and chain of custody for each sample. Samples will be maintained in a secured area with access limited to the sample manager or principal investigator. Laboratory personnel will retrieve the sample(s) from the sample manager and complete the required information on the Internal Chain of Custody Record. The forms to be used for maintaining the Chain of Custody Record are shown in Tables A.6 – A.8. Upon completion of the analysis, the analyst will return the sample and complete the internal CoC, relinquishing the sample back to the sample manager. The shipping log to be used for TAPS/COS samples is shown in Table A.9.

A.2.3 Quality Control Requirements

A.2.3.1 Sample Batches
A sample batch represents 10 samples or as noted by individual test methods. APSC samples will not be analyzed in batches containing samples from other sources. APSC samples will be analyzed individually or in batches with other APSC samples only.

A.2.3.2 Instrumentation
Instruments and equipment to be used for sample analysis must have met all required maintenance and calibration requirements before analysis begins. Maintenance and calibration documentation must be completed prior to initiation of sample analysis.

A.2.3.3 Analysts
Analysts, students and technicians involved in sample testing must have met training requirements for the procedure to be used and have 'current' status documentation, UAF certification, other recognized accreditation from an approved accrediting agency, or approval by APSC. One primary analyst is expected to complete all tests comprising a single sample batch for a single test procedure.

A.2.3.4 Accuracy

Procedure accuracy will be checked through the analysis of approved calibration material. Calibration material will be analyzed at the beginning of each batch of APSC samples analyzed and at the beginning of each work day and after every 10th sample and at the end of each work day or whenever a different analyst is involved with sample preparation or sample analysis. Calibration material will also be analyzed at the end of each batch of APSC samples. Any deviation from this process must be documented in the individual SOPs and approved by APSC.

Acceptance criteria must be met before sample analysis can begin or continue. Acceptance criteria will be defined in the individual SOPs. Failure to meet acceptance criteria at the beginning of a sample batch or work shift requires that corrective actions be taken (and documented) and approved by the principal investigator before analysis can restart. Restart requires demonstration of satisfactorily meeting the acceptance criteria. Failure to meet acceptance criteria at the end of a sample batch requires that corrective actions be taken (and documented) and approved by the principal investigator. Unless specific casual analysis results clearly indicate otherwise, all sample test results are invalid since acceptance criteria were last met.

A.2.3.5 Precision

Procedure precision will be checked through the analysis of laboratory replicates. Samples will be split during aliquoting for assignment to various batches for analysis. Precision will be measured using the following guidelines in cases where representative sub-samples can be obtained.

Both known and blind replicates will be prepared during this process. At least one replicate will be analyzed in each sample batch or by each analyst completing test results within the batch. Test results for (known) laboratory replicates must be checked against their acceptance criteria promptly. Acceptance criteria must be met to continue the analysis process. Failure to meet acceptance criteria requires that corrective actions be taken (and documented) and approved by the principal investigator before analysis can restart. Restart requires demonstration of satisfactorily meeting acceptance criteria.

Test results for (blind) laboratory replicates must be checked by the principal investigator against their acceptance criteria promptly (before the end of work shift). Acceptance criteria must be met to continue the analysis process. Failure to meet acceptance criteria requires that corrective actions be taken (and documented) and approved by the principal investigator before analysis can restart. Restart requires demonstration of satisfactorily meeting acceptance criteria.

A.2.4 Instrumentation/Equipment Testing, Inspection, and Maintenance

Analytical instrumentation will be inspected once a month by UAF trained technicians, other personnel approved by APSC, or the analyst before use. Regular preventative maintenance will be performed according to the equipment manufacturer's recommendations and these records may be reviewed by APSC if needed.

Repair maintenance will be completed as needed, and no instrument or equipment will be used when known to be in a state of repair, regardless of the perceived margins for data quality or safety associated with the repair required. Instruments requiring repair will be clearly tagged as "Out of Service." When an instrument or piece of equipment is identified as requiring repairs, then validation of the previous test made with such instrumentation or equipment is required.

A.2.4.1 Instrument/Equipment Calibration and Frequency

Analytical instrumentation calibration/standardization will be performed in accordance with UAF's standard policy using the associated approved analytical SOPs or the manufacturer's recommendations following UAF SOPs. When an instrument or piece of equipment is identified as needing calibration, then validation of the previous test made with such instrumentation or equipment is required. No testing will be performed by equipment out of the calibration date.

A.2.5 Data Management

The UAF project team will ensure that all mathematical operations and analyses performed on raw ("as-collected") data are reviewed for correctness. This review examines any change to data form of expression, location, quantity, or dimensionality to ensure that it is completed correctly and consistent with the approved SOPs. These operations include data recording, validation, transformation, transmittal, reduction, analysis, management, storage and retrieval.

A.2.5.1 Data Recording

UAF will complete internal checks (including verification and validation checks) to ensure data quality during data encoding in the data entry process for all the data collected.

A.2.5.2 Data Validation

Data validation is based on the acceptance criteria specified in the appropriate technical SOP or published industry procedure (i.e. ASTM or GPA). A project specific QA requirements matrix will be developed.

A.2.5.3 Data Transformation

Data transformation is the conversion of individual data point values into related test result values. Raw data (individual data point) obtained in the laboratory are converted to test resultant values by various calculation routines as detailed in the appropriate technical SOPs, associated reporting form, and/or industry standard method. Where applicable, example calculation routines and/or "dummy spreadsheets" are to be included as an adjunct to the technical SOPs. Calculations will be provided in the final data package to adequately follow the progress from raw to reported data.

A.2.5.4 Data Transmittal

Data transmittal occurs when data are transferred from one person or location to another or when data are copied from one form to another (e.g. copying raw data from a notebook onto a data entry form for keying into a computer file and electronic transfer of data over a telephone or computer network). The UAF project team will conduct and document the review of data transmittal, which will include verification of computer entry from logbooks and/or instrument printouts.

A.2.5.5 Data Reduction

Data reduction includes all processes that change the number of data items. This process is distinct from data transformation, in that it entails an irreversible reduction in the size of the data set and an associated loss of detail. Wherever practical, raw data are reduced using automated data processing. This involves the use of validated spreadsheets. A validated set of raw data is utilized to ensure that inadvertent changes have not been made to the calculation routines contained in these spreadsheets. Manual calculations are documented in the specific method logbook form. As a part of the data verification process, manual calculations will be verified by the UAF project team by performing a random set of sample calculations and checking the same.

A.2.5.6 Data Analysis

As a part of the overall QA/QC of the project, UAF will provide a quality assurance document that summarizes the findings of the QA samples to include statistical representations where appropriate to include at a minimum comparison to the method or SOP precision in the QAPjP QA Requirements Matrix.

A.2.5.7 Data Tracking

Data management includes tracking the status of data as they are collected, transmitted and processed. Management of sample analysis and data review is monitored in general for all the data collected.

A.2.5.8 Data Storage and Retrieval

All electronic and hard copy records associated with this UAF-APSC collaborative project produced by UAF will be retained in original form and format for a minimum of 5 years. These records include all logbooks, procedures, analyst notes, instrument calibration information, instrument output files (e.g. chromatograph output data files), raw and finished data, and data validation and verification records. Accordingly, UAF will retain the capability to re-examine and reprocess any raw data, (e.g. retain current chromatographic data processing software and its native hardware platform).

Electronic data will be archived on a removable media and placed with the associated job file folder for ease of retrieval. The data archived will be in the original commercially available format as processed (i.e. Excel®, Word®, etc.) copied electronically where feasible to CD-ROM media device supplied by APSC for the project. Copies of data pages from bound, controlled logbooks will also be placed in the respective job file folder for ease of retrieval.

A.3 ASSESSMENT/OVERSIGHT

A.3.1 Assessments and Response Actions

At the request of the UAF-APSC Project Supervisor, the APSC Analytical Services Lead (ASL) will conduct an assessment or the APSC Internal Audit Manager (IAM) (or designee) will conduct an audit of the UAF laboratory activities addressed under this QAPjP. The APSC ASL or IAM (or designee) will have the authority to issue a Stop Work Order upon finding a significant condition that would adversely affect the quality and usability of the data. The UAF Principal Investigator will have the responsibility for initiating and implanting response actions

associated with findings identified during the on-site-audit. Once the response actions have been implemented, the APSC ASL or IAM (or designee) will perform a follow-up assessment/audit to verify and document that the response actions were implemented effectively.

In-house audits performed by UAF Laboratories may be conducted in accordance with this QAPjP or its own Quality Management Procedures (QMP). When performed and APSC work is associated with the audit, APSC will receive a copy of the audit report along with accompanying information explaining any impacts (measured or estimated) on the APSC work so audited. The information will address examples of conditions indicating out-of-control situations, who is responsible for initiating the corrective actions, and what steps may be taken.

A.3.1.1 Reports to Management
Once the project is complete and the resulting data obtained, the UAF principal investigator will prepare a final project report. The report will include a summary of the activities performed during the project and present the resulting data explaining its likely meanings, draw conclusions, etc. (Statements about problems concerning data quality will be presented along with an explanation of their impact, if any, on the certainty of the data reported).

A.4 DATA VALIDATION AND USABILITY

The purpose of this element is to state the criteria for deciding the degree to which each data item has met its quality specifications. APSC will estimate the potential effect that each deviation from this QAPjP may have on the usability of the associated data item, its contribution to the quality of the reduced and analyzed data, and its effect on the decisions made from these data.

A.4.1 Data Review, Validation and Verification Requirements
UAF data will be reviewed by the project team. Second level review will be the responsibility of the UAF principal investigator or designee. The second level review will be done, in general, on all the data collected by UAF. The UAF Principal investigator will also be responsible for overall data validation and final approval of the data, in accordance with project purpose and use of the data.

A.4.2 Validation and Verification Methods
The process of data verification requires UAF to confirm by examination or provision of objective evidence that the requirements of the specified QC acceptance criteria have been met. Verification examines the result of a given activity to determine conformance to the stated requirements for that activity. For example, have the data been collected according to a specified method and have the collected data been faithfully recorded and transmitted? Do the data fulfill specified data format requirements? The UAF process of data verification must effectively ensure the accuracy of data by using validated methods and protocols. As a part of this verification process, UAF will utilize a data review and data package checklist to provide documentation of the review and ensure it meets the specified requirements.

A.4.3 Reconciliation with User Requirements

Completeness will also be evaluated to determine if the completeness goal for this project has been met. If the QC data and/or other data quality indicators do not meet the project's requirements as outlined in this QAPjP (including the accuracy of calibration materials), the data may be discarded, and retest and re-sampling, if necessary, may occur. The UAF principal investigator and project team will evaluate the cause of the failure (if possible) and make the decision to discard that data and re-sample. If the failure is tied to the analysis, calibration and maintenance techniques will be reassessed as identified by the appropriate lab personnel. If the failure is associated with the sample collection and re-sampling is needed, the samplers will be retained.

Table A.1 Data package checklist

i.   Sample Identification Number:

ii.   Type of Sample Tested: Welker Sample/Pooled Sample/Other Samples
Explain.

iii.   Type of Tests carried out on the sample:

iv.   Date and Time of Tests. List all the tests:

v.   Analyst Name:

vi.   Was the equipment functional? If no, explain the corrective steps taken.

vii.   Was the equipment calibrated properly? Was the calibration satisfactory? Explain.

viii.   Is the final data reviewed? If yes, data reviewed by:

Table A.2 Receiving Log for TAPS/COS Samples.

University of Alaska Fairbanks - Petroleum Development Laboratory						Form: SLF - 10

Receiving Log for TAPS/COS Samples

RECEIVER: Petroleum Engineering Department 437 Duckering Bldg., P. O. Box 755880 Fairbanks, AK 99775-5880		SHIPPER:			CUSTODY RECORD / REPORT TO: Project Manager: Dr. Godwin A. Chukwu (807) 474-7748
Date Received:		Carrier and Waybill Number:			Received By:

			Sample Identification Information			
#	Identification Number	Welker Cylinder No. (Only for Welker Samples)	COC Seal Number	Container Type, Size	Shipping Documents (yes/no)	Sample Condition Upon Receipt at UAF
1						
2						
3						
4						
5						
6						
7						
8						
9						
10						
11						
12						
13						
14						
15						
16						

Chain of Custody Records		(Internal UAF-PDL)		
Action Taken with Shipment		Date	Relinquished By	Received By

Table A.3 Sample Log-In and Identification of TAPS/COS Test Samples.

University of Alaska Fairbanks - Petroleum Development Laboratory						Form: SLF-100
Sample Log-in and identification of TAPS/COS Test Samples						
LABORATORY: Petroleum Engineering Department 437 Duckering Bldg., P.O. Box 755880 Fairbanks, AK 99775-5880			CUSTODY RECORD / REPORT TO: Project Manager: Godwin A. Chukwu			

Sample Identification Information			Sampling Information			Sample Condition Upon Receipt at UAF-PDL
	Cylinder No.			Date	Pressure	Container	
UAF-PDL ID No.	(Only for Walker Samples)	Contractor I.D. No.	Origin/Location	(If Applicable)	(If Applicable)	Type,Size	Cylinder/Sample Condition
Analyst Signature:			Date of Log-in:				

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Table A.4 Sample Log-In and Identification of a TAPS/COS "Pooled" Test Sample.

University of Alaska Fairbanks - Petroleum Development Laboratory					Form: SLF-200
Sample Log-In and Identification of a TAPS/COS "Pooled" Test Sample					
LABORATORY: Petroleum Engineering Department 437 Duckering Bldg., P.O. Box 755880 Fairbanks, AK 99775-5880			CUSTODY RECORD / REPORT TO: Project Manager: Dr. Godwin A. Chukwu (907) 474-7748		
Sample Identification			Sample Information		
Date	UAF-PDL ID No.	Container Type, Size	Sample Volume, ml	Description of Sample	Reason for "Pooling' Sample
Conditions during "Pooling"					
Date	"Pooling" Temperature	"Pooling" Pressure	Sample # (below)	Volume Added, ml	Comments
Identification of Samples Used To Produce "Pooled" Sample					
#	UAF-PDL Sample ID No.	Container Type, Size	Sample Volume, ml	Description of Sample	Comments
1					
2					
3					
4					
5					
6					
7					
Analyst Signature:			Date:		

Table A.5 Sample Log-In and Identification of TAPS/COS Test Aliquots.

University of Alaska Fairbanks - Petroleum Development Laboratory								Form: SLF-300
Sample Log-In and Identification of TAPS/COS Test Aliquots								
LABORATORY:	Petroleum Engineering Department				CUSTODY RECORD / REPORT TO:			
	437 Duckering Bldg., P.O. Box 755880				Project Manager: Dr. Godwin A. Chukwu			
	Fairbanks, AK 99775-5880				(907) 474-7748			
Initial Sample Identification								
UAF-PDL ID No.			Contractor ID No.			Sample Description		
Reconditioning and Aliquoting Conditions								
Date		Analyst Signature		Equil. Temp	Equil. Time		Pressure	Comments
Test Aliquot Identification								
Aliquot ID No.		Aliquot Volume		Container Type & Size			Designated Test & Comments	

Table A.6 Chain of Custody Record for TAPS/COS Test Samples.

University of Alaska Fairbanks - Petroleum Development Laboratory									Form: COC-100
Chain of Custody Record for TAPS/COS Test Samples									
LABORATORY: Petroleum Engineering Department 437 Duckering Bldg., P.O. Box 755880 Fairbanks, AK 99775-5880				CUSTODY RECORD / REPORT TO: Project Manager. Dr. Godwin A. Chukwu					
Sample Description:									

	Sample Identification Information			Field Sampling Information				Sample Condition Upon Receipt at UAF-PDL	
		Cylinder No.			Date	Pressure	Container	Cylinder/Sample Condition	
Custodian	UAF-PDL ID No.	(Only for Walker Samples)	Contractor I.D. No.	Origin/Location	(If Applicable)	(If Applicable)	Type, Size		

Cylinder/Sample History

Date	Cylinder/Sample Location	Action taken with Sample & comments		Relinquished By		Received By	

Final Action:	Action By:		Date:

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Table A.7 Chain of Custody Record for TAPS/COS "Pooled" Test Sample.

University of Alaska Fairbanks - Petroleum Development Laboratory						Form: COC-200
Chain of Custody Record for TAPS/COS "Pooled" Test Sample						
LABORATORY:				CUSTODY RECORD / REPORT TO:		
Petroleum Engineering Department				Project Manager: Dr. Godwin A. Chukwu		
437 Duckering Bldg., P.O. Box 755880				Petroleum Engineering Department		
Fairbanks, AK 99775-5880				437 Duckering Bldg., P.O. Box 755880		
				Fairbanks, AK 99775-5880		
"Pooled" Sample Information						
Custodian	UAF-PDL ID No.	Container Type	Sample Volume	Sample Description		Reason for "Pooling" Sample
Sample History						
Date	"Pooled" Sample Location		Action taken with "Pooled" Sample		Relinquished By	Received By
Final Action:				Action By:		Date:

Table A.8 Chain of Custody Record for TAPS/COS Test Aliquots.

University of Alaska Fairbanks - Petroleum Development Laboratory					Form: COC-300
Chain of Custody Record for TAPS/COS Test Aliquots					
LABORATORY:	Petroleum Engineering Department		CUSTODY RECORD / REPORT TO:		
	437 Duckering Bldg., P.O. Box 755880		Project Manager: Dr. Godwin A. Chukwu		
	Fairbanks, AK 99775-5880				
Sample Description:					
Sample Identification					
Aliquot ID No.	Container Type & Size	Aliquot Volume, ml	Custodian (Analyst)	Date of Aliquoting	
Sample History					
Date	Aliquot Location	Action taken with aliquot & comments	Relinquished By	Received By	
Final Action:			Action By:	Date:	

Table A.9 Shipping Log for TAPS/COS Samples.

University of Alaska Fairbanks - Petroleum Development Laboratory					Form: SLF - 20
Shipping Log for TAPS/COS Samples					

SHIP TO:		SHIPPER: Petroleum Engineering Department 437 Duckering Bldg., P.O. Box 755880 Fairbanks, AK 99775-5880			CUSTODY RECORD / REPORT TO: Project Manager: Dr. Godwin A. Chukwu (907) 474-7748
Date Shipped:		Carrier and Waybill Number:			Shipped By:

			Sample Identification Information			
#	UAF-PDL ID	Welker Cylinder No. (Only for Welker Samples)	Sample Description	Container Type, Size	Shipping Documents (yes/no)	Sample Condition Upon Leaving UAF-PDL
1						
2						
3						
4						
5						
6						
7						
8						
9						
10						
11						
12						
13						
14						
15						
16						

Chain of Custody Records (Internal UAF-PDL)

Action Taken with Shipment	Date	Relinquished By	Received By

ATTACHMENT 1

Evaluation of GTL #1 and Crude Oil Blends
For Yield Stress Values

A REPORT BY WESTPORT TEHCNOLOGY CENTER

WESTPORT
TECHNOLOGY CENTER INTERNATIONAL

RT-01-005   January 2001

Evaluation of GTL and Crude Oil Blends
For Yield Point Values

Final Report

Work By:
S. Brown, N. Magri, R. Collins

Prepared for Department of Petroleum Engineering, University of Alaska   W.O.# M10327

Table of Contents

Introduction

The gel strength of various GTL (gas to liquid samples) and North Slope crude oil blends were determined by the rotating vane method. The tests for determining the yield stress, or yield point, of the cooled crude oil blends, were performed following Westport's Standard Laboratory Procedure (SLP) 307, "Crude Oil Yield Point Determination by Vane Viscometry". This determination was made with Brookfield rotary viscometers and vane spindles, which extend horizontally through a sample, minimizing the impact of slippage at the spindle wall. This method determines the minimum amount of torque necessary to initiate oil movement at low shear, and subsequent gel breakdown after initiation of flow. These data can be directly used in modeling of crude oil behavior in pipelines, during start-up conditions.

Additionally, extended gas chromatography was performed to define the quantitative composition from C2 through C30+ on samples (a) and (b) listed below and on GTL plus crude oil blends (i) through (iv).

Test Results

Twenty-four test samples were prepared by weight for testing, four each at the six ratios listed below:

Initially, 2 samples of Wax Distillate:GTL mixes were prepared as follows:

(a). 25% wax distillate + 75% Light Hydrocarbon GTL
(b). 50% wax distillate + 50% Light Hydrocarbon GTL

Blended samples of (a) and (b) with crude oil in the following ratios, respectively:

(i). Sample (a) + crude oil in the ratio of 1:4
(ii). Sample (a) + crude oil in the ratio of 1:3
(iii). Sample (b) + crude oil in the ratio of 1:4
(iv). Sample (b) + crude oil in the ratio of 1:3

Blended Light Hydrocarbon GTL sample with crude oil in the following ratios:

(v). LH sample + Crude oil in the ratio of 1:4
(vi). LH sample + crude oil in the ratio of 1:3

The client supplied the wax and light hydrocarbon GTL samples. The crude oil sample was supplied by the Alyeska Pipeline Service Company taken from the flowing TAPS mixed stream at Pump Station 1. The paraffin wax was separated from Shellwax® 200 by a modified ASTM-1160 Vacuum Distillation process to produce only a 20% overhead fraction. This wax fraction was then mixed with the light hydrocarbon GTL liquid as outlined above for samples (a) and (b). All blend mixes were carried out by weight to weight measurements.

Samples were tested at selected temperatures as the crude oil blends were cold ramped from 90°F to -20°F over a twenty-one day period. The maximum recorded torque obtained during vane rotation at a constant speed of 0.01 rpm was converted into a yield stress value. The summary of vane test results is presented in Table 1 and Figures 1 and 2. Table 2 presents the C2 to C30+

data from quantitative gas chromatography of samples (i) through (iv). The GC data is also presented in graphical format on pages 36 – 41 of Section 2.

Initially, test temperatures were set at 9°, 0° and –20°F. However, testing at 9°F (Tests 1-8) indicated gel strengths beyond the measurable limits of the viscometers for samples (i) through (iv), and relatively high strengths with the GTL/Crude oil blends (v) and (vi) containing no wax distillate material. The cold ramp was continued and testing resumed (Tests 9 and 10) at 0°F for only samples (v) and (vi).

Due to the high yield stress values encountered it was decided to drop testing at –20°F and test the remaining samples at higher temperatures in an attempt to determine gelation onset and buildup. Therefore, all remaining samples, sealed in their closed cups were re-heated to 150°F in the environmental chambers for several hours. slowly decreased in temperature to 90°F, then cold ramping re-established on the 21-day ramp cycle.

Tests 11 and 12 were performed at 60°F on the higher content wax/GTL samples (iii) and (iv) resulting in 0.07 and 0.0 dynes/sqcm, respectively. The other samples were not tested in an effort to conserve sample quantity.

Tests 13 through 18 were performed at 40°F on samples (i), (ii), (iii) and (iv). Tests 13 and 15 resulted in maximum torque readings beyond the selected viscometer limits. Repeats were performed after changing the viscometer to one with a higher spring rating. The measured yield stresses were 20.7, 63.7, 539 and 1192 dynes/sqcm with respect to sample number.

Test 19 through 23 were performed at 20°F on samples (i). (ii), (iv), (v) and (vi). Test 23 on sample (v) was beyond the strongest viscometer's limit. and the yield stress can only be reported as greater than 1589 dyne/sqcm. The trend of higher yield stresses with higher wax content was again observed. Samples (i) and (ii) had yield stresses of 739 and 1384 dyne/sqcm. The blends of GTL and crude oil showed gel onset with yield stresses of 71 and 92 dyne/sqcm for samples (v) and (vi). respectively.

There was some variability seen with sample (vi) on Tests 3 and 8 at 9°F, (229 versus 438 dyne/sqcm) that could not be attributed to procedural variation. This is not uncommon when weak gel structures have formed. If sample quantity had been higher, a third repeat test would have been warranted. Test 8 results fall within the data trend having a higher yield value in comparison with Test 2 (364 dyne/sqcm), which had a lower wax/GTL ratio; therefore a high degree of confidence can be associated with the Test 8 yield stress value even without a backup test result.

TABLE 1

Yield Stress Data for GTL & Crude Oil Blendes

Test Sample (mixture reference)	Test Number (Sequence)	Brookfield Viscometer Model	Temperature Temperature (°F)	Yield Point Maximum Torque (dyne-cm)	Yield Stress (dynes/sqcm)
(i)	14	RV	40	747	20.7
(i)	21	HB	20	26736	739
(i)	7	HB	6	>57496	>1589
(ii)	13	LV	40	>674	>18.6
(ii)	18	RV	40	2307	63.7
(ii)	22	HB	20	50079	1384
(ii)	6	HB	6	>57496	>1589
(iii)	12	LV	60	2.69	0.07
(iii)	15	RV	40	>7187	>199
(iii)	16	HB	40	19491	539
(iii)	5	HB	6	>57496	>1589
(iv)	11	HB	60	0.0	0
(iv)	17	HB	40	43122	1192
(iv)	23	HB	20	>57496	>1589
(iv)	4	HB	6	>57496	>1589
(v)	20	HB	20	2587	71
(v)	1	RV	6	>7187	>199
(v)	2	HB	6	13167	364
(v)	10	HB	0	24206	669
(vi)	19	HB	20	3335	92
(vi)	3	HB	6	8279*	229*
(vi)	8	HB	6	15869	438
(vi)	9	HB	0	22366	618

* Test results appears to be anomalous in comparison to data trends.

(i)   (25% wax distillate + 75% GTL) + Crude Oil in the ratio of 1:4
(ii)   (25% wax distillate + 75% GTL) + Crude Oil in the ratio of 1:3
(iii)   (50% wax distillate + 50% GTL) + Crude Oil in the ratio of 1:4
(iv)   (50% wax distillate + 50% GTL) + Crude Oil in the ratio of 1:3
(v)   GTL + Crude Oil in the ratio of 1:4
(vi)   GTL + Crude Oil in the ratio of 1:3

The precision of the Vane test method and results, based on running multiple samples can be stated as:

Repeatability (r) - The difference between successive results obtained by the same operator in the same laboratory with the same apparatus under constant operating conditions on identical test material would, in the normal and correct operation of this method, have a relative standard deviation at or below 15%.

Figure 1: Yield Stress vs Temperature
GLT & Crude Oil Blends

Figure 2: Yield Stress vs Temperature
GLT & Crude Oil Blends
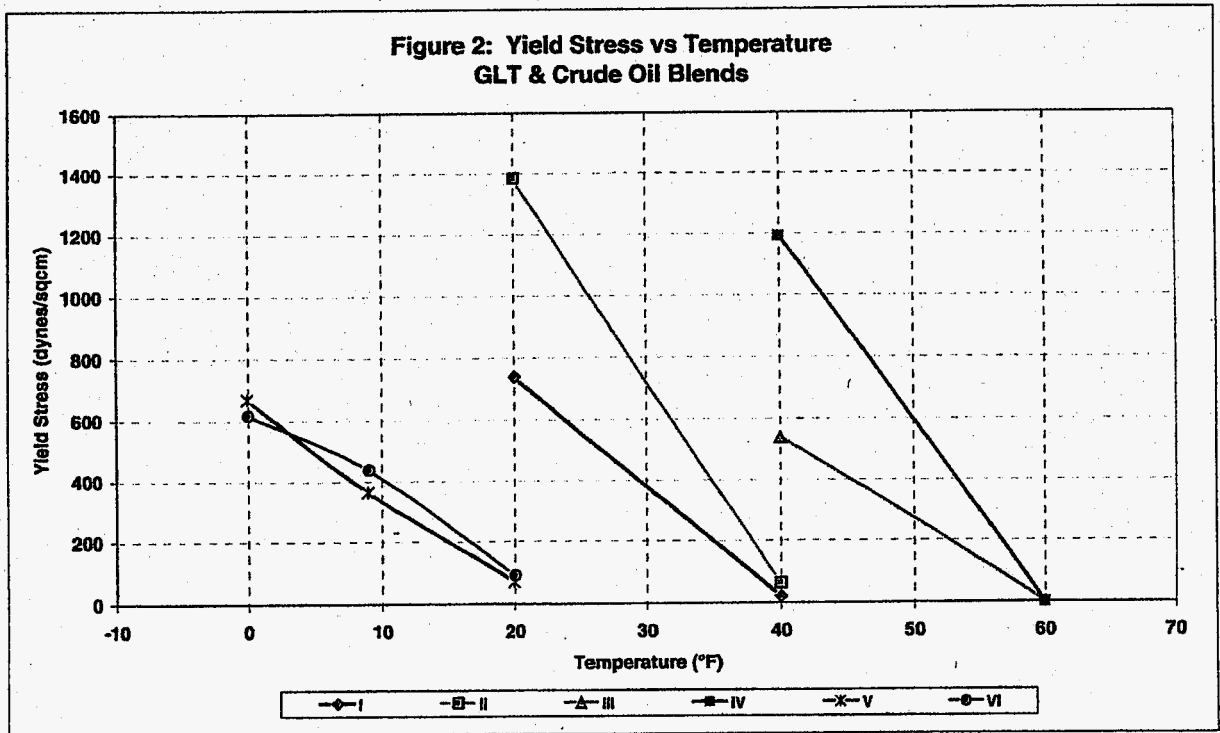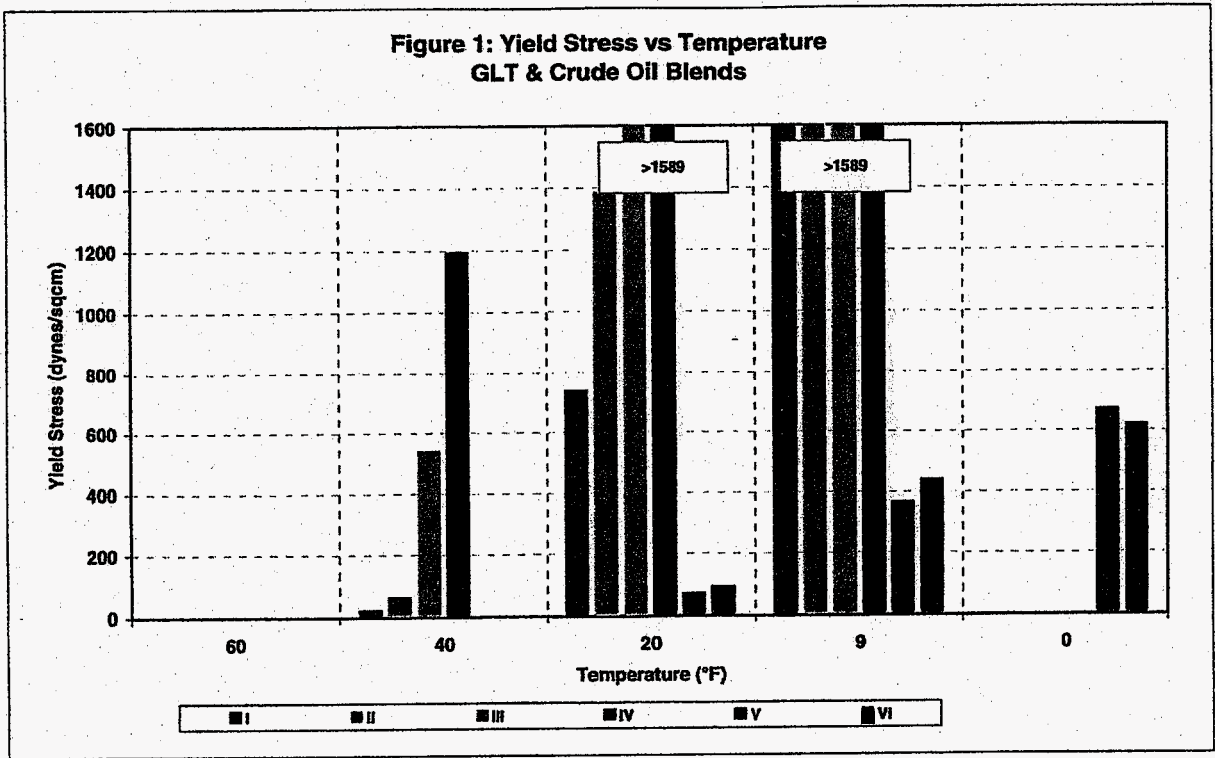
Table 2: Quantitative Composition of Blends by Capillary Gas Chromatography

Sample	Gas to liquids product (GTL)	25% Wax / 75% GTL	50% Wax/ 50% GTL		TAPS Mix Crude Oil @ PS-1	25:75 Wax/GTL 80g @ 320g oil	25:75 Wax/GTL 100g @ 300g oil	50:50 Wax/GTL 80g @ 320g oil	50:50 Wax/GTL 100g @ 300g oil
ChemStation File	J261N.D	I865N.D	I866N.D		J260REG.D	I861ISO.D	I862ISO.D	I864ISO.D	I863ISO.D
Components	Wt%	Wt%	Wt%		Wt%	Wt%	Wt%	Wt%	Wt%
C2	0.002	0.003	0.003		0.009	0.000	0.001	0.002	0.001
C3	0.145	0.107	0.066		0.167	0.033	0.050	0.088	0.069
IC4	0.003	0.011	0.007		0.259	0.128	0.149	0.210	0.160
NC4	1.215	0.904	0.245		0.954	0.734	0.852	0.951	0.797
IC5	0.146	0.110	0.069		0.630	0.502	0.508	0.568	0.484
NC5	2.972	2.212	1.367		0.967	1.098	1.195	1.074	1.015
C6	7.826	5.800	3.901		1.836	2.758	3.050	2.443	2.463
BENZENE	0.003	0.002	0.001		0.340	0.270	0.255	0.271	0.244
C7	8.889	6.633	4.167		3.358	3.904	4.060	3.509	3.476
TOLUENE	0.029	0.022	0.016		0.792	0.623	0.588	0.621	0.565
C8	8.764	6.570	4.147		4.078	4.572	4.662	4.111	4.092
C9	8.539	6.384	4.071		4.115	4.506	4.579	4.042	4.048
C10	7.940	6.230	3.972		3.715	4.121	4.202	3.696	3.737
C11	7.290	5.607	3.989		3.174	3.663	3.760	3.291	3.367
C12	6.446	5.241	3.827		2.989	3.403	3.490	3.112	3.208
C13	5.748	4.908	3.854		3.308	3.584	3.640	3.360	3.413
C14	5.065	4.610	3.942		3.160	3.421	3.492	3.293	3.367
C15	4.414	4.367	4.101		3.108	3.312	3.359	3.266	3.342
C16	3.811	4.189	4.346		2.757	3.042	3.105	3.083	3.194
C17	3.273	4.096	4.699		2.636	2.914	2.985	3.047	3.184
C18	2.799	4.071	5.119		2.590	2.919	2.991	3.145	3.292
C19	2.389	4.070	5.522		2.543	2.847	2.923	3.154	3.329
C20	2.026	4.001	5.753		2.296	2.653	2.747	3.050	3.230
C21	1.705	3.795	5.674		2.134	2.493	2.571	2.876	3.079
C22	1.421	3.422	5.236		2.037	2.324	2.409	2.711	2.894
C23	1.165	2.914	4.511		1.898	2.122	2.169	2.456	2.600
C24	0.939	2.353	3.650		1.781	1.909	1.948	2.180	2.286
C25	0.747	1.816	2.804		1.669	1.723	1.744	1.929	1.984
C26	0.607	1.369	2.086		1.624	1.572	1.592	1.718	1.757
C27	0.583	1.094	1.584		1.557	1.470	1.478	1.563	1.570
C28	0.433	0.764	1.104		1.532	1.375	1.378	1.437	1.422
C29	0.247	0.458	0.710		1.482	1.249	1.274	1.301	1.270
C30+	2.418	1.866	5.457		34.506	28.757	26.796	28.444	27.061
Total	100.000	100.000	100.000		100.000	100.000	100.000	100.000	100.000

Wt% Wax	0.00	50.00	25.00		0.00	5.00	6.25	10.00	12.50
Wt% GTL	100.00	50.00	75.00		0.00	15.00	18.75	10.00	12.50
Wt% Crude	0.00	0.00	0.00		100.00	80.00	75.00	80.00	75.00

SECTION 1

Yield Stress versus Elapsed Time
For
GTL & Crude Oil Blends

Summary of Test Method

Westport's SLP-307 consists of determining the yield point of a crude oil by measuring the torque on a spindle, using a Brookfield viscometer, rotating at 0.01 rpm in the material. The spindle to be used consists of four rectangular vanes dimensioned (0.75"w x 2.25"h) and oriented at 90 degree increments around the central axis. The sample cup is dimensioned (1.5" id x 4.0"h). Vertical orientation of vanes within the sample cup is dimensioned (1.00" from top and 0.75" from bottom).

The crude oil blends were initially heated to 150°F to destroy all temperature and shear histories and then cooled to 100°F at which point they were loaded into the vane closed-cup apparatus. The closed-cup apparatus holds the vanes rigidly during cooling and aging and prevents loss of light ends through evaporation. After loading into the cup apparatus the samples were cooled in an environmental chamber at a controlled rate to below 0°F. The cooling rate mimics the expected rate of cooling of the Trans-Alaska pipeline oil in the case of shut-in.

Samples were withdrawn from the environmental chamber at five test temperatures (approximately 60, 40, 20, 9 and 0°F) and transferred to a refrigerated circulator that maintains the sample at test temperature. The spindle was attached to the Brookfield viscometer (LV, RV or HB) before the spindle clamping mechanism was released. The clamping mechanism was released and the viscometer was started at 0.01 rpm and torque as a function of time was measured, until a maximum reading was obtained. The maximum torque (dyne/cm) obtained is divided by a vane parameter constant K to obtain the yield stress (dynes/sqcm). The constant K is calculated based on the dimensions of the vanes. (K=36.19 cm^3, for a Vane with D=0.75 inch (1.905 cm) and H=2.25 inch (5.715 cm). For further detailed information Westport's SLP-307 is attached in Appendix B, page 79.

Temperature Ramping Profile

The temperature decay curve used for test sample preparation was taken from Trans-Alaska Pipeline cold restart data supplied by the Alyeska Pipeline Service Company. Based on this curve, selective temperatures were entered into the program menu of the environmental cooling chamber, a cryogenic chamber cooled by liquid Nitrogen vapor. Temperatures are recorded on strip chart display with digital inscriptions at 12-hour intervals. The programmed temperature ramp is presented in Table 3 and Figure 3, along with the selected test points.

Table 3: Test Temperature Profile
VANE VISCOMETRY

Environmental Chamber Temperature Ramp Program				
Step #	Days	Hours	Temp °F	Temp °C
0	0	0	90	32.2
1	2.5	60	70	21.1
2	5.0	120	50	10.0
3	7.5	180	35	1.7
4	10.0	240	20	-6.7
5	12.5	300	10	-12.2
6	15.0	360	0	-17.8
7	17.5	420	-10	-23.3
8	21.0	504	-20	-28.9
9	Hold	576	-20	-28.9

App. Test Temp.	60	40	20	9	0	-20
Samples Tested	(iii, iv)	(i, ii, iii, iv)	(i, ii, iv, v, vi)	(i, ii, iii, iv, v, vi)	(v, vi)	none

Figure 3: Test Temperature Profile
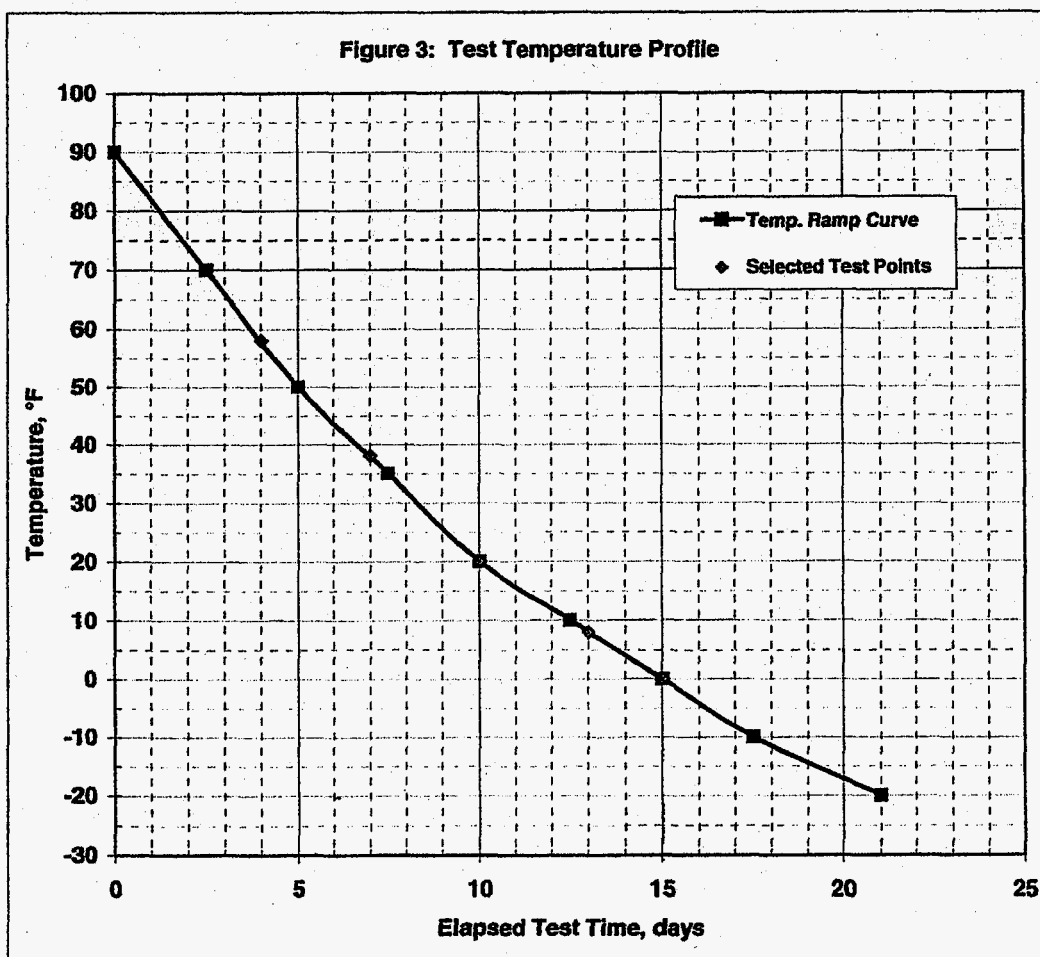
Determination of Yield Point

Figures 4 through 26 on the following pages present torque buildup versus elapsed test time. The yield point is determined from the maximum torque response, usually followed by a decline indicating any gel structure present was broken by the applied stress and degrades under continued shearing. In most tests, several minutes of "no torque" response at test initiation are recorded; this response is associated with the time for the 'S'-hook connections to "tighten" before movement, or stress, is applied to the vane shaft. One test sample was lost during aging. Numerical data for the remaining twenty-three tests are presented in Appendix A, pages 42 - 78.

Sample (i):   (25% wax distillate + 75% GTL) + Crude Oil in a ratio of 1:4

Figure 4: Test 14

Figure 4 presents torque response (dyne/cm) versus elapsed test time at a test temperature of 40°F. The test was performed with Sample (i) on the Brookfield RV viscometer. A maximum torque of 747 dyne/cm was recorded giving a yield point of 20.7 dynes/sqcm.

Figure 5: Test 21

Figure 5 presents torque (dyne/cm) versus elapsed test time at a test temperature of 20°F. The test was performed with Sample (i) on the Brookfield HB viscometer. A maximum torque of 26736 dyne/cm was recorded giving a yield point of 739 dynes/sqcm.

Figure 6: Test 7

Figure 6 presents torque (dyne/cm) versus elapsed test time at a test temperature of 9°F. The test was performed with Sample (i) on the Brookfield HB viscometer. A maximum torque limit of the HB viscometer was reached (57496 dyne/cm); therefore the yield point was greater than 1589 dynes/sqcm. The HB viscometer has the highest rated spring torque available for this testing.

Sample (ii):   (25% wax distillate + 75% GTL) + Crude Oil in a ratio of 1:3

Figure 7: Test 13

Figure 7 presents torque response (dyne/cm) versus elapsed test time at a test temperature of 40°F. The test was performed with Sample (ii) on the Brookfield LV viscometer. A maximum torque limit of the LV viscometer was reached (674 dyne/cm); therefore the yield point was greater than 18.6 dynes/sqcm. The LV viscometer has the lowest rated spring torque available for this testing. The data acquisition program failed to initialize during the first seven minutes of this test, as can be seen by the gap in data points at the test start. A second sample was tested on the RV viscometer in Test 18.

Figure 8: Test 18

Figure 8 presents torque (dyne/cm) versus elapsed test time at a test temperature of 40°F. The test was performed with Sample (ii) on the Brookfield RV viscometer. A maximum torque of 2307 dyne/cm was recorded giving a yield point of 63.7 dynes/sqcm.

Figure 9: Test 22

Figure 9 presents torque (dyne/cm) versus elapsed test time at a test temperature of 20°F. The test was performed with Sample (ii) on the Brookfield HB viscometer. A maximum torque of 50079 dyne/cm was recorded giving a yield point of 1384 dynes/sqcm.

Figure 10: Test 6

Figure 10 presents torque (dyne/cm) versus elapsed test time at a test temperature of 9°F. The test was performed with Sample (ii) on the Brookfield HB viscometer. A maximum torque limit of the HB viscometer was reached (57496 dyne/cm); therefore the yield point was greater than 1589 dynes/sqcm. The HB viscometer has the highest rated spring torque available for this testing.

Sample (iii):   (50% wax distillate + 50% GTL) + Crude Oil in a ratio of 1:4

Figure 11: Test 12

Figure 11 presents torque response (dyne/cm) versus elapsed test time at a test temperature of 60°F. The test was performed with Sample (iii) on the Brookfield LV viscometer. A maximum torque of 2.69 dyne/cm was recorded giving a yield point of 0.07 dynes/sqcm. At this temperature there was very little, if any, gel structure present.

Figure 12: Test 15

Figure 12 presents torque (dyne/cm) versus elapsed test time at a test temperature of 40°F. The test was performed with Sample (iii) on the Brookfield RV viscometer. A maximum torque limit of the RV viscometer was reached (7187 dyne/cm); therefore the yield point was greater than 199 dynes/sqcm. A second sample was tested on the HB viscometer in Test 16.

Figure 13: Test 16

Figure 13 presents torque (dyne/cm) versus elapsed test time at a test temperature of 40°F. The test was performed with Sample (iii) on the Brookfield HB viscometer. A maximum torque of 19491 dyne/cm was recorded giving a yield point of 539 dynes/sqcm.

Figure 14: Test 5

Figure 14 presents torque (dyne/cm) versus elapsed test time at a test temperature of 9°F. The test was performed with Sample (iii) on the Brookfield HB viscometer. A maximum torque limit of the HB viscometer was reached (57496 dyne/cm); therefore the yield point was greater than 1589 dynes/sqcm. The HB viscometer has the highest rated spring torque available for this testing.

Sample (iv):   (50% wax distillate + 50% GTL) + Crude Oil in a ratio of 1:3

Figure 15: Test 11

Figure 15 presents torque response (dyne/cm) versus elapsed test time at a test temperature of 60°F. The test was performed with Sample (iv) on the Brookfield HB viscometer. The selection of the HB viscometer for this test was not appropriate and no torque response was recorded. At this temperature there was very little, if any, gel structure present. With the limited number of samples no repeat test was performed at this temperature.

Figure 16: Test 17

Figure 16 presents torque (dyne/cm) versus elapsed test time at a test temperature of 40°F. The test was performed with Sample (iv) on the Brookfield HB viscometer. A maximum torque of 43122 dyne/cm was recorded giving a yield point of 1192 dynes/sqcm.

Figure 17: Test 23

Figure 17 presents torque (dyne/cm) versus elapsed test time at a test temperature of 20°F. The test was performed with Sample (iv) on the Brookfield HB viscometer. A maximum torque limit of the HB viscometer was reached (57496 dyne/cm); therefore the yield point was greater than 1589 dynes/sqcm. The HB viscometer has the highest rated spring torque available for this testing.

Figure 18: Test 4

Figure 18 presents torque (dyne/cm) versus elapsed test time at a test temperature of 9°F. The test was performed with Sample (iv) on the Brookfield HB viscometer. A maximum torque limit of the HB viscometer was reached (57496 dyne/cm); therefore the yield point was greater than 1589 dynes/sqcm. The HB viscometer has the highest rated spring torque available for this testing.

Sample (v):   GTL + Crude Oil in a ratio of 1:4

Figure 19: Test 20

Figure 19 presents torque response (dyne/cm) versus elapsed test time at a test temperature of 20°F. The test was performed with Sample (v) on the Brookfield HB viscometer. A maximum torque of 2587 dyne/cm was recorded giving a yield point of 71 dynes/sqcm.

Figure 20: Test 1

Figure 16 presents torque (dyne/cm) versus elapsed test time at a test temperature of 9°F. The test was performed with Sample (v) on the Brookfield RV viscometer. A maximum torque limit of the RV viscometer was reached (7187 dyne/cm); therefore the yield point was greater than 199 dynes/sqcm. A second sample was tested on the HB viscometer in Test 2.

Figure 21: Test 2

Figure 17 presents torque (dyne/cm) versus elapsed test time at a test temperature of 9°F. The test was performed with Sample (v) on the Brookfield HB viscometer. A maximum torque of 13167 dyne/cm was recorded giving a yield point of 364 dynes/sqcm.

Figure 22: Test 10

Figure 18 presents torque (dyne/cm) versus elapsed test time at a test temperature of 0°F. The test was performed with Sample (v) on the Brookfield HB viscometer. A maximum torque of 24206 dyne/cm was recorded giving a yield point of 669 dynes/sqcm.

Sample (vi):   GTL + Crude Oil in a ratio of 1:3

Figure 23: Test 19

Figure 23 presents torque response (dyne/cm) versus elapsed test time at a test temperature of 20°F. The test was performed with Sample (vi) on the Brookfield HB viscometer. A maximum torque of 3335 dyne/cm was recorded giving a yield point of 92 dynes/sqcm.

Figure 24: Test 3

Figure 24 presents torque (dyne/cm) versus elapsed test time at a test temperature of 9°F. The test was performed with Sample (vi) on the Brookfield HB viscometer. A maximum torque of 8279 dyne/cm was recorded giving a yield point of 229 dynes/sqcm. This result was unexpectedly lower than Test 2 (364 dynes/sqcm), which had a lower GTL ratio. Therefore, for quality purposes a second sample was tested at this temperature in Test 8.

Figure 25: Test 8

Figure 25 presents torque (dyne/cm) versus elapsed test time at a test temperature of 9°F. The test was performed with Sample (vi) on the Brookfield HB viscometer. A maximum torque of 15869 dyne/cm was recorded giving a yield point of 438 dynes/sqcm. This result followed the data trend of having a higher yield point as GTL ratios increased.

Figure 26: Test 9

Figure 26 presents torque (dyne/cm) versus elapsed test time at a test temperature of 0°F. The test was performed with Sample (vi) on the Brookfield HB viscometer. A maximum torque of 22366 dyne/cm was recorded giving a yield point of 618 dynes/sqcm. This result was lower than Test 10 with Sample (v), but without repeat tests for data assurance Test 9 results are well within the 10-15% variability range for the Vane test.

Figure 4: Sample (i) at 40°F

Yield Point by Vane Viscometry
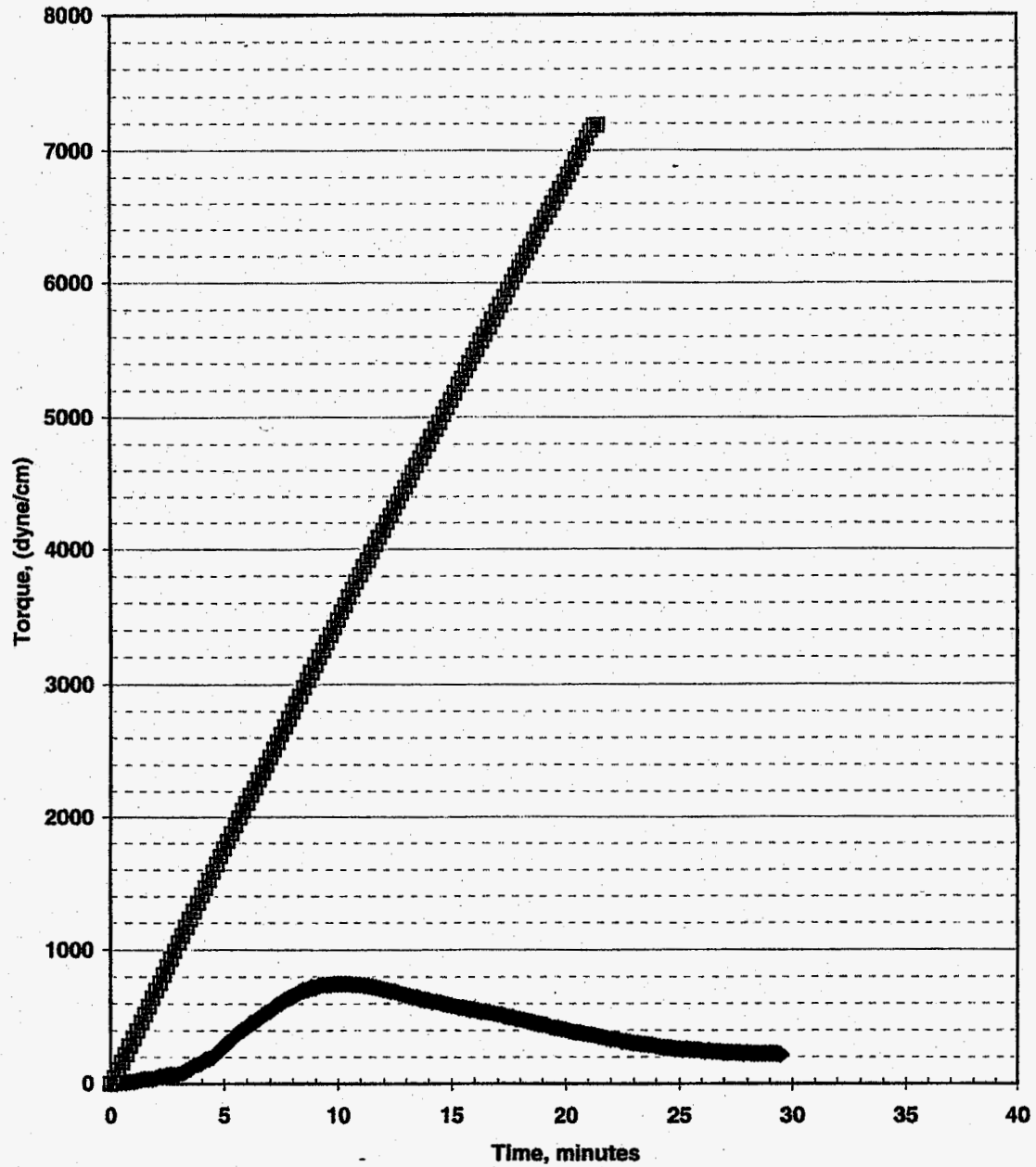
Sample: 25wax:75lt hc @ 1:4 crude oil ratio 40°F

Locked Spindle

Figure 5: Sample (i) at 20°F

Yield Point by Vane Viscometry

Sample: 25wax:75lt hc @ 1:4 crude oil ratio 20°F

Locked Spindle

Figure 6: Sample (i) at 9°F

Yield Point by Vane Viscometry

Legend:
- Sample: 25wax:75lt hc @ 1:4 crude oil ratio 9°F
- Locked Spindle
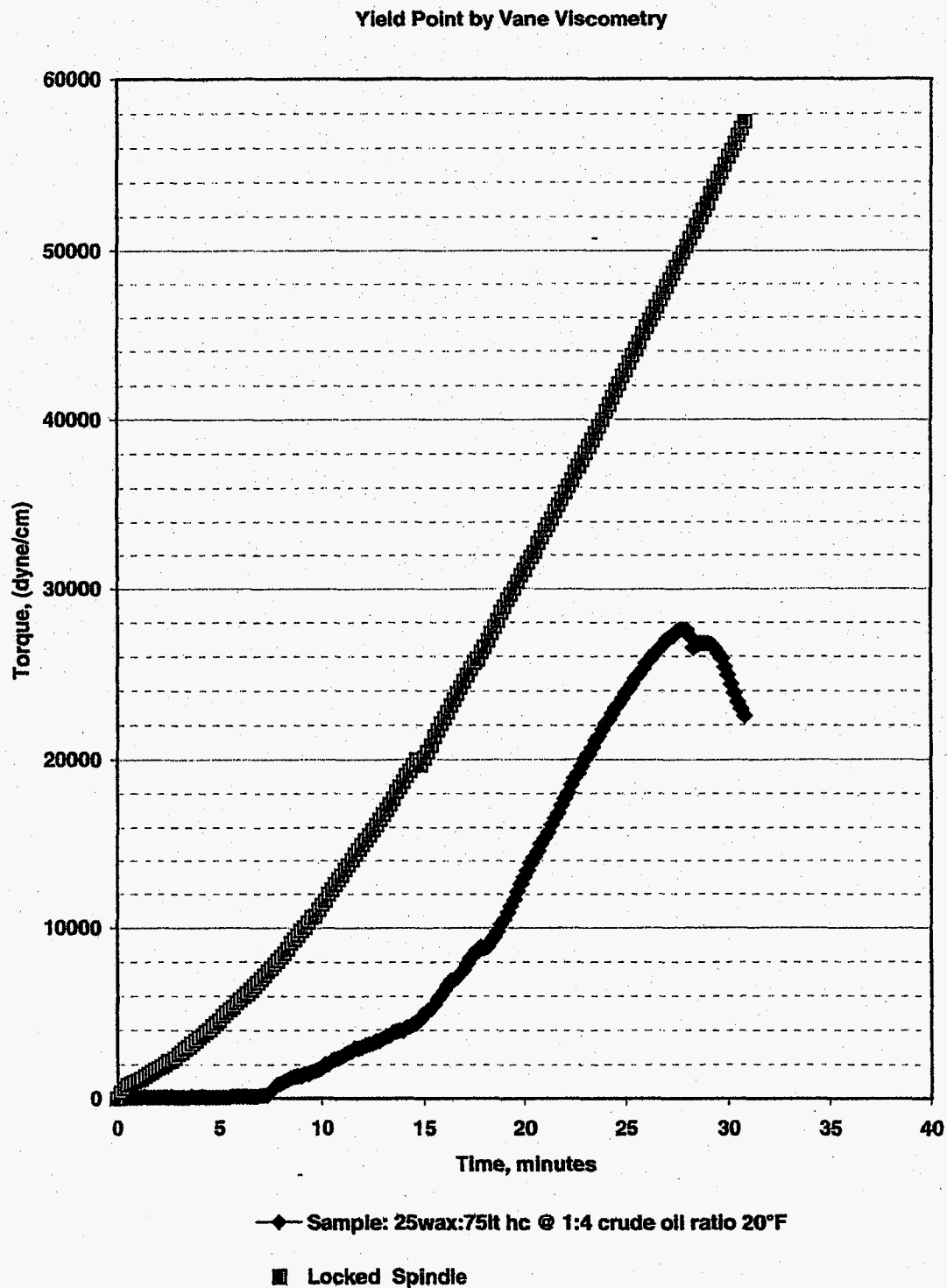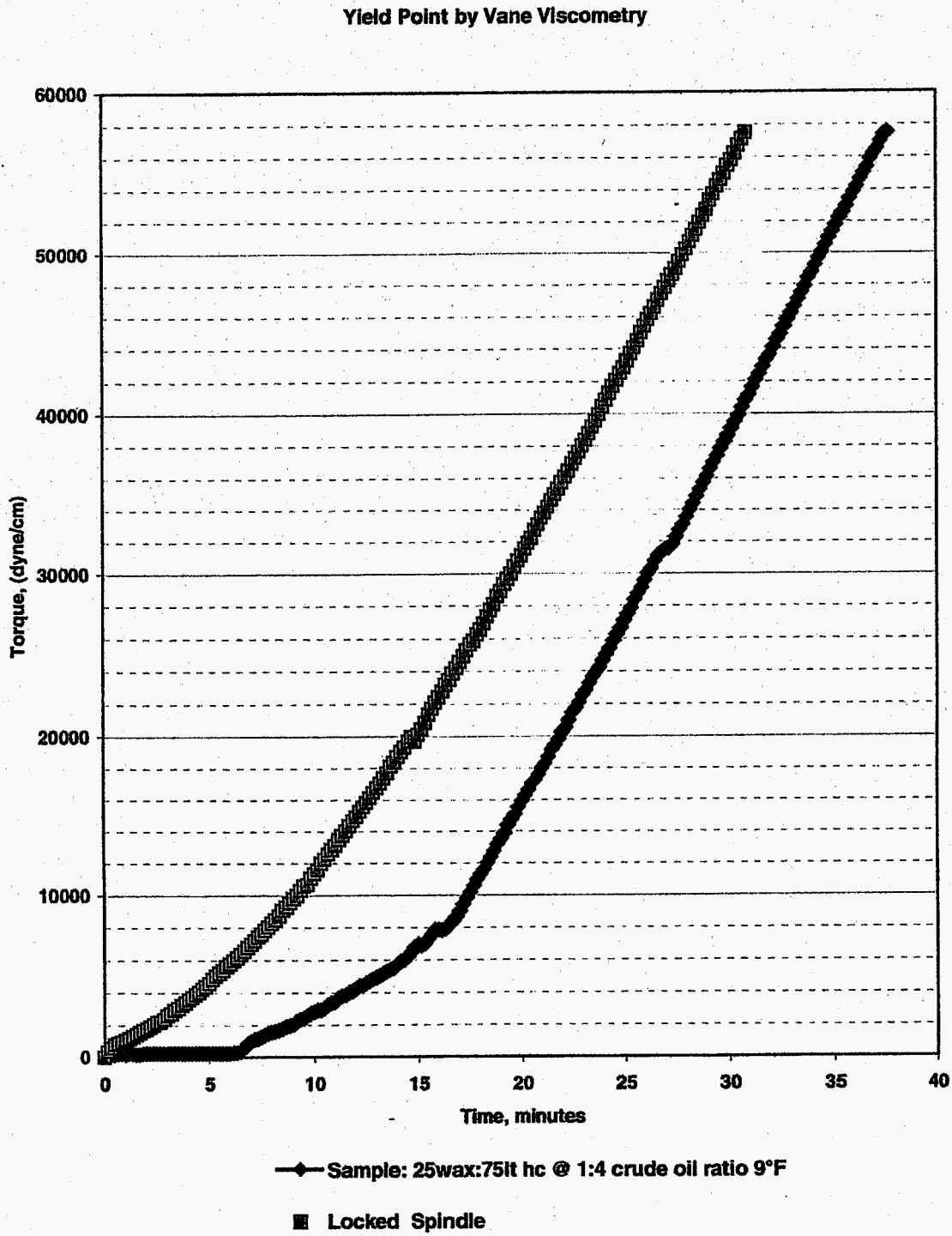
Figure 7: Sample (ii) at 40°F

Yield Point by Vane Viscometry

Sample: 25wax:75lt hc @ 1:3 crude oil ratio 40°F Vane

Locked Spindle

Figure 8: Sample (ii) at 40°F

Yield Point by Vane Viscometry

—♦—Sample: 25wax:75GTL @ 1:3 crude oil ratio 40°F Vane

▩ Locked Spindle
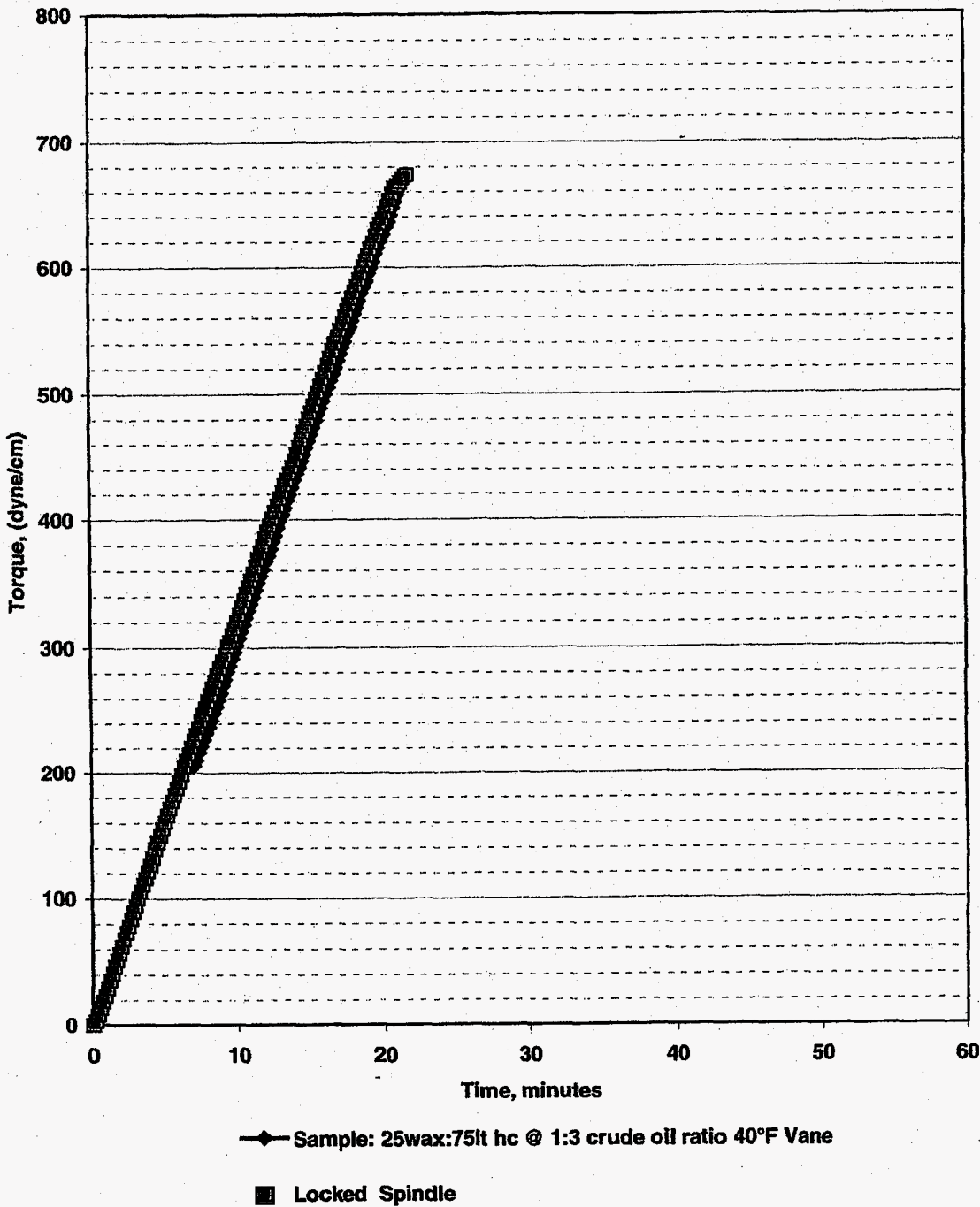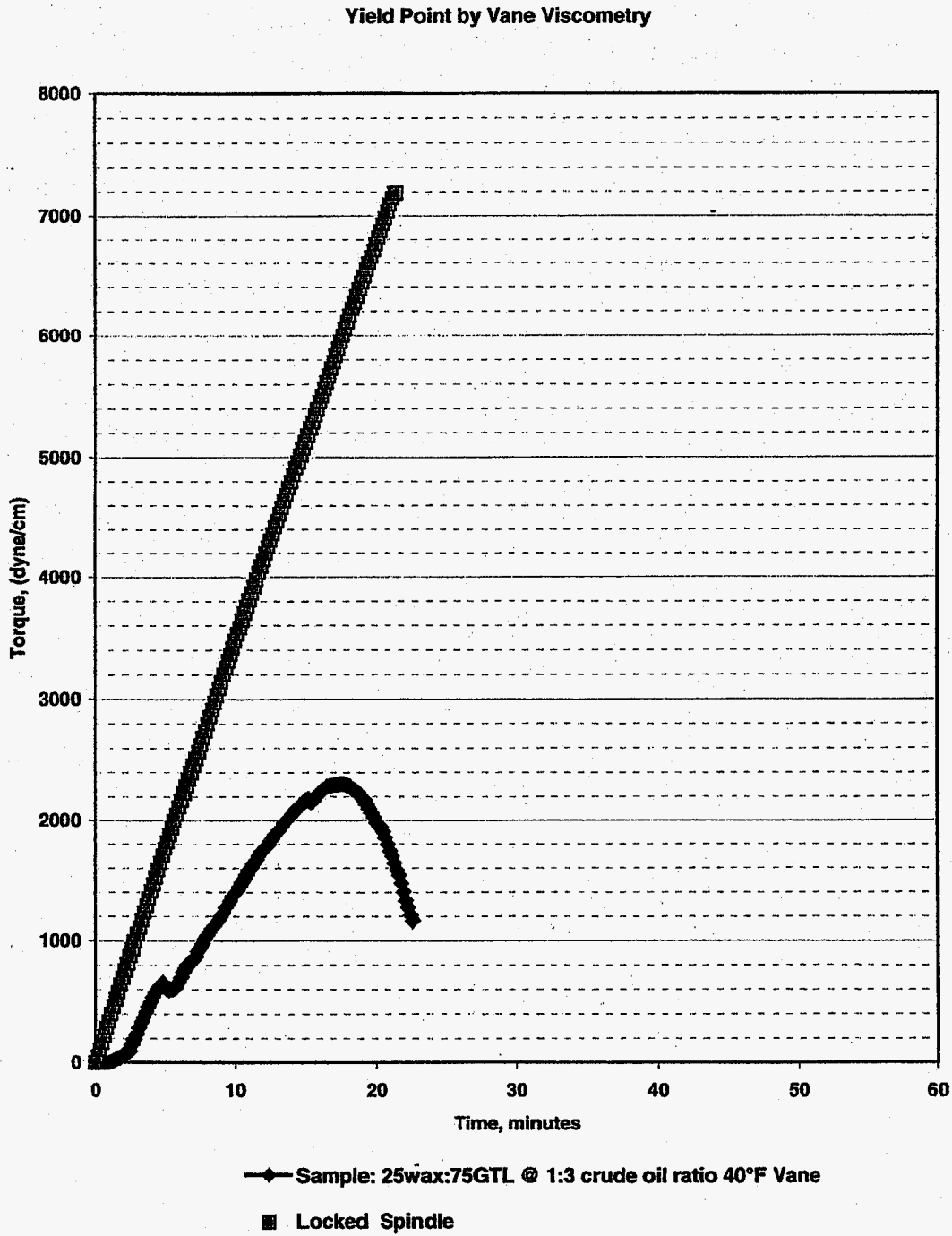
Figure 9: Sample (ii) at 20°F

Yield Point by Vane Viscometry

Sample: 25wax:75lt hc @ 1:3 crude oil ratio 20°F Vane

■ Locked Spindle
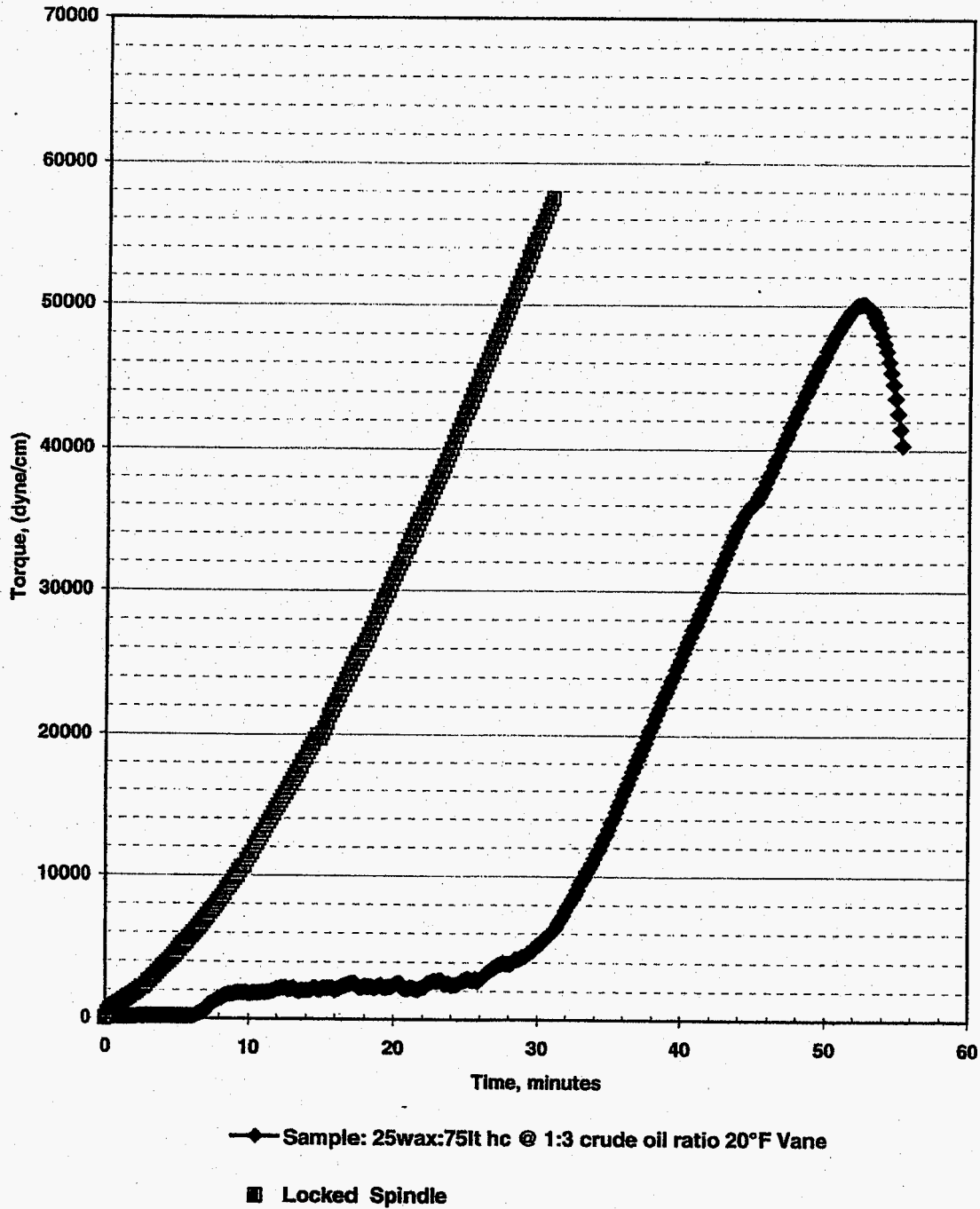
Figure 10: Sample (ii) at 9°F

Yield Point by Vane Viscometry

Figure 11: Sample (iii) at 60°F

Yield Point by Vane Viscometry

Legend:
— ◆ — Sample: 50wax:50lt hc @ 1:4 crude oil ratio 60°F Vane
▣ Locked Spindle

(Y-axis: Torque, (dyne/cm) — ranging 0 to 800; X-axis: Time, minutes — ranging 0 to 60)

Figure 12: Sample (iii) at 40°F

Yield Point by Vane Viscometry

Sample: 50wax:50lt hc @ 1:4 crude oil ratio 40°F Vane

Locked Spindle

Figure 13: Sample (iii) at 40°F

Yield Point by Vane Viscometry

Chart: X-axis "Time, minutes" (0 to 90), Y-axis "Torque, (dyne/cm)" (0 to 70000)

◆ Sample: 50wax:50lt hc @ 1:4 crude oil ratio 40°F Vane

▦ Locked Spindle

Figure 14: Sample (iii) at 9°F

Yield Point by Vane Viscometry

— Sample: 50wax:50lt hc @ 1:4 crude oil ratio 9°F Vane

■ Locked Spindle

Figure 15: Sample (iv) at 60°F

Yield Point by Vane Viscometry

— Sample: 50wax:50lt hc @ 1:3 crude oil ratio 60°F Vane

■ Locked Spindle

Figure 16: Sample (iv) at 40°F

Yield Point by Vane Viscometry

Sample: 50wax:50lt hc @ 1:3 crude oil ratio 40°F Vane

Locked Spindle
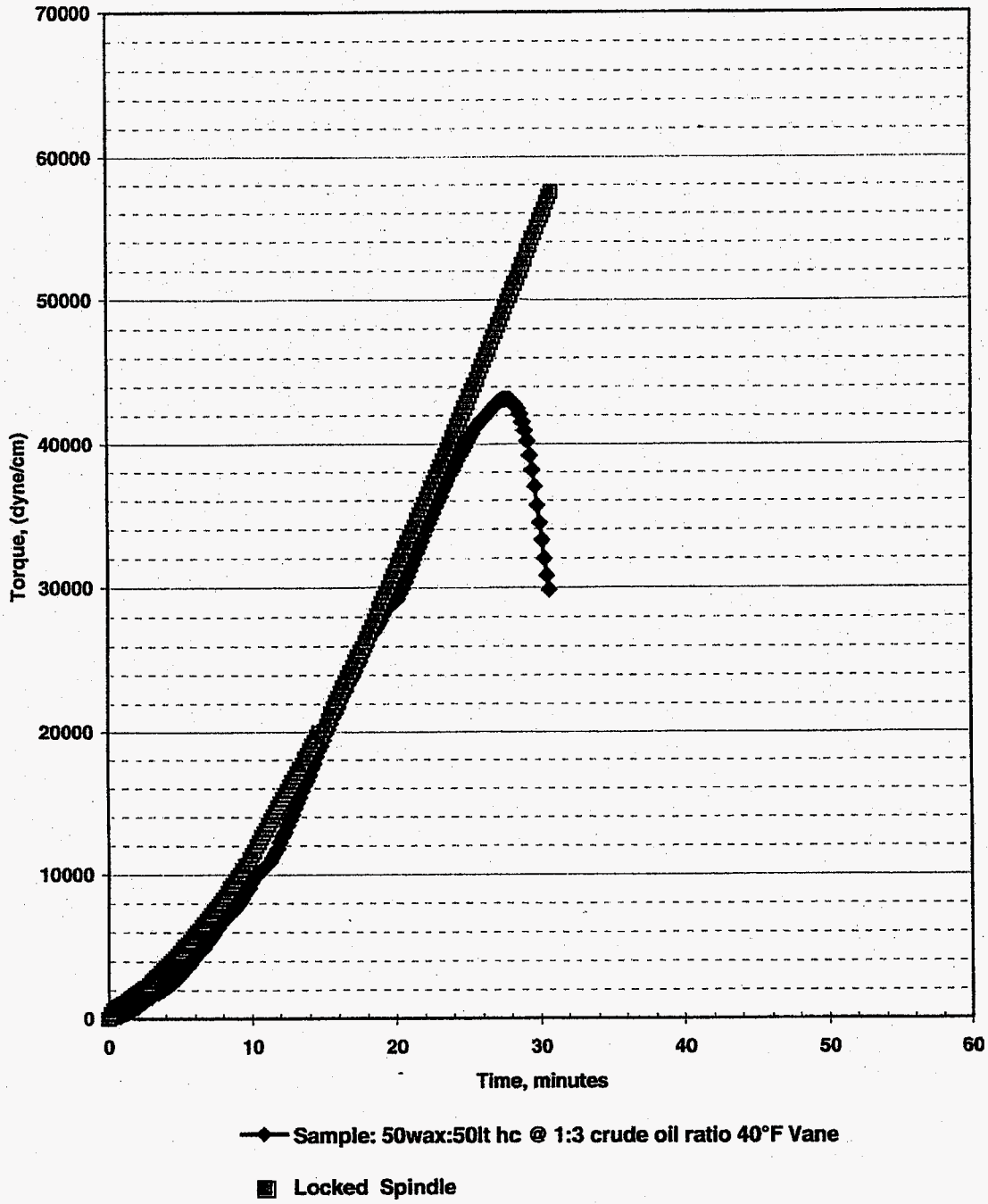
Figure 17: Sample (iv) at 20°F

Yield Point by Vane Viscometry

Sample: 50wax:50lt hc @ 1:3 crude oil ratio 20°F Vane

Locked Spindle

Figure 18: Sample (iv) at 9°F

Yield Point by Vane Viscometry

- Sample: 50wax:50lt hc @ 1:3 crude oil ratio 9°F Vane
- Locked Spindle
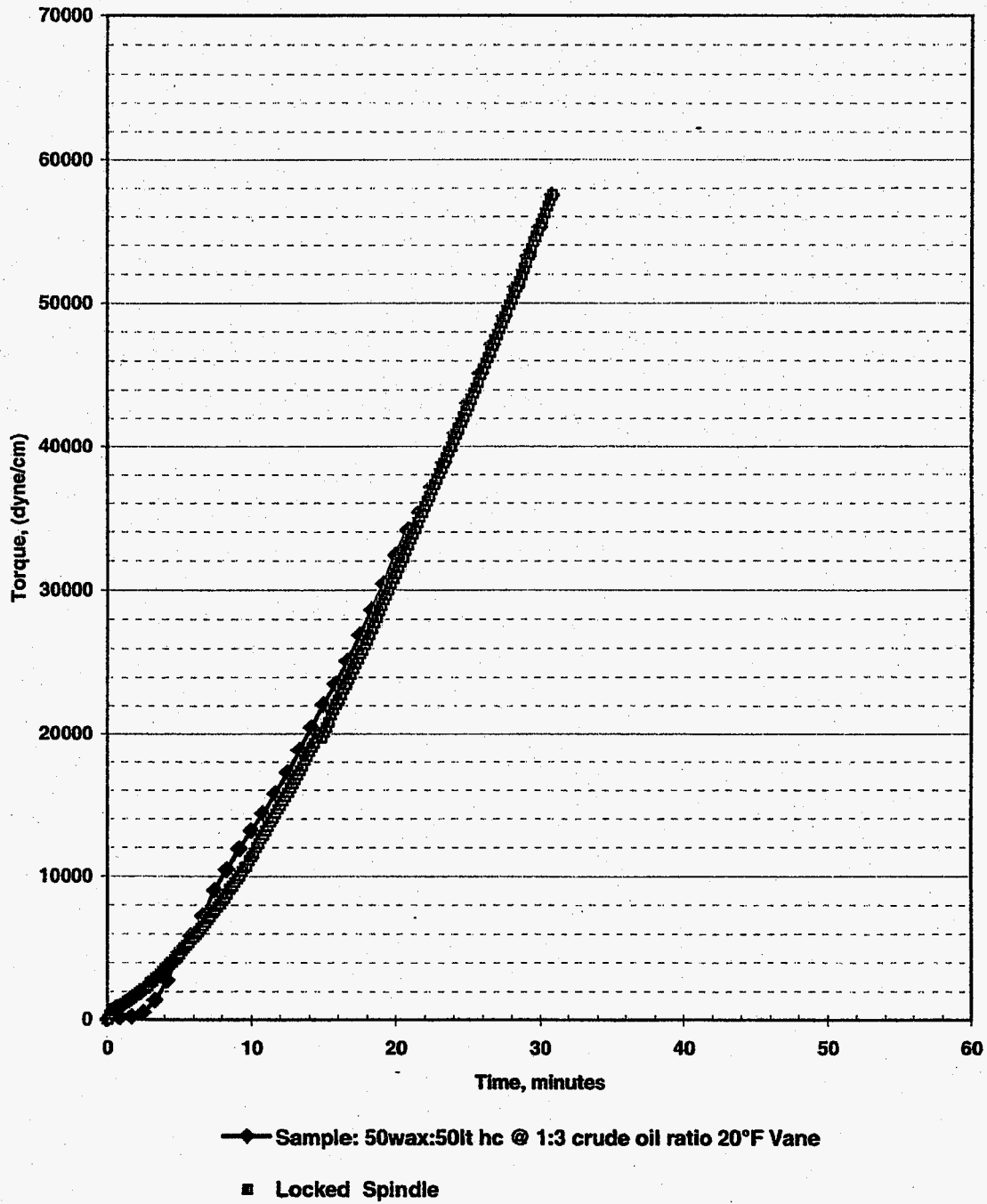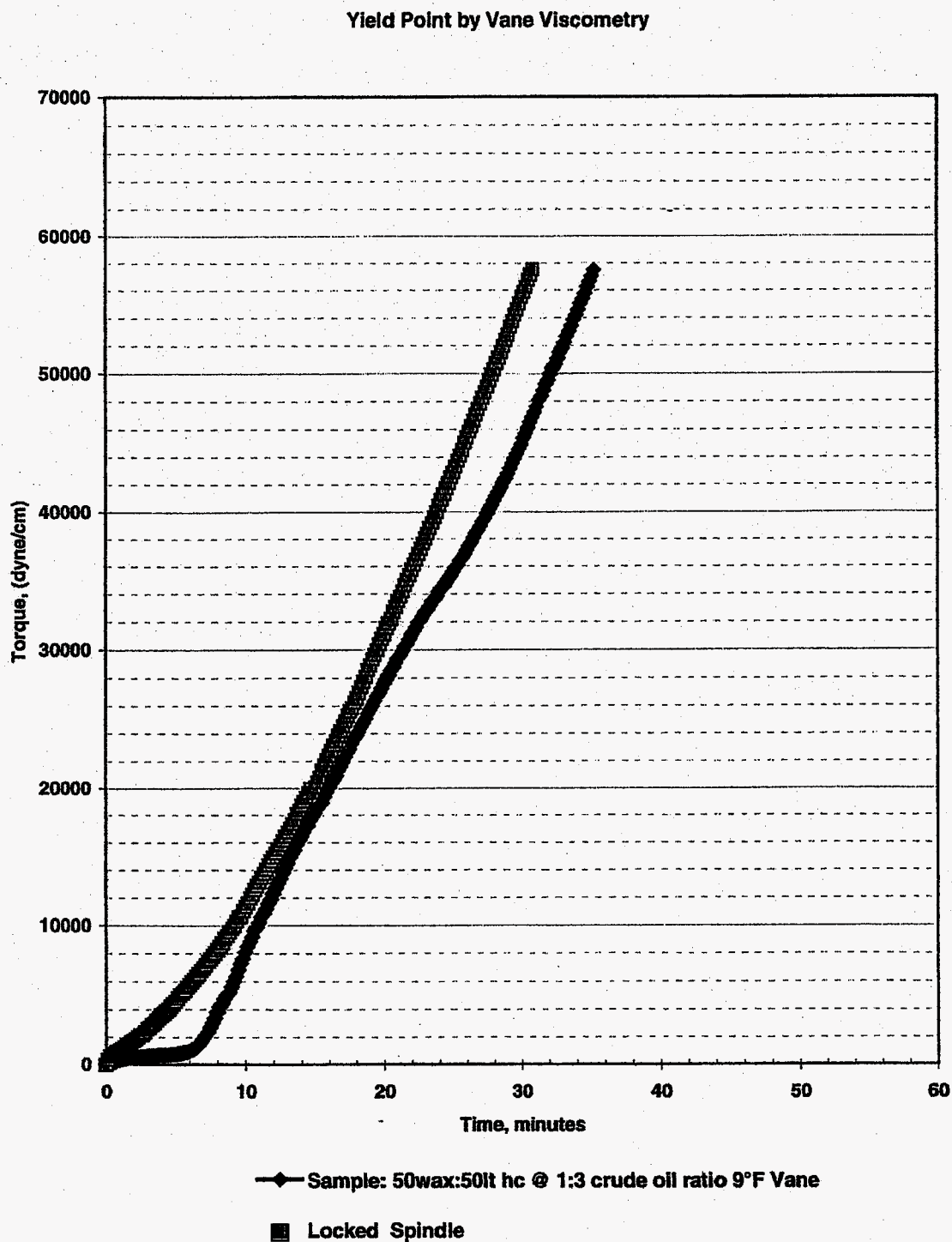
Figure 19: Sample (v) at 20°F

Yield Point by Vane Viscometry

Sample: Lt Hc GTL + Crude Oil 1:4 ratio 20°F Vane

Locked Spindle
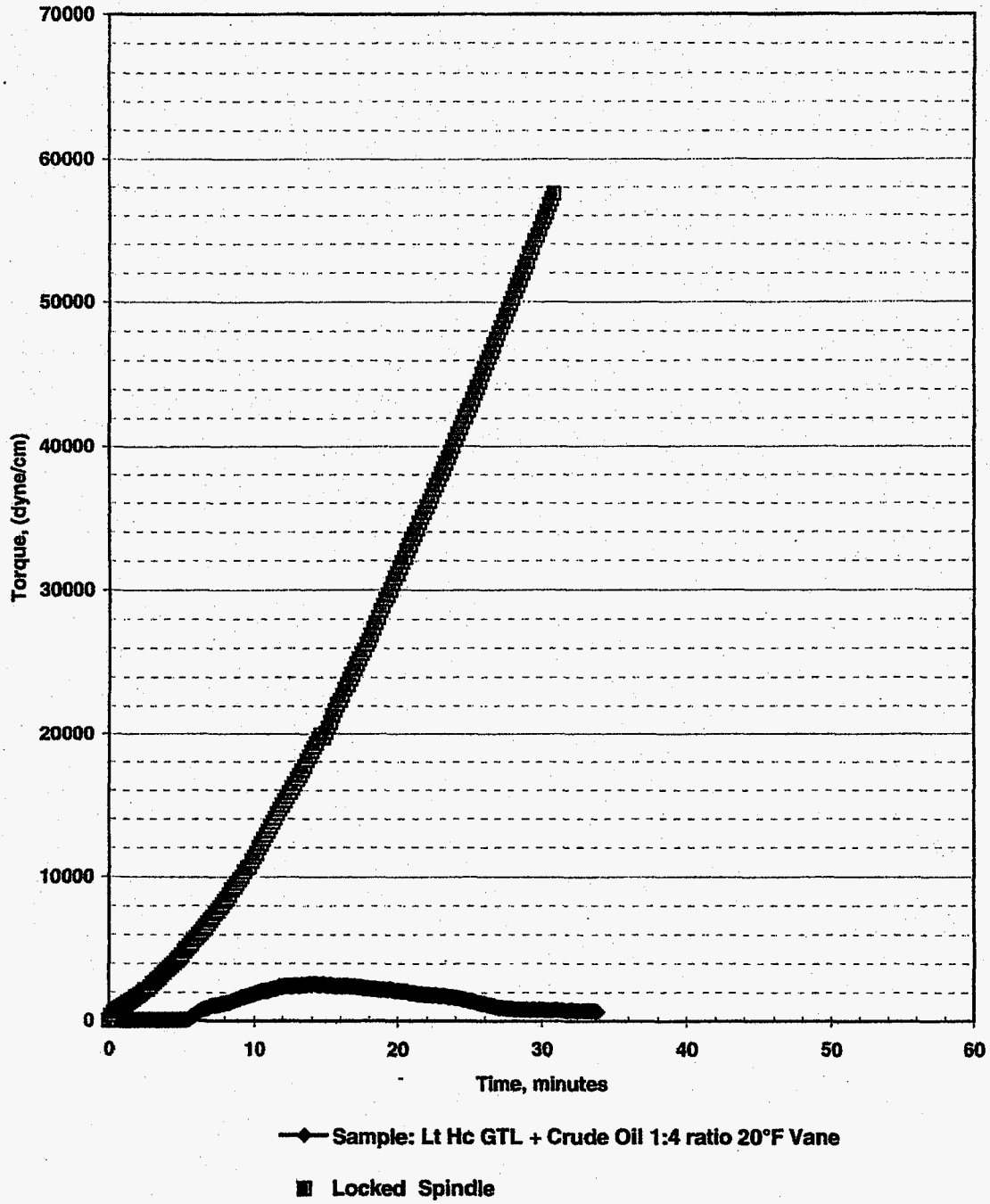
Figure 20: Sample (v) at 9°F

Yield Point by Vane Viscometry

—◆—Sample: Lt Hc GTL + Crude Oil 1:4 ratio 9°F Vane

■ Locked Spindle
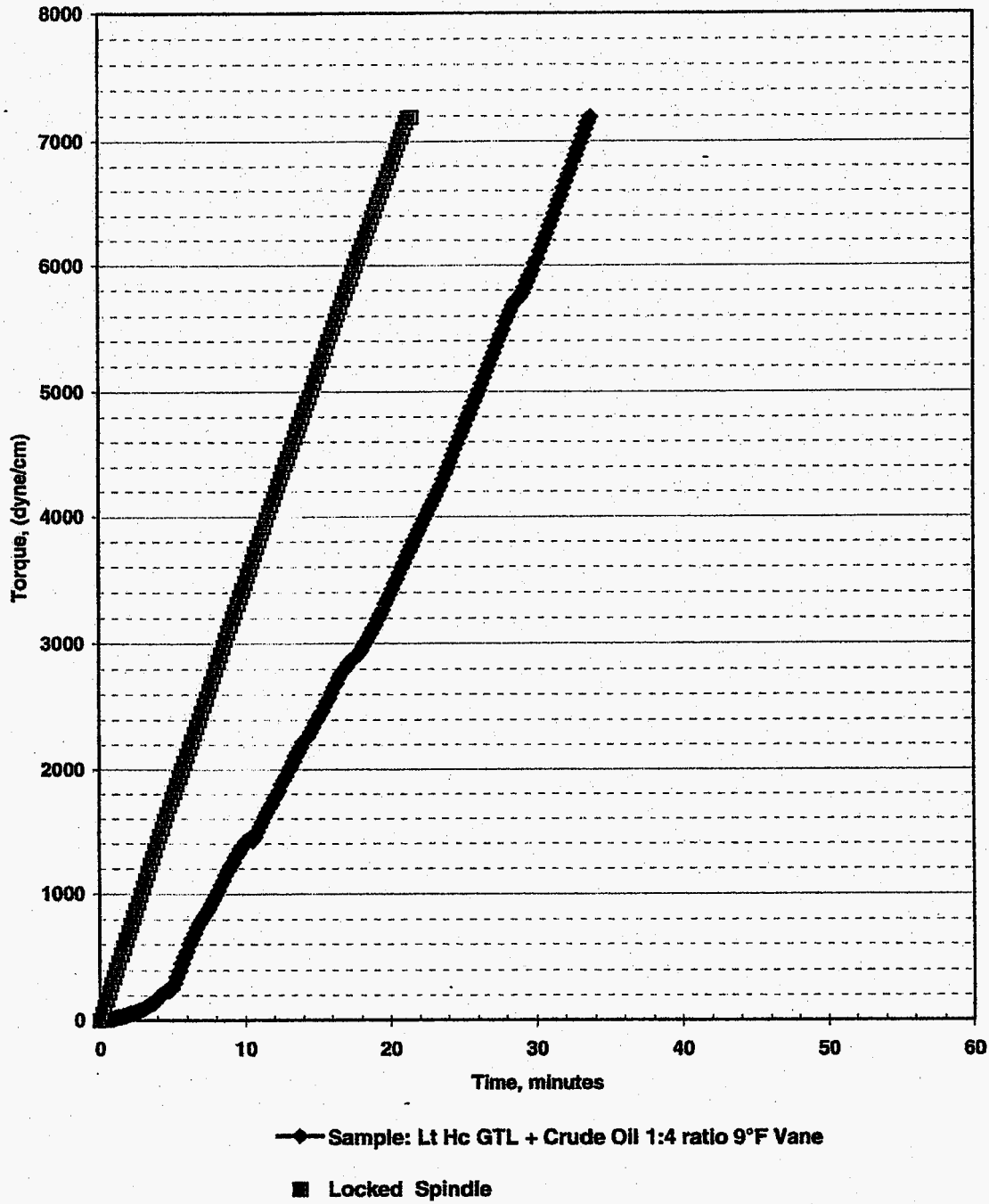
Figure 21: Sample (v) at 9°F

Yield Point by Vane Viscometry

Sample: Lt Hc GTL + Crude Oil 1:4 ratio 9°F Vane

Locked Spindle
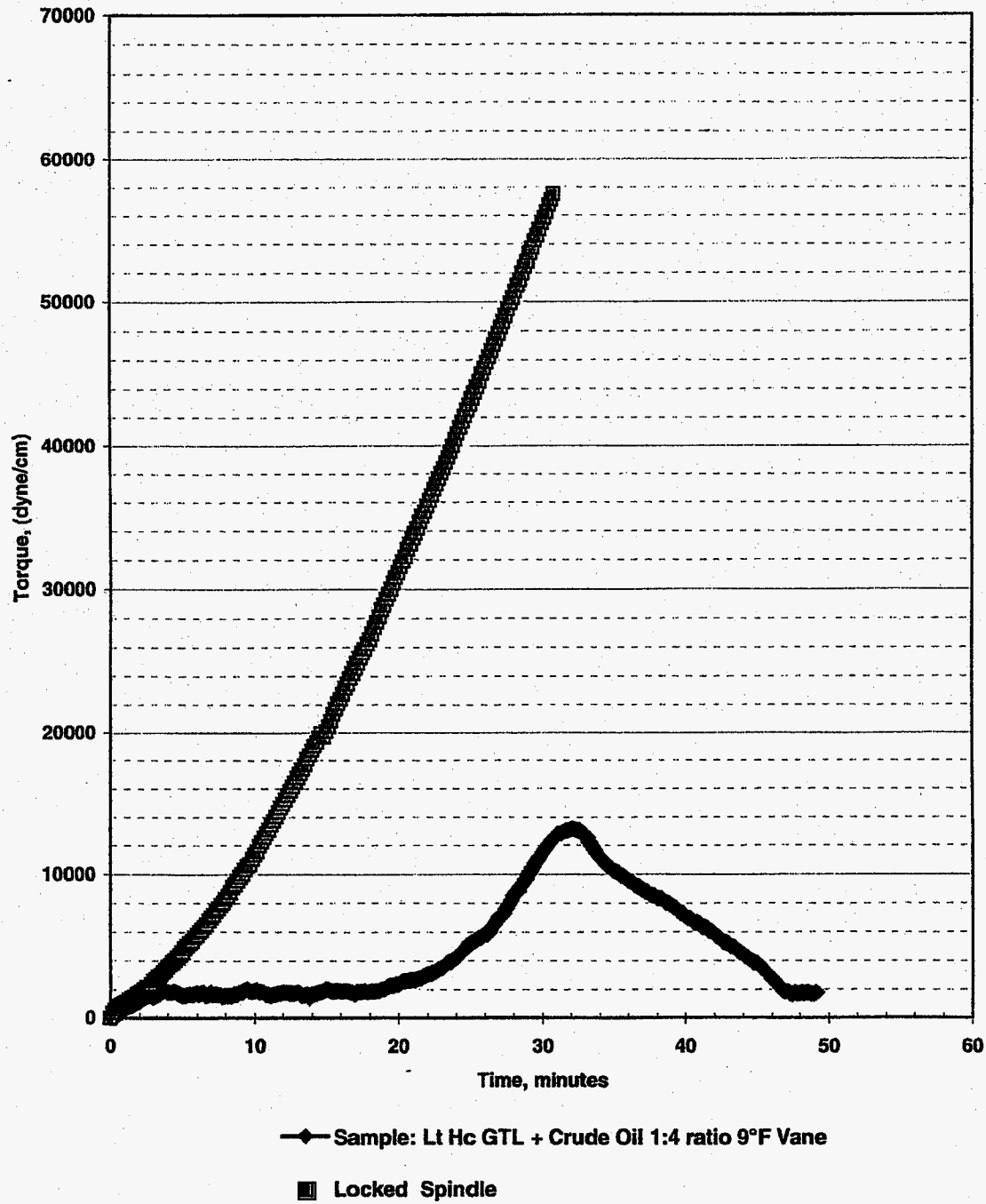
Figure 22: Sample (v) at 0°F

Yield Point by Vane Viscometry

Legend:
- —◆— Sample: Lt Hc GTL + Crude Oil 1:4 ratio 0°F Vane
- ▣ Locked Spindle
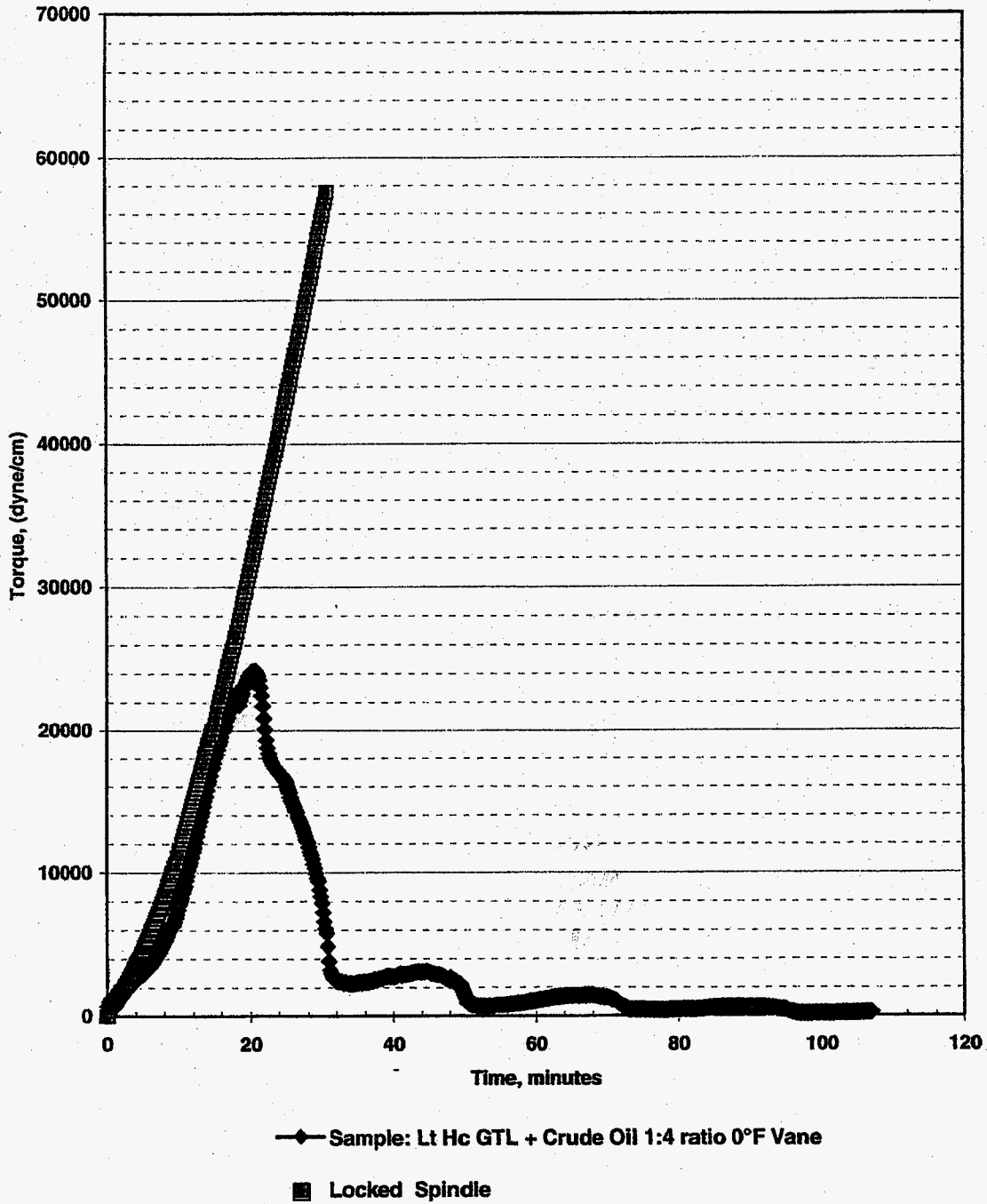
Figure 23: Sample (vi) at 20°F

Yield Point by Vane Viscometry

Sample: Lt Hc GTL + Crude Oil 1:3 ratio 20°F Vane

Locked Spindle

Figure 24: Sample (vi) at 9°F

Yield Point by Vane Viscometry

Figure 25: Sample (vi) at 9°F

Yield Point by Vane Viscometry

Legend:
- Sample: Lt Hc GTL + Crude Oil 1:3 ratio 9°F Vane
- Locked Spindle

Figure 26: Sample (vi) at 0°F

Yield Point by Vane Viscometry

- ◆ Sample: Lt Hc GTL + Crude Oil 1:3 ratio 0°F Vane
- ■ Locked Spindle

SECTION 2

Quantitative Composition of GTL & Crude Oil Blends By Capillary Gas Chromatography

EXTENDED GAS CHROMATOGRAPHY - LIQUID HYDROCARBON ANALYSIS

This liquid hydrocarbon test method was developed to analyze a wide range of liquid samples from light condensate oils through heavy black oils. The GC apparatus has undergone extensive testing and modifications. It has proven to be a reliable means to obtain an accurate composition on most liquid hydrocarbon samples.

Liquid hydrocarbon samples may or may not require an internal standard to be added. All black oils require an internal standard for quantification, but condensates that elute completely do not. If a liquid is questionable, then the internal standard is added. Carbon disulfide is added to viscous samples.

Once the sample has been prepared, it is placed in an auto sampler vial. An auto sampler is used because the analysis time for a liquid is 90 minutes, and the samples are most often analyzed automatically over night. The gas chromatograph is connected to a personal computer for file storage and retrieval. Data can be recalled and re-analyzed using various parameters.

The GC instrument also undergoes a vigorous quality control scheme that takes almost the entire day due the length of the analysis. Quality Control samples are used prior to analyses of samples. They consist of a Discrimination Standard (Normal Alkanes prepared in Undecane) to check the system initially, an Aromatic Standard to calculate response factors for the desired aromatics, D-2887 Reference Gas Oil No.1, and either of two crude oil standards prepared and rigorously analyzed by various methods. Once the instrument meets all of these requirements, test samples are analyzed and the crude oil standard chosen will be analyzed after every fifth sample in the sequence.

For further detail, the standard laboratory procedure can be reviewed in Appendix C.

Compositional analyses are presented on the following pages in both tabular and graphical format. The two distillation wax and light hydrocarbon GTL mixtures at 25%:75% and 50%:50% weight ratios are presented first. Blended samples (i) through (iv) were also analyzed. The raw GTL and North Slope crude oil sample were similarly analyzed. The GTL and crude blends (samples v and vi) were not analyzed.

Quantitative Composition of GTL and Crude Oil Blends

Sample	Gas to liquids product (GTL)	25% Wax / 75% GTL	50% Wax / 50% GTL	TAPS Mix Crude Oil @ PS-1	25:75 Wax/GTL 80g @ 320g oil	25:75 Wax/GTL 100g @ 300g oil	50:50 Wax/GTL 80g @ 320g oil	50:50 Wax/GTL 100g @ 300g oil
ChemStation File	J261N.D	I865N.D	I866N.D	J260REG.D	I861ISO.D	I862ISO.D	I864ISO.D	I863ISO.D
Components	Wt%	Wt%	Wt%	Wt%	Wt%	Wt%	Wt%	Wt%
C2	0.002	0.003	0.003	0.009	0.000	0.001	0.000	0.001
C3	0.145	0.107	0.066	0.167	0.033	0.050	0.055	0.069
IC4	0.003	0.011	0.007	0.259	0.128	0.149	0.000	0.160
NC4	1.215	0.904	0.245	0.954	0.734	0.852	0.951	0.797
IC5	0.146	0.110	0.069	0.630	0.502	0.508	0.555	0.484
NC5	2.972	2.212	1.367	0.967	1.095	1.195	1.174	1.015
C6	7.826	5.800	3.901	1.836	2.758	3.050	2.443	2.463
BENZENE	0.003	0.002	0.001	0.340	0.270	0.255	0.272	0.244
C7	8.889	6.633	4.167	3.358	3.904	4.060	3.509	3.476
TOLUENE	0.029	0.022	0.016	0.792	0.623	0.538	0.611	0.565
C8	8.764	6.570	4.147	4.078	4.572	4.662	4.111	4.092
C9	8.539	6.384	4.071	4.115	4.506	4.579	4.142	4.048
C10	7.940	6.230	3.972	3.715	4.121	4.202	3.696	3.737
C11	7.290	5.607	3.989	3.174	3.663	3.760	3.292	3.367
C12	6.446	5.241	3.627	2.939	3.403	3.490	3.222	3.206
C13	5.748	4.908	3.554	3.308	3.584	3.640	3.350	3.413
C14	5.065	4.610	3.942	3.160	3.421	3.490	3.393	3.367
C15	4.414	4.367	4.101	3.108	3.312	3.359	3.266	3.342
C16	3.811	4.189	4.348	2.757	3.042	3.105	3.133	3.194
C17	3.273	4.096	4.699	2.636	2.914	2.985	3.147	3.184
C18	2.799	4.071	5.119	2.590	2.919	2.991	3.145	3.292
C19	2.389	4.070	5.522	2.543	2.847	2.923	3.154	3.329
C20	2.026	4.001	5.753	2.296	2.653	2.747	3.150	3.230
C21	1.705	3.795	5.674	2.134	2.493	2.571	2.876	3.079
C22	1.421	3.422	5.236	2.037	2.324	2.409	2.711	2.894
C23	1.165	2.914	4.511	1.898	2.122	2.163	2.456	2.600
C24	0.939	2.353	3.650	1.781	1.909	1.948	2.130	2.286
C25	0.747	1.816	2.804	1.669	1.723	1.744	1.929	1.984
C26	0.607	1.369	2.086	1.624	1.572	1.592	1.715	1.757
C27	0.583	1.094	1.584	1.557	1.470	1.478	1.553	1.570
C28	0.433	0.764	1.104	1.532	1.375	1.378	1.437	1.422
C29	0.247	0.458	0.710	1.482	1.249	1.274	1.311	1.270
C30+	2.418	1.866	5.457	34.506	28.757	26.795	25.444	27.061
Total	100.000	100.000	100.000	100.000	100.000	100.000	100.000	100.000

Wt% Wax	0.00	50.00	25.00	0.00	5.00	6.25	10.00	12.50
Wt% GTL	100.00	50.00	75.00	0.00	15.00	18.75	10.00	12.50
Wt% Crude	0.00	0.00	0.00	100.00	80.00	75.00	80.00	75.00

Figure 27

Blend Composition

Legend:
- 50:50 Wax/GTL 80g @ 320g oil
- 50:50 Wax/GTL 100g @ 300g oil
- 25:75 Wax/GTL 80g @ 320g oil
- 25:75 Wax/GTL 100g @ 300g oil
- TAPS Mix Crude Oil @ PS-1

Figure 28

Crude Oil, GTL and GTL-Wax Blend Composition

Legend:
- Gas to liquids product (GTL)
- 25% Wax / 75% GTL
- 50% Wax / 50% GTL

Figure 29

50% Wax / 50% GTL

Components

Figure 30

25% Wax / 75% GTL

Components

Figure 31

25:75 Wax/GTL 80g @ 320g oil

Components

Figure 32

25:75 Wax/GTL 100g @ 300g oil

Components

Figure 33

50:50 Wax/GTL 80g @ 320g oil

Figure 34

50:50 Wax/GTL 100g @ 300g oil
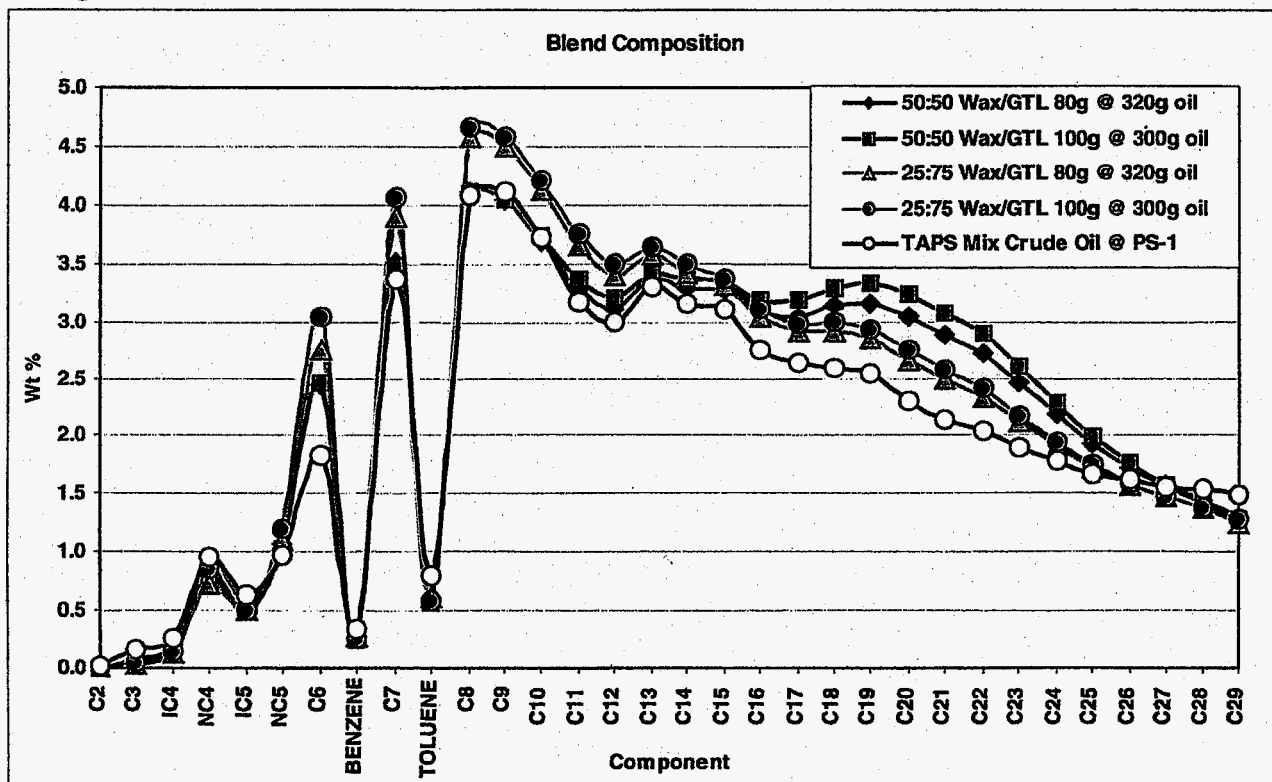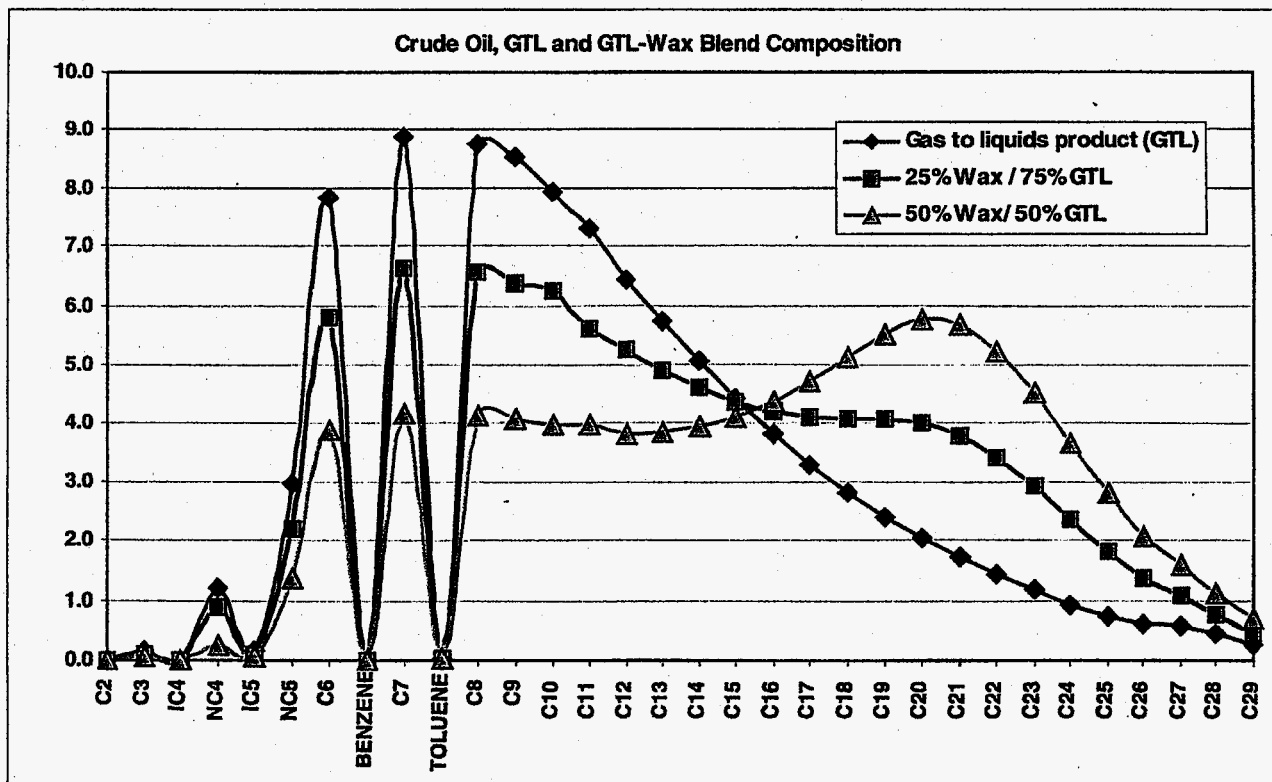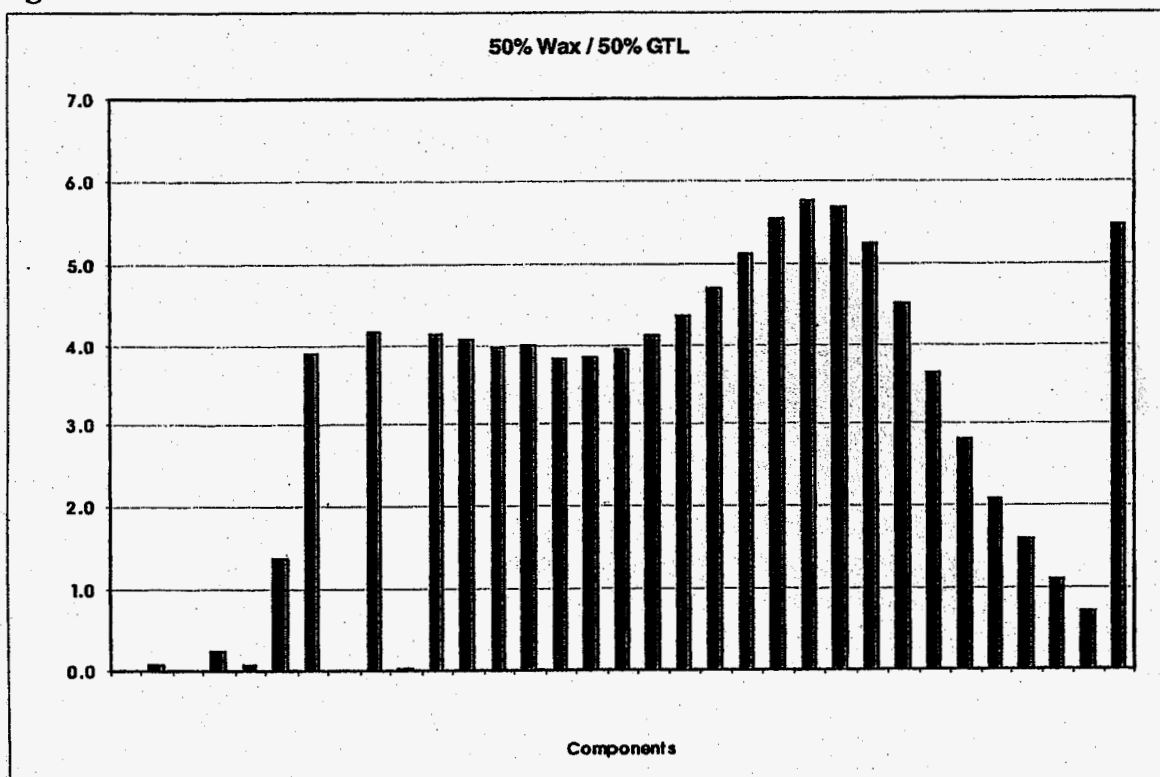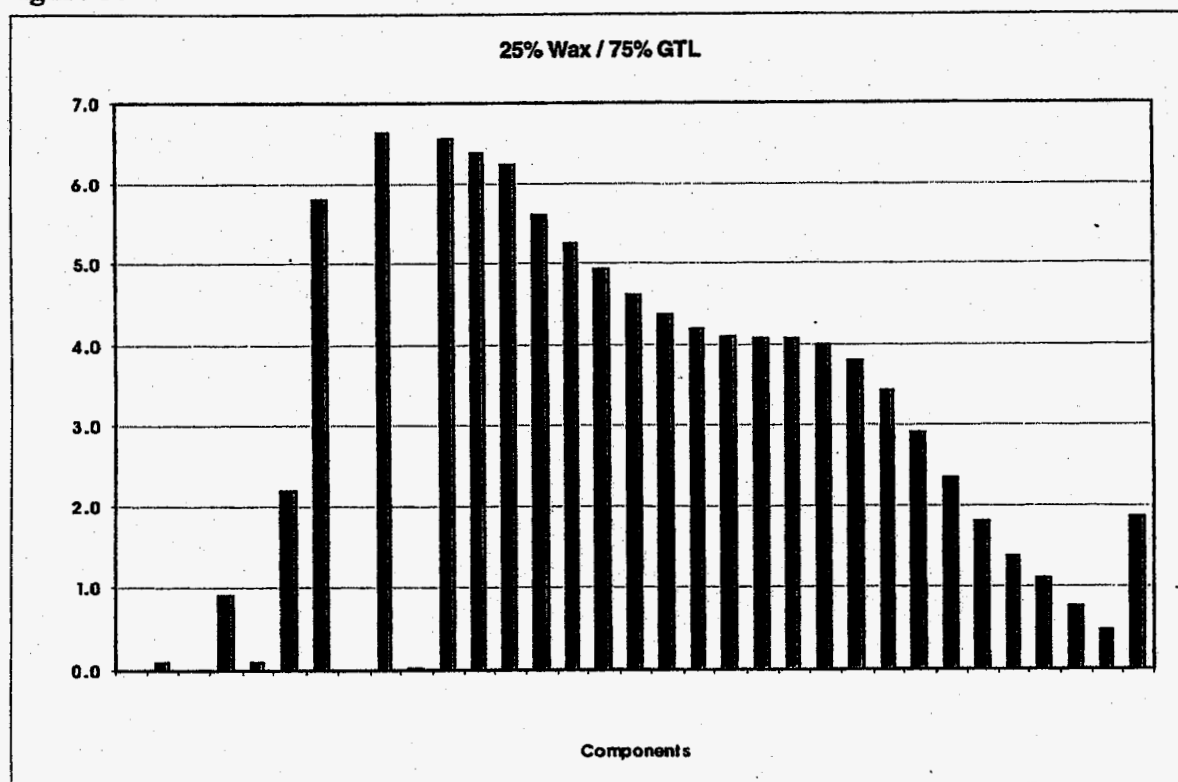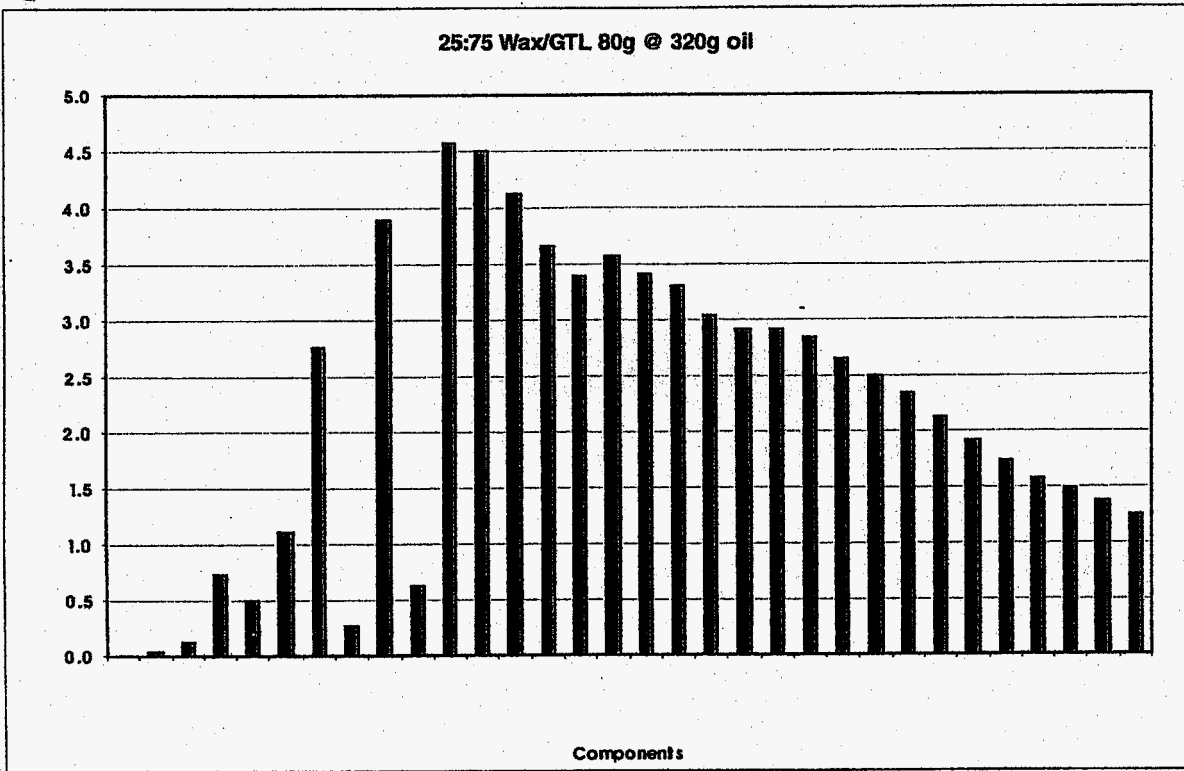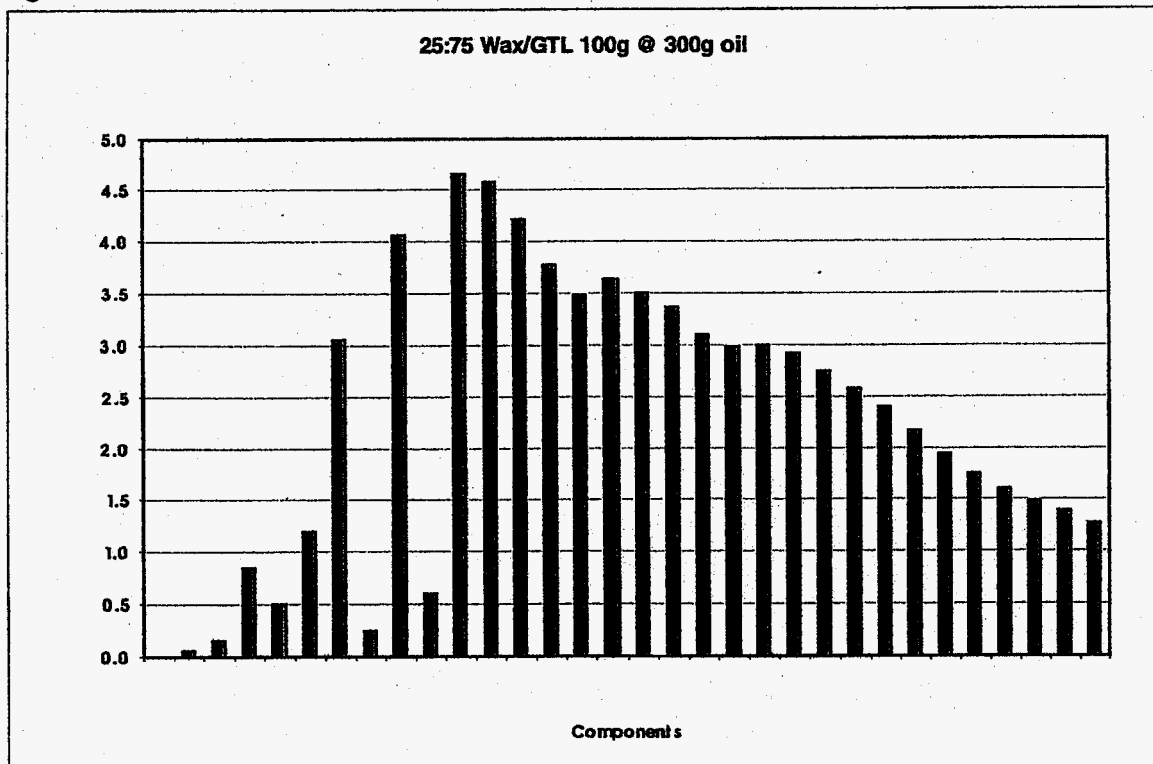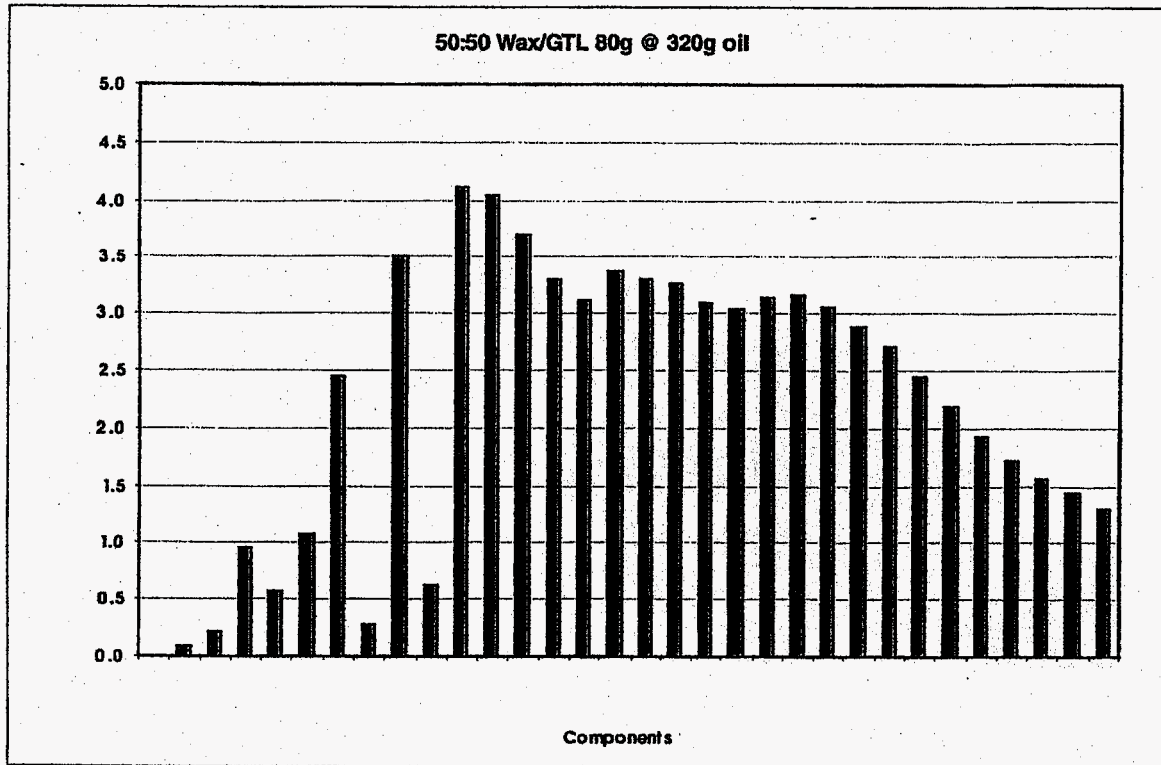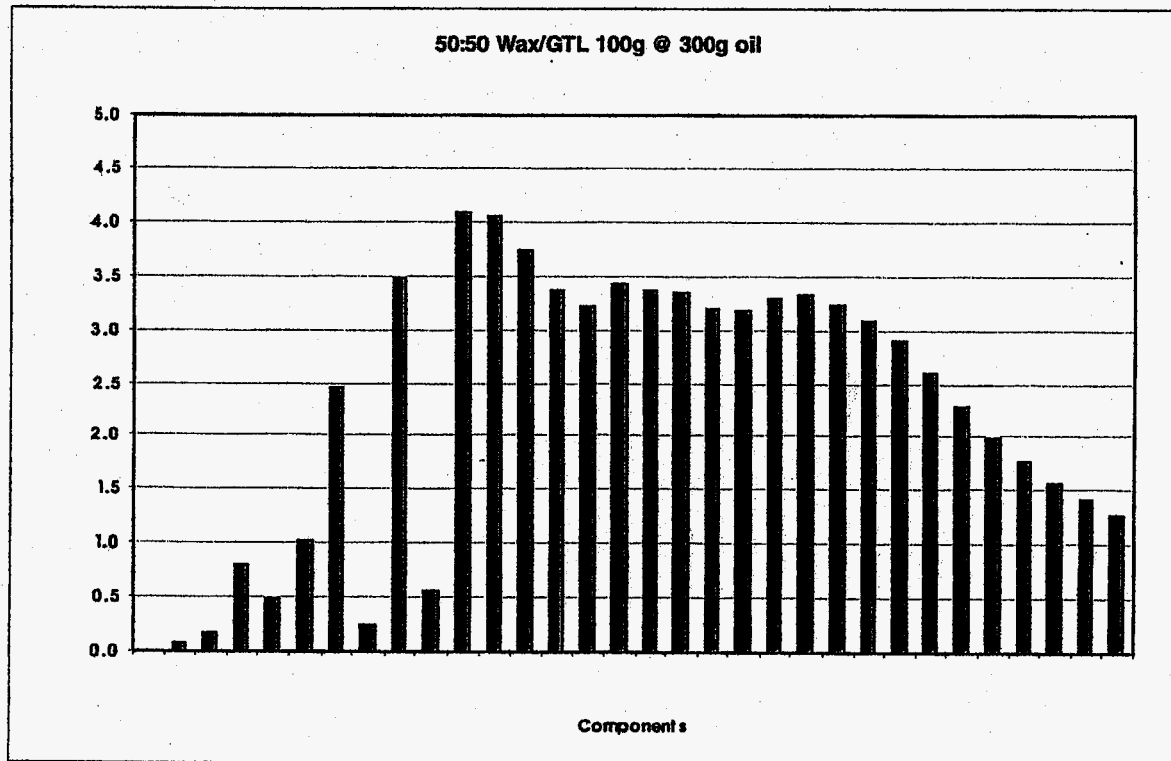
Sample (i)

25% Wax : 75% GTL + Crude Oil in the Ratio of 1:4

Sample: 25wax:75lt hc @ 1:4 crude oil ratio			Sample: 25wax:75lt hc @ 1:4 crude oil ratio			Sample: 25wax:75lt hc @ 1:4 crude oil ratio		
Temperature :	40°F		Temperature :	20°F		Temperature :	9°F	
Spindle:	Vane		Spindle:	Vane		Spindle:	Vane	
Model:	RV		Model:	HB		Model:	HB	
Cum. Time minutes	Torque %	Stress (dyne/cm)	Cum. Time minutes	Torque %	Stress (dyne/cm)	Cum. Time minutes	Torque %	Stress (dyne/cm)
0.00	0.0	0.0	0.00	0.1	57	0.00	0.0	0
0.17	0.1	7.2	0.17	0.1	57	0.17	0.3	172
0.33	0.1	7.2	0.33	0.1	57	0.33	0.3	172
0.50	0.1	7.2	0.50	0.1	57	0.50	0.3	172
0.67	0.1	7.2	0.67	0.1	57	0.67	0.4	230
0.83	0.2	14.4	0.83	0.1	57	0.83	0.4	230
1.00	0.2	14.4	1.00	0.1	57	1.00	0.3	172
1.17	0.3	21.6	1.17	0.1	57	1.17	0.4	230
1.33	0.4	28.7	1.33	0.2	115	1.33	0.4	230
1.50	0.5	35.9	1.50	0.1	57	1.50	0.3	172
1.67	0.5	35.9	1.67	0.1	57	1.67	0.3	172
1.83	0.5	35.9	1.83	0.1	57	1.83	0.4	230
2.00	0.6	43.1	2.00	0.1	57	2.00	0.4	230
2.17	0.8	57.5	2.17	0.1	57	2.17	0.4	230
2.33	0.8	57.5	2.33	0.1	57	2.33	0.4	230
2.50	0.9	64.7	2.50	0.1	57	2.50	0.4	230
2.67	1.0	71.9	2.67	0.2	115	2.67	0.4	230
2.83	0.9	64.7	2.83	0.1	57	2.83	0.4	230
3.00	1.0	71.9	3.00	0.1	57	3.00	0.4	230
3.17	1.1	79.1	3.17	0.1	57	3.17	0.4	230
3.33	1.4	100.6	3.33	0.1	57	3.33	0.4	230
3.50	1.6	115.0	3.50	0.1	57	3.50	0.4	230
3.67	1.9	136.6	3.67	0.2	115	3.67	0.4	230
3.83	2.0	143.7	3.83	0.1	57	3.83	0.4	230
4.00	2.2	158.1	4.00	0.1	57	4.00	0.4	230
4.17	2.5	179.7	4.17	0.2	115	4.17	0.4	230
4.33	2.6	186.9	4.33	0.1	57	4.33	0.4	230
4.50	2.8	201.2	4.50	0.1	57	4.50	0.4	230
4.67	3.2	230.0	4.67	0.1	57	4.67	0.4	230
4.83	3.5	251.5	4.83	0.1	57	4.83	0.4	230
5.00	3.9	280.3	5.00	0.1	57	5.00	0.4	230
5.17	4.3	309.0	5.17	0.1	57	5.17	0.4	230
5.33	4.6	330.6	5.33	0.1	57	5.33	0.4	230
5.50	5.0	359.4	5.50	0.1	57	5.50	0.4	230
5.67	5.3	380.9	5.67	0.2	115	5.67	0.4	230
5.83	5.6	402.5	5.83	0.2	115	5.83	0.4	230
6.00	5.9	424.0	6.00	0.2	115	6.00	0.4	230
6.17	6.3	452.8	6.17	0.2	115	6.17	0.5	287
6.33	6.5	467.2	6.33	0.2	115	6.33	0.5	287
6.50	6.8	488.7	6.50	0.2	115	6.50	0.6	345
6.67	7.1	510.3	6.67	0.2	115	6.67	1.0	575
6.83	7.4	531.8	6.83	0.2	115	6.83	1.5	862
7.00	7.6	546.2	7.00	0.3	172	7.00	1.7	977
7.17	7.9	567.8	7.17	0.3	172	7.17	1.9	1092
7.33	8.2	589.3	7.33	0.3	172	7.33	2.0	1150
7.50	8.4	603.7	7.50	0.7	402	7.50	2.2	1265
7.67	8.7	625.3	7.67	1.1	632	7.67	2.4	1380
7.83	8.9	639.6	7.83	1.4	805	7.83	2.6	1495
8.00	9.1	654.0	8.00	1.5	862	8.00	2.7	1552
8.17	9.3	668.4	8.17	1.7	977	8.17	2.8	1610
8.33	9.5	682.8	8.33	1.9	1092	8.33	2.9	1667
8.50	9.7	697.1	8.50	2.1	1207	8.50	3.1	1782
8.67	9.8	704.3	8.67	2.2	1265	8.67	3.3	1897
8.83	10.0	718.7	8.83	2.3	1322	8.83	3.4	1955
9.00	10.0	718.7	9.00	2.3	1322	9.00	3.5	2012
9.17	10.2	733.1	9.17	2.5	1437	9.17	3.9	2242
9.33	10.3	740.3	9.33	2.5	1437	9.33	4.1	2357
9.50	10.3	740.3	9.50	2.7	1552	9.50	4.2	2415
9.67	10.3	740.3	9.67	2.8	1610	9.67	4.5	2587
9.83	10.4	747.4	9.83	2.9	1667	9.83	4.7	2702
10.00	10.4	747.4	10.00	3.1	1782	10.00	4.9	2817
10.17	10.4	747.4	10.17	3.5	2012	10.17	5.1	2932
10.33	10.4	747.4	10.33	3.5	2012	10.33	5.1	2932
10.50	10.4	747.4	10.50	3.8	2185	10.50	5.3	3047

Sample: 25wax:75lt hc @ 1:4 crude oil ratio			Sample: 25wax:75lt hc @ 1:4 crude oil ratio			Sample: 25wax:75lt hc @ 1:4 crude oil ratio		
Temperature : 40°F			Temperature : 20°F			Temperature : 9°F		
Spindle: Vane			Spindle: Vane			Spindle: Vane		
Model: RV			Model: HB			Model: HB		
Cum. Time minutes	Torque %	Stress (dyne/cm)	Cum. Time minutes	Torque %	Stress (dyne/cm)	Cum. Time minutes	Torque %	Stress (dyne/cm)
10.67	10.3	740.3	10.67	4.0	2300	10.67	5.7	3277
10.83	10.3	740.3	10.83	4.1	2357	10.83	5.9	3392
11.00	10.3	740.3	11.00	4.3	2472	11.00	6.0	3450
11.17	10.2	733.1	11.17	4.5	2587	11.17	6.4	3680
11.33	10.2	733.1	11.33	4.7	2702	11.33	6.6	3795
11.50	10.1	725.9	11.50	4.8	2760	11.50	6.7	3852
11.67	10.0	718.7	11.67	5.1	2932	11.67	7.0	4025
11.83	9.9	711.5	11.83	5.1	2932	11.83	7.1	4082
12.00	9.8	704.3	12.00	5.2	2990	12.00	7.4	4255
12.17	9.7	697.1	12.17	5.4	3105	12.17	7.7	4427
12.33	9.6	690.0	12.33	5.5	3162	12.33	7.7	4427
12.50	9.6	690.0	12.50	5.6	3220	12.50	8.0	4600
12.67	9.4	675.6	12.67	5.8	3335	12.67	8.2	4715
12.83	9.3	668.4	12.83	5.9	3392	12.83	8.4	4830
13.00	9.2	661.2	13.00	6.1	3507	13.00	8.6	4945
13.17	9.1	654.0	13.17	6.3	3622	13.17	8.8	5060
13.33	9.0	646.8	13.33	6.4	3680	13.33	9.0	5175
13.50	9.0	646.8	13.50	6.7	3852	13.50	9.2	5290
13.67	8.8	632.5	13.67	6.8	3910	13.67	9.4	5405
13.83	8.7	625.3	13.83	6.9	3967	13.83	9.6	5520
14.00	8.7	625.3	14.00	6.9	3967	14.00	10.0	5750
14.17	8.5	610.9	14.17	7.2	4140	14.17	10.3	5922
14.33	8.5	610.9	14.33	7.3	4197	14.33	10.5	6037
14.50	8.4	603.7	14.50	7.5	4312	14.50	10.8	6210
14.67	8.3	596.5	14.67	7.7	4427	14.67	11.3	6497
14.83	8.2	589.3	14.83	8.1	4657	14.83	11.8	6785
15.00	8.1	582.1	15.00	8.5	4887	15.00	12.1	6957
15.17	8.0	575.0	15.17	8.9	5117	15.17	12.0	6900
15.33	7.9	567.8	15.33	9.1	5232	15.33	12.2	7015
15.50	7.9	567.8	15.50	9.5	5462	15.50	12.7	7302
15.67	7.8	560.6	15.67	10.1	5807	15.67	13.2	7589
15.83	7.7	553.4	15.83	10.5	6037	15.83	13.7	7877
16.00	7.7	553.4	16.00	11.0	6325	16.00	13.7	7877
16.17	7.6	546.2	16.17	11.5	6612	16.17	13.6	7819
16.33	7.5	539.0	16.33	12.0	6900	16.33	13.8	7934
16.50	7.5	539.0	16.50	12.1	6957	16.50	14.2	8164
16.67	7.4	531.8	16.67	12.3	7072	16.67	14.6	8394
16.83	7.3	524.7	16.83	12.7	7302	16.83	15.1	8682
17.00	7.2	517.5	17.00	13.1	7532	17.00	15.7	9027
17.17	7.2	517.5	17.17	13.7	7877	17.17	16.4	9429
17.33	7.0	503.1	17.33	14.3	8222	17.33	17.1	9832
17.50	6.9	495.9	17.50	14.6	8394	17.50	17.7	10177
17.67	6.9	495.9	17.67	15.1	8682	17.67	18.4	10579
17.83	6.7	481.5	17.83	15.4	8854	17.83	19.1	10982
18.00	6.7	481.5	18.00	15.3	8797	18.00	19.7	11327
18.17	6.6	474.3	18.17	15.4	8854	18.17	20.2	11614
18.33	6.5	467.2	18.33	16.0	9199	18.33	20.9	12017
18.50	6.4	460.0	18.50	16.4	9429	18.50	21.5	12362
18.67	6.3	452.8	18.67	17.0	9774	18.67	22.2	12764
18.83	6.2	445.6	18.83	17.7	10177	18.83	22.9	13167
19.00	6.2	445.6	19.00	18.2	10464	19.00	23.5	13512
19.17	6.1	438.4	19.17	18.9	10867	19.17	24.1	13857
19.33	5.9	424.0	19.33	19.6	11269	19.33	25.0	14374
19.50	5.8	416.8	19.50	20.3	11672	19.50	25.6	14719
19.67	5.8	416.8	19.67	21.1	12132	19.67	26.2	15064
19.83	5.7	409.7	19.83	21.9	12592	19.83	27.0	15524
20.00	5.6	402.5	20.00	22.6	12994	20.00	27.7	15926
20.17	5.5	395.3	20.17	23.3	13397	20.17	28.3	16271
20.33	5.4	388.1	20.33	24.1	13857	20.33	28.9	16616
20.50	5.3	380.9	20.50	24.7	14202	20.50	29.6	17019
20.67	5.3	380.9	20.67	25.3	14546	20.67	30.0	17249
20.83	5.2	373.7	20.83	26.1	15006	20.83	30.6	17594
21.00	5.1	366.5	21.00	26.7	15351	21.00	31.3	17996
21.17	5.1	366.5	21.17	27.3	15696	21.17	31.8	18284

Sample: 25wax:75lt hc @ 1:4 crude oil ratio			Sample: 25wax:75lt hc @ 1:4 crude oil ratio			Sample: 25wax:75lt hc @ 1:4 crude oil ratio		
Temperature :	40°F		Temperature :	20°F		Temperature :	9°F	
Spindle:	Vane		Spindle:	Vane		Spindle:	Vane	
Model:	RV		Model:	HB		Model:	HB	
Cum. Time	Torque	Stress	Cum. Time	Torque	Stress	Cum. Time	Torque	Stress
minutes	%	(dyne/cm)	minutes	%	(dyne/cm)	minutes	%	(dyne/cm)
21.33	5.0	359.4	21.33	28.1	16156	21.33	32.5	18686
21.50	4.9	352.2	21.50	28.8	16559	21.50	33.3	19146
21.67	4.8	345.0	21.67	29.3	16846	21.67	33.8	19434
21.83	4.7	337.8	21.83	30.1	17306	21.83	34.4	19779
22.00	4.6	330.6	22.00	30.8	17709	22.00	35.2	20239
22.17	4.6	330.6	22.17	31.4	18054	22.17	35.7	20526
22.33	4.6	330.6	22.33	32.2	18514	22.33	36.5	20986
22.50	4.4	316.2	22.50	32.9	18916	22.50	37.3	21446
22.67	4.4	316.2	22.67	33.4	19204	22.67	37.8	21733
22.83	4.3	309.0	22.83	34.1	19606	22.83	38.4	22078
23.00	4.2	301.9	23.00	34.8	20009	23.00	39.2	22538
23.17	4.2	301.9	23.17	35.3	20296	23.17	39.7	22826
23.33	4.1	294.7	23.33	36.0	20699	23.33	40.4	23228
23.50	4.1	294.7	23.50	36.7	21101	23.50	41.1	23631
23.67	4.0	287.5	23.67	37.1	21331	23.67	41.6	23918
23.83	4.0	287.5	23.83	37.8	21733	23.83	42.1	24206
24.00	3.9	280.3	24.00	38.4	22078	24.00	42.8	24608
24.17	3.8	273.1	24.17	38.8	22308	24.17	43.5	25011
24.33	3.7	265.9	24.33	39.4	22653	24.33	44.1	25356
24.50	3.7	265.9	24.50	40.0	22998	24.50	44.9	25816
24.67	3.7	265.9	24.67	40.4	23228	24.67	45.6	26218
24.83	3.6	258.7	24.83	40.9	23516	24.83	46.3	26621
25.00	3.6	258.7	25.00	41.5	23861	25.00	47.1	27081
25.17	3.5	251.5	25.17	41.9	24091	25.17	47.8	27483
25.33	3.6	258.7	25.33	42.4	24378	25.33	48.4	27828
25.50	3.5	251.5	25.50	43.0	24723	25.50	49.3	28346
25.67	3.5	251.5	25.67	43.4	24953	25.67	50.0	28748
25.83	3.5	251.5	25.83	43.9	25241	25.83	50.6	29093
26.00	3.4	244.4	26.00	44.5	25586	26.00	51.4	29553
26.17	3.3	237.2	26.17	44.9	25816	26.17	52.2	30013
26.33	3.4	244.4	26.33	45.3	26046	26.33	52.7	30300
26.50	3.3	237.2	26.50	45.7	26276	26.50	53.5	30760
26.67	3.3	237.2	26.67	46.1	26506	26.67	54.1	31105
26.83	3.3	237.2	26.83	46.4	26678	26.83	54.4	31278
27.00	3.2	230.0	27.00	46.9	26966	27.00	54.7	31450
27.17	3.2	230.0	27.17	47.1	27081	27.17	54.9	31565
27.33	3.2	230.0	27.33	47.3	27196	27.33	55.3	31795
27.50	3.2	230.0	27.50	47.7	27426	27.50	56.1	32255
27.67	3.2	230.0	27.67	47.9	27541	27.67	56.9	32715
27.83	3.1	222.8	27.83	47.9	27541	27.83	57.5	33060
28.00	3.2	230.0	28.00	47.9	27541	28.00	58.3	33520
28.17	3.1	222.8	28.17	47.2	27138	28.17	59.1	33980
28.33	3.1	222.8	28.33	46.1	26506	28.33	59.8	34383
28.50	3.1	222.8	28.50	46.3	26621	28.50	60.6	34843
28.67	3.1	222.8	28.67	46.5	26736	28.67	61.4	35303
28.83	3.1	222.8	28.83	46.5	26736	28.83	62.1	35705
29.00	3.1	222.8	29.00	46.5	26736	29.00	62.8	36107
29.17	3.1	222.8	29.17	46.4	26678	29.17	63.6	36567
29.33	3.1	222.8	29.33	46.0	26448	29.33	64.3	36970
29.50	3.0	215.6	29.50	45.5	26161	29.50	65.0	37372
29.67	3.0	215.6	29.67	45.0	25873	29.67	65.8	37832
29.83	3.0	215.6	29.83	44.1	25356	29.83	66.5	38235
30.00	3.0	215.6	30.00	43.3	24896	30.00	67.2	38637
30.17	2.9	208.4	30.17	42.4	24378	30.17	68.0	39097
30.33	3.0	215.6	30.33	41.5	23861	30.33	68.7	39500
30.50	3.0	215.6	30.50	40.6	23343	30.50	69.4	39902
30.67	3.0	215.6	30.67	39.9	22941	30.67	70.2	40362
30.83	3.0	215.6	30.83	39.2	22538	30.83	71.0	40822
31.00	3.0	215.6				31.00	71.6	41167
31.17	2.9	208.4				31.17	72.4	41627
31.33	2.9	208.4				31.33	73.2	42087
31.50	3.0	215.6				31.50	73.8	42432
31.67	3.0	215.6				31.67	74.6	42892
31.83	2.9	208.4				31.83	75.4	43352

Sample: 25wax:75lt hc @ 1:4 crude oil ratio — Temperature: 40°F — Spindle: Vane — Model: RV

Cum. Time minutes	Torque %	Stress (dyne/cm)
32.00	3.0	215.6
32.17	2.9	208.4
32.33	2.9	208.4
32.50	2.9	208.4
32.67	2.9	208.4
32.83	2.9	208.4
33.00	2.9	208.4
33.17	2.9	208.4
33.33	2.9	208.4
33.50	2.9	208.4
33.67	2.9	208.4
33.83	2.9	208.4
34.00	2.9	208.4
34.17	2.9	208.4
34.33	2.9	208.4
34.50	2.8	201.2
34.67	2.9	208.4
34.83	2.9	208.4
35.00	2.9	208.4
35.17	2.9	208.4
35.33	2.8	201.2
35.50	2.8	201.2
35.67	2.9	208.4
35.83	2.8	201.2
36.00	2.8	201.2
36.17	2.8	201.2
36.33	2.8	201.2
36.50	2.8	201.2
36.67	2.9	208.4
36.83	2.8	201.2
37.00	2.8	201.2
37.17	2.9	208.4
37.33	2.8	201.2
37.50	2.8	201.2
37.67	2.8	201.2
37.83	2.8	201.2
38.00	2.8	201.2
38.17	2.7	194.0
38.33	2.7	194.0
38.50	2.7	194.0
38.67	2.7	194.0
38.83	2.7	194.0
39.00	2.7	194.0
39.17	2.7	194.0
39.33	2.6	186.9
39.50	2.6	186.9
39.67	2.6	186.9
39.83	2.6	186.9
40.00	2.6	186.9
40.17	2.5	179.7
40.33	2.6	186.9
40.50	2.5	179.7
40.67	2.5	179.7
40.83	2.5	179.7
41.00	2.4	172.5
41.17	2.4	172.5
41.33	2.4	172.5
41.50	2.4	172.5
41.67	2.3	165.3
41.83	2.3	165.3
42.00	2.3	165.3
42.17	2.3	165.3
42.33	2.3	165.3
42.50	2.3	165.3

Sample: 25wax:75lt hc @ 1:4 crude oil ratio — Temperature: 20°F — Spindle: Vane — Model: HB

Cum. Time minutes	Torque %	Stress (dyne/cm)

Sample: 25wax:75lt hc @ 1:4 crude oil ratio — Temperature: 9°F — Spindle: Vane — Model: HB

Cum. Time minutes	Torque %	Stress (dyne/cm)
32.00	75.9	43639
32.17	76.7	44099
32.33	77.5	44559
32.50	76.1	44904
32.67	78.9	45364
32.83	79.7	45824
33.00	80.4	46227
33.17	81.1	46629
33.33	81.9	47089
33.50	82.6	47492
33.67	83.3	47894
33.83	84.2	48412
34.00	84.8	48757
34.17	85.6	49217
34.33	86.4	49677
34.50	87.0	50022
34.67	87.8	50481
34.83	88.6	50941
35.00	89.3	51344
35.17	90.0	51746
35.33	90.8	52206
35.50	91.4	52551
35.67	92.1	52954
35.83	92.9	53414
36.00	93.6	53816
36.17	94.2	54161
36.33	95.0	54621
36.50	95.7	55024
36.67	96.3	55369
36.83	97.2	55886
37.00	97.8	56231
37.17	98.3	56519
37.33	99.1	56979
37.50	99.8	57381
37.67	100.0	57496

Sample: 25wax:75lt hc @ 1:4 crude oil ratio			Sample: 25wax:75lt hc @ 1:4 crude oil ratio			Sample: 25wax:75lt hc @ 1:4 crude oil ratio		
Temperature : 40°F			Temperature : 20°F			Temperature : 9°F		
Spindle: Vane			Spindle: Vane			Spindle: Vane		
Model: RV			Model: HB			Model: HB		
Cum. Time minutes	Torque %	Stress (dyne/cm)	Cum. Time minutes	Torque %	Stress (dyne/cm)	Cum. Time minutes	Torque %	Stress (dyne/cm)
42.67	2.2	158.1						
42.83	2.2	158.1						
43.00	2.2	158.1						
43.17	2.2	158.1						
43.33	2.1	150.9						
43.50	2.1	150.9						
43.67	2.1	150.9						
43.83	2.1	150.9						
44.00	2.1	150.9						
44.17	2.1	150.9						
44.33	2.1	150.9						
44.50	2.0	143.7						
44.67	2.0	143.7						
44.83	2.0	143.7						
45.00	1.9	136.6						
45.17	2.0	143.7						
45.33	1.9	136.6						
45.50	2.0	143.7						
45.67	1.9	136.6						
45.83	1.9	136.6						
46.00	1.9	136.6						
46.17	1.8	129.4						
46.33	1.8	129.4						
46.50	1.8	129.4						
46.67	1.8	129.4						
46.83	1.8	129.4						
47.00	1.8	129.4						
47.17	1.8	129.4						
47.33	1.7	122.2						
47.50	1.8	129.4						
47.67	1.7	122.2						
47.83	1.7	122.2						
48.00	1.7	122.2						
48.17	1.7	122.2						
48.33	1.7	122.2						
48.50	1.7	122.2						
48.67	1.7	122.2						
48.83	1.7	122.2						
49.00	1.7	122.2						
49.17	1.7	122.2						
49.33	1.6	115.0						
49.50	1.7	122.2						
49.67	1.7	122.2						
49.83	1.6	115.0						
50.00	1.6	115.0						
50.17	1.7	122.2						
50.33	1.6	115.0						
50.50	1.6	115.0						
50.67	1.7	122.2						
50.83	1.6	115.0						
51.00	1.6	115.0						
51.17	1.6	115.0						
51.33	1.6	115.0						
51.50	1.6	115.0						
51.67	1.6	115.0						
51.83	1.7	122.2						
52.00	1.6	115.0						
52.17	1.7	122.2						
52.33	1.7	122.2						
52.50	1.7	122.2						
52.67	1.7	122.2						
52.83	1.7	122.2						
53.00	1.7	122.2						
53.17	1.7	122.2						

Sample: 25wax:75lt hc @ 1:4 crude oil ratio			Sample: 25wax:75lt hc @ 1:4 crude oil ratio			Sample: 25wax:75lt hc @ 1:4 crude oil ratio		
Temperature : 40°F			Temperature : 20°F			Temperature : 9°F		
Spindle: Vane			Spindle: Vane			Spindle: Vane		
Model: RV			Model: HB			Model: HB		
Cum. Time minutes	Torque %	Stress (dyne/cm)	Cum. Time minutes	Torque %	Stress (dyne/cm)	Cum. Time minutes	Torque %	Stress (dyne/cm)
53.33	1.7	122.2						
53.50	1.7	122.2						
53.67	1.7	122.2						
53.83	1.7	122.2						
54.00	1.7	122.2						
54.17	1.7	122.2						
54.33	1.7	122.2						
54.50	1.7	122.2						
54.67	1.7	122.2						
54.83	1.7	122.2						
55.00	1.7	122.2						
55.17	1.7	122.2						
55.33	1.7	122.2						
55.50	1.7	122.2						
55.67	1.7	122.2						
55.83	1.8	129.4						
56.00	1.7	122.2						
56.17	1.7	122.2						
56.33	1.7	122.2						
56.50	1.7	122.2						
56.67	1.7	122.2						
56.83	1.7	122.2						
57.00	1.7	122.2						
57.17	1.7	122.2						
57.33	1.7	122.2						
57.50	1.7	122.2						
57.67	1.7	122.2						
57.83	1.7	122.2						
58.00	1.7	122.2						
58.17	1.6	115.0						
58.33	1.7	122.2						
58.50	1.6	115.0						
58.67	1.7	122.2						
58.83	1.7	122.2						
59.00	1.7	122.2						
59.17	1.7	122.2						
59.33	1.7	122.2						
59.50	1.7	122.2						
59.67	1.7	122.2						
59.83	1.7	122.2						
60.00	1.7	122.2						
60.17	1.7	122.2						

Sample (ii)

25% Wax : 75% GTL + Crude Oil in the Ratio of 1:3

| Sample: 25wax:75GTL @ 1:3 crude oil ratio | Temperature: 40°F | Spindle: Vane | Model: LV | Sample: 25wax:75GTL @ 1:3 crude oil ratio | Temperature: 40°F | Spindle: Vane | Model: RV | Sample: 25wax:75GTL @ 1:3 crude oil ratio | Temperature: 20°F | Spindle: Vane | Model: HB | Sample: 25wax:75GTL @ 1:3 crude oil ratio | Temperature: 9°F | Spindle: Vane | Model: HB |

Cum. Time minutes	Torque %	Stress (dyne/cm)	Cum. Time minutes	Torque %	Stress (dyne/cm)	Cum. Time minutes	Torque %	Stress (dyne/cm)	Cum. Time minutes	Torque %	Stress (dyne/cm)
0.00	0	0	0.00	0.0	0.0	0.00	0.1	57	0.00	0.0	0
7.00	30.5	205	0.17	0.0	0.0	0.17	0.2	115	0.17	0.4	230
7.17	31.2	210	0.33	0.0	0.0	0.33	0.2	115	0.33	0.4	230
7.33	32.1	216	0.50	0.0	0.0	0.50	0.2	115	0.50	0.5	287
7.50	32.9	222	0.67	0.0	0.0	0.67	0.2	115	0.67	0.4	230
7.67	33.7	227	0.83	0.0	0.0	0.83	0.2	115	0.83	0.4	230
7.83	34.5	232	1.00	0.0	0.0	1.00	0.2	115	1.00	0.5	287
8.00	35.2	237	1.17	0.1	7.2	1.17	0.2	115	1.17	0.5	287
8.17	36.0	243	1.33	0.2	14.4	1.33	0.2	115	1.33	0.4	230
8.33	36.9	249	1.50	0.4	28.7	1.50	0.2	115	1.50	0.4	230
8.50	37.6	253	1.67	0.5	35.9	1.67	0.2	115	1.67	0.5	287
8.67	38.4	259	1.83	0.6	43.1	1.83	0.2	115	1.83	0.5	287
8.83	39.2	264	2.00	0.8	57.5	2.00	0.2	115	2.00	0.5	287
9.00	40.1	270	2.17	1.0	71.9	2.17	0.2	115	2.17	0.5	287
9.17	40.8	275	2.33	1.1	79.1	2.33	0.3	172	2.33	0.5	287
9.33	41.7	281	2.50	1.3	93.4	2.50	0.3	172	2.50	0.5	287
9.50	42.4	286	2.67	2.1	150.9	2.67	0.2	115	2.67	0.5	287
9.67	43.2	291	2.83	2.7	194.0	2.83	0.3	172	2.83	0.5	287
9.83	44.0	296	3.00	3.3	237.2	3.00	0.3	172	3.00	0.4	230
10.00	44.8	302	3.17	4.0	287.5	3.17	0.3	172	3.17	0.5	287
10.17	45.6	307	3.33	4.6	330.6	3.33	0.3	172	3.33	0.5	287
10.33	46.4	313	3.50	5.2	373.7	3.50	0.3	172	3.50	0.5	287
10.50	47.2	318	3.67	5.8	416.8	3.67	0.3	172	3.67	0.5	287
10.67	48.0	323	3.83	6.4	460.0	3.83	0.3	172	3.83	0.5	287
10.83	48.8	329	4.00	6.9	495.9	4.00	0.3	172	4.00	0.5	287
11.00	49.6	334	4.17	7.5	539.0	4.17	0.3	172	4.17	0.6	345
11.17	50.4	340	4.33	8.0	575.0	4.33	0.3	172	4.33	0.5	287
11.33	51.2	345	4.50	8.4	603.7	4.50	0.3	172	4.50	0.6	345
11.50	52.0	350	4.67	8.8	632.5	4.67	0.3	172	4.67	0.6	345
11.67	52.8	356	4.83	9.1	654.0	4.83	0.3	172	4.83	0.6	345
11.83	53.6	361	5.00	8.9	639.6	5.00	0.3	172	5.00	0.6	345
12.00	54.5	367	5.17	8.4	603.7	5.17	0.3	172	5.17	0.6	345
12.17	55.1	371	5.33	8.3	596.5	5.33	0.3	172	5.33	0.6	345
12.33	55.9	377	5.50	8.4	603.7	5.50	0.3	172	5.50	0.7	402
12.50	56.8	383	5.67	8.4	603.7	5.67	0.3	172	5.67	0.8	460
12.67	57.5	387	5.83	8.6	618.1	5.83	0.3	172	5.83	1.1	632
12.83	58.4	393	6.00	9.1	654.0	6.00	0.3	172	6.00	1.3	747
13.00	59.2	399	6.17	9.7	697.1	6.17	0.3	172	6.17	1.4	805
13.17	59.9	404	6.33	10.3	740.3	6.33	0.4	230	6.33	1.4	805
13.33	60.7	409	6.50	10.8	776.2	6.50	0.6	345	6.50	1.6	920
13.50	61.6	415	6.67	11.2	804.9	6.67	0.7	402	6.67	1.7	977
13.67	62.3	420	6.83	11.4	819.3	6.83	0.8	460	6.83	1.7	977
13.83	63.1	425	7.00	11.7	840.9	7.00	0.9	517	7.00	2.2	1265
14.00	64.0	431	7.17	12.1	869.6	7.17	1.5	862	7.17	2.7	1552
14.17	64.7	436	7.33	12.7	912.7	7.33	1.8	1035	7.33	2.9	1667
14.33	65.5	441	7.50	13.1	941.5	7.50	1.9	1092	7.50	3.3	1897
14.50	66.3	447	7.67	13.6	977.4	7.67	2.1	1207	7.67	3.5	2012
14.67	67.1	452	7.83	14.1	1013.4	7.83	2.3	1322	7.83	3.6	2070
14.83	67.9	457	8.00	14.5	1042.1	8.00	2.5	1437	8.00	3.9	2242
15.00	68.7	463	8.17	14.9	1070.9	8.17	2.5	1437	8.17	4.2	2415
15.17	69.5	468	8.33	15.3	1099.6	8.33	2.8	1610	8.33	4.4	2530
15.33	70.3	474	8.50	15.6	1121.2	8.50	2.9	1667	8.50	4.6	2645
15.50	71.1	479	8.67	15.9	1142.7	8.67	3.0	1725	8.67	4.9	2817
15.67	71.9	484	8.83	16.3	1171.5	8.83	3.0	1725	8.83	5.1	2932
15.83	72.6	489	9.00	16.7	1200.2	9.00	3.0	1725	9.00	5.4	3105
16.00	73.4	494	9.17	17.1	1229.0	9.17	3.2	1840	9.17	5.8	3335
16.17	74.2	500	9.33	17.7	1272.1	9.33	3.2	1840	9.33	5.9	3392
16.33	75.0	505	9.50	18.0	1293.7	9.50	3.2	1840	9.50	6.4	3680
16.50	75.8	511	9.67	18.5	1329.6	9.67	3.2	1840	9.67	6.8	3910
16.67	76.6	516	9.83	19.0	1365.5	9.83	3.3	1897	9.83	7.0	4025
16.83	77.3	521	10.00	19.3	1387.1	10.00	3.1	1782	10.00	7.3	4197
17.00	78.1	526	10.17	19.6	1408.7	10.17	3.1	1782	10.17	7.6	4370
17.17	78.9	532	10.33	20.1	1444.6	10.33	3.2	1840	10.33	7.8	4485
17.33	79.7	537	10.50	20.6	1480.5	10.50	3.2	1840	10.50	8.2	4715
17.50	80.4	542	10.67	21.0	1509.3	10.67	3.2	1840	10.67	8.3	4772
17.67	81.3	548	10.83	21.5	1545.2	10.83	3.2	1840	10.83	8.5	4887
17.83	82.0	552	11.00	22.0	1581.1	11.00	3.3	1897	11.00	8.9	5117
18.00	82.8	558	11.17	22.3	1602.7	11.17	3.2	1840	11.17	9.2	5290
18.17	83.6	563	11.33	22.8	1638.6	11.33	3.3	1897	11.33	9.6	5520
18.33	84.3	568	11.50	23.2	1667.4	11.50	3.3	1897	11.50	9.8	5635
18.50	85.1	573	11.67	23.7	1703.3	11.67	3.4	1955	11.67	10.1	5807
18.67	86.0	579	11.83	24.1	1732.1	11.83	3.5	2012	11.83	10.5	6037
18.83	86.7	584	12.00	24.4	1753.6	12.00	3.7	2127	12.00	10.8	6210
19.00	87.4	589	12.17	24.7	1775.2	12.17	3.7	2127	12.17	11.0	6325
19.17	88.3	595	12.33	24.9	1789.6	12.33	3.8	2185	12.33	11.3	6497
19.33	89.0	600	12.50	25.4	1825.5	12.50	3.8	2185	12.50	11.8	6785
19.50	89.7	604	12.67	25.7	1847.1	12.67	3.8	2185	12.67	12.0	6900
19.67	90.6	610	12.83	26.2	1883.0	12.83	3.6	2070	12.83	12.4	7130
19.83	91.3	615	13.00	26.5	1904.6	13.00	3.7	2127	13.00	12.6	7244

Sample: 25wax:75GTL @ 1:3 crude oil ratio
Temperature: 40°F
Spindle: Vane
Model: LV

Cum. Time minutes	Torque %	Stress (dyne/cm)
20.00	92.1	620
20.17	92.9	626
20.33	93.7	631
20.50	94.4	636
20.67	95.2	641
20.83	96.1	647
21.00	96.8	652
21.17	97.6	658
21.33	98.4	663
21.50	99.1	668
21.67	99.9	673
21.83	100.0	674

Sample: 25wax:75GTL @ 1:3 crude oil ratio
Temperature: 40°F
Spindle: Vane
Model: RV

Cum. Time minutes	Torque %	Stress (dyne/cm)
13.17	26.7	1918.9
13.33	27.1	1947.7
13.50	27.5	1976.4
13.67	27.8	1998.0
13.83	28.1	2019.5
14.00	28.4	2041.1
14.17	28.7	2062.7
14.33	29.1	2091.4
14.50	29.4	2113.0
14.67	29.6	2127.4
14.83	29.9	2148.9
15.00	30.2	2170.5
15.17	30.4	2184.8
15.33	30.0	2156.1
15.50	30.1	2163.3
15.67	30.5	2192.0
15.83	30.8	2213.6
16.00	31.1	2235.2
16.17	31.4	2256.7
16.33	31.6	2271.1
16.50	31.8	2285.5
16.67	31.9	2292.7
16.83	31.9	2292.7
17.00	32.0	2299.8
17.17	32.0	2299.8
17.33	32.0	2299.8
17.50	32.1	2307.0
17.67	32.1	2307.0
17.83	32.0	2299.8
18.00	31.9	2292.7
18.17	31.8	2285.5
18.33	31.5	2263.9
18.50	31.4	2256.7
18.67	31.2	2242.3
18.83	30.8	2213.6
19.00	30.5	2192.0
19.17	30.2	2170.5
19.33	29.7	2134.5
19.50	29.2	2098.6
19.67	28.7	2062.7
19.83	28.2	2026.7
20.00	27.6	1983.6
20.17	27.2	1954.9
20.33	27.1	1947.7
20.50	26.5	1904.6
20.67	25.8	1854.2
20.83	25.0	1796.8
21.00	24.2	1739.3
21.17	23.6	1696.1
21.33	22.8	1638.6
21.50	22.0	1581.1
21.67	21.4	1538.0
21.83	20.5	1473.3
22.00	19.5	1401.5
22.17	18.5	1329.6
22.33	17.7	1272.1
22.50	16.8	1207.4
22.67	16.2	1164.3

Sample: 25wax:75GTL @ 1:3 crude oil ratio
Temperature: 20°F
Spindle: Vane
Model: HB

Cum. Time minutes	Torque %	Stress (dyne/cm)
13.17	3.8	2185
13.33	3.5	2012
13.50	3.4	1955
13.67	3.5	2012
13.83	3.5	2012
14.00	3.5	2012
14.17	3.5	2012
14.33	3.6	2070
14.50	3.8	2185
14.67	3.8	2185
14.83	3.6	2070
15.00	3.6	2070
15.17	3.8	2185
15.33	3.8	2185
15.50	3.8	2185
15.67	3.8	2185
15.83	3.8	2185
16.00	3.5	2012
16.17	3.7	2127
16.33	3.8	2185
16.50	4.0	2300
16.67	4.1	2357
16.83	4.2	2415
17.00	4.3	2472
17.17	4.4	2530
17.33	4.4	2530
17.50	4.1	2357
17.67	3.9	2242
17.83	3.9	2242
18.00	4.1	2357
18.17	4.0	2300
18.33	4.0	2300
18.50	4.0	2300
18.67	3.8	2185
18.83	3.9	2242
19.00	4.1	2357
19.17	4.2	2415
19.33	3.9	2242
19.50	3.9	2242
19.67	4.0	2300
19.83	4.0	2300
20.00	4.1	2357
20.17	4.3	2472
20.33	4.4	2530
20.50	4.4	2530
20.67	3.8	2185
20.83	3.7	2127
21.00	3.7	2127
21.17	3.9	2242
21.33	3.8	2185
21.50	3.7	2127
21.67	3.6	2070
21.83	3.8	2185
22.00	3.9	2242
22.17	4.0	2300
22.33	4.4	2530
22.50	4.5	2587
22.67	4.6	2645
22.83	4.7	2702
23.00	4.6	2645
23.17	4.8	2760
23.33	4.8	2760
23.50	4.3	2472
23.67	4.3	2472
23.83	4.4	2530
24.00	4.2	2415
24.17	4.2	2415
24.33	4.2	2415
24.50	4.3	2472
24.67	4.5	2587
24.83	4.7	2702
25.00	4.8	2760
25.17	5.0	2875
25.33	4.8	2760
25.50	4.7	2702
25.67	5.0	2875
25.83	4.6	2645
26.00	4.9	2817
26.17	5.1	2932

Sample: 25wax:75GTL @ 1:3 crude oil ratio
Temperature: 9°F
Spindle: Vane
Model: HB

Cum. Time minutes	Torque %	Stress (dyne/cm)
13.17	12.8	7359
13.33	13.3	7647
13.50	13.6	7819
13.67	13.7	7877
13.83	14.1	8107
14.00	14.2	8164
14.17	14.4	8279
14.33	14.8	8509
14.50	14.9	8567
14.67	15.3	8797
14.83	15.5	8912
15.00	15.6	8969
15.17	16.0	9199
15.33	16.4	9429
15.50	16.7	9602
15.67	17.2	9889
15.83	17.6	10119
16.00	18.0	10349
16.17	18.2	10464
16.33	18.7	10752
16.50	19.1	10982
16.67	19.6	11269
16.83	20.4	11729
17.00	20.9	12017
17.17	21.1	12132
17.33	21.7	12477
17.50	22.4	12879
17.67	22.7	13052
17.83	23.4	13454
18.00	23.9	13742
18.17	24.1	13857
18.33	24.5	14087
18.50	25.0	14374
18.67	25.5	14661
18.83	26.3	15121
19.00	26.7	15351
19.17	27.2	15639
19.33	27.9	16041
19.50	28.6	16444
19.67	29.1	16731
19.83	29.7	17076
20.00	30.4	17479
20.17	30.9	17766
20.33	31.5	18111
20.50	32.2	18514
20.67	32.8	18859
20.83	33.5	19261
21.00	34.2	19664
21.17	34.8	20009
21.33	35.5	20411
21.50	36.4	20929
21.67	37.0	21274
21.83	37.8	21733
22.00	38.6	22193
22.17	39.2	22538
22.33	39.9	22941
22.50	40.6	23343
22.67	41.4	23803
22.83	42.0	24148
23.00	42.8	24608
23.17	43.5	25011
23.33	44.1	25356
23.50	45.0	25873
23.67	45.7	26276
23.83	46.3	26621
24.00	47.2	27138
24.17	47.9	27541
24.33	48.6	27943
24.50	49.3	28346
24.67	50.1	28805
24.83	50.6	29093
25.00	51.4	29553
25.17	52.2	30013
25.33	52.8	30358
25.50	53.6	30818
25.67	54.3	31220
25.83	54.9	31565
26.00	55.7	32025
26.17	56.6	32543

Sample: 25wax:75GTL @ 1:3 crude oil ratio			Sample: 25wax:75GTL @ 1:3 crude oil ratio			Sample: 25wax:75GTL @ 1:3 crude oil ratio			Sample: 25wax:75GTL @ 1:3 crude oil ratio		
Temperature :	40°F		Temperature :	40°F		Temperature :	20°F		Temperature :	9°F	
Spindle:	Vane		Spindle:	Vane		Spindle:	Vane		Spindle:	Vane	
Model:	LV		Model:	RV		Model:	HB		Model:	HB	
Cum. Time minutes	Torque %	Stress (dyne/cm)	Cum. Time minutes	Torque %	Stress (dyne/cm)	Cum. Time minutes	Torque %	Stress (dyne/cm)	Cum. Time minutes	Torque %	Stress (dyne/cm)
						26.33	5.4	3105	26.33	57.2	32888
						26.50	5.7	3277	26.50	57.9	33290
						26.67	5.9	3392	26.67	58.8	33808
						26.83	6.1	3507	26.83	59.4	34153
						27.00	6.3	3622	27.00	60.1	34555
						27.17	6.5	3737	27.17	61.0	35073
						27.33	6.7	3852	27.33	61.7	35475
						27.50	6.8	3910	27.50	62.3	35820
						27.67	6.9	3967	27.67	63.2	36337
						27.83	6.8	3910	27.83	63.9	36740
						28.00	6.8	3910	28.00	64.6	37142
						28.17	6.8	3910	28.17	65.5	37660
						28.33	7.0	4025	28.33	66.2	38062
						28.50	7.3	4197	28.50	66.9	38465
						28.67	7.3	4197	28.67	67.8	38982
						28.83	7.4	4255	28.83	68.5	39385
						29.00	7.6	4370	29.00	69.2	39787
						29.17	7.7	4427	29.17	70.0	40247
						29.33	8.0	4600	29.33	70.8	40707
						29.50	8.2	4715	29.50	71.4	41052
						29.67	8.4	4830	29.67	72.2	41512
						29.83	8.7	5002	29.83	73.0	41972
						30.00	9.1	5232	30.00	73.6	42317
						30.17	9.1	5232	30.17	74.4	42777
						30.33	9.4	5405	30.33	75.1	43179
						30.50	9.8	5635	30.50	75.7	43524
						30.67	10.0	5750	30.67	76.5	43984
						30.83	10.2	5865	30.83	77.2	44387
						31.00	10.6	6095	31.00	77.8	44732
						31.17	10.8	6210	31.17	78.6	45192
						31.33	11.2	6440	31.33	79.3	45594
						31.50	11.7	6727	31.50	79.9	45939
						31.67	12.1	6957	31.67	80.7	46399
						31.83	12.6	7244	31.83	81.4	46802
						32.00	13.2	7589	32.00	81.9	47089
						32.17	13.7	7877	32.17	82.6	47492
						32.33	14.1	8107	32.33	83.3	47894
						32.50	14.6	8394	32.50	83.7	48124
						32.67	15.1	8682	32.67	84.1	48354
						32.83	15.7	9027	32.83	84.8	48757
						33.00	16.4	9429	33.00	85.5	49159
						33.17	16.9	9717	33.17	86.1	49504
						33.33	17.3	9947	33.33	87.0	50022
						33.50	17.9	10292	33.50	87.8	50481
						33.67	18.4	10579	33.67	88.5	50884
						33.83	18.9	10867	33.83	89.3	51344
						34.00	19.6	11269	34.00	90.1	51804
						34.17	20.2	11614	34.17	90.8	52206
						34.33	20.6	11844	34.33	91.6	52666
						34.50	21.3	12247	34.50	92.5	53184
						34.67	22.0	12649	34.67	93.1	53529
						34.83	22.4	12879	34.83	93.9	53989
						35.00	23.1	13282	35.00	94.8	54506
						35.17	23.9	13742	35.17	95.4	54851
						35.33	24.4	14029	35.33	96.3	55369
						35.50	25.2	14489	35.50	97.1	55829
						35.67	26.0	14949	35.67	97.6	56116
						35.83	26.6	15294	35.83	98.4	56576
						36.00	27.4	15754	36.00	99.3	57094
						36.17	28.1	16156	36.17	99.9	57439
						36.33	28.7	16501	36.33	100.0	57496
						36.50	29.5	16961			
						36.67	30.3	17421			
						36.83	30.9	17766			
						37.00	31.7	18226			
						37.17	32.4	18629			
						37.33	33.1	19031			
						37.50	33.8	19434			
						37.67	34.6	19894			
						37.83	35.3	20296			
						38.00	35.9	20641			
						38.17	36.7	21101			
						38.33	37.4	21504			
						38.50	38.0	21848			
						38.67	38.8	22308			
						38.83	39.5	22711			
						39.00	40.0	22998			
						39.17	40.7	23401			
						39.33	41.4	23803			

Appendix B

Standard Laboratory Procedure

(SLP-307)

"Crude Oil Yield Point Determination By Vane Viscometry"

Westport Technology Center **Standard Laboratory Procedure**

| Subject: **Crude Oil Yield Point Determination by Vane Viscometry** | Document No: SLP-307 |
| Date: July 31, 2000 | Revision 3 | Page 1 of 13 |

| Prepared by: Neal Magri |
| Applicable to: Westport Technology Center International |

Technical Review by: Bayram Kalpakci	Date: July 31, 2000
Safety Review by: Robert Jaros	Date: July 31, 2000
Quality Assurance Review: John Shillinglaw	Date: July 31, 2000

Scope: This test method describes the use of the Brookfield viscometer for the determination of the yield point of crude oils.

Safety Precautions: Approved safety glasses with side shield and protective clothing must be worn at all times in the laboratory. Protective gloves are required to be worn when handling crude oil and solvents. Keep away heat, sparks and open flame. Use only with adequate ventilation, (i.e. sampling performed within a fume hood if possible). Avoid contact with skin, eyes and clothing. Avoid breathing mist or vapor. Keep containers closed. Open containers with caution.

Important: Crude oil and container will be hot after the initial heating during the beneficiation process. Handle and dispose of syringes and needles properly. Empty containers may contain toxic, flammable/combustible or explosive residue or vapors. Do not cut, grind, drill, weld, reuse or dispose containers unless adequate precautions are taken against these hazards. Observe ALL PRECAUTIONARY LABELING.

| **Hazard:** **CRUDE OIL**, Vapors may be harmful. Possible aspiration hazard if swallowed, can enter lungs and cause damage. May be irritating to the skin, eyes and respiratory tract. May release toxic hydrogen sulfide vapors. Skin cancer hazard based on tests with laboratory animals. Contains **BENZENE**—a cancer hazard. Extremely flammable liquid. Vapor may cause flash fire. |

Reference Documents: ASTM D2983 Standard Test Method for Low-Temperature Viscosity of Automotive Fluid Lubricants; Westport Standard Procedure SLP-305 Rheological Properties of Crude Oils by Rotational Viscometer; Brookfield Digital Viscometer Model DV-II+ Version 2.0, Operating Instructions, Manual No. M/92-161-F1193; Brookfield WinGather Software, Operating Instructions, Manual No. M/95-320-C398; Operations and Programming Manual for Sigma Systems M26-C3 Environmental Chamber with Programmable Temperature Controller / Model CC-3. Dzuy, N.Q., Boger, D.V., "Yield Stress Measurement for Concentrated Suspensions", J. Rheology, 27 (4), 321-9 (1983).

Westport Technology Center   Standard Laboratory Procedure

Subject: **Crude Oil Yield Point Determination by Vane Viscometry**	Document No: SLP-307
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Summary of Test Method

This test method consists of determining the yield point of a crude oil by measuring the torque on a spindle, using a Brookfield viscometer, rotating at 0.01 rpm in the material. The spindle to be used consists of four rectangular vanes dimensioned (0.75"w x 2.25"h) and oriented at 90 degree increments around the central axis. The sample cup is dimensioned (1.5" id x 4.0"h). Vertical orientation of vanes within the sample cup is dimensioned (1.00" from top and 0.75" from bottom). The crude oil is initially heated to 150°F to destroy all temperature and shear histories and then cooled to 90°F at which point it is loaded into the cup apparatus. The cup apparatus holds the vanes rigidly during cooling and aging and prevents loss of light ends through evaporation. After loading into the cup apparatus the sample is cooled in an environmental chamber at a controlled rate to -20°F. The cooling rate mimics the expected rate of cooling of the pipeline oil in the case of shut-in.

Samples are withdrawn from the environmental chamber at 10 test temperatures (approximately 80, 65, 50, 40, 20, 10, 0, -10, -15 and -20°F) and transferred to a refrigerated circulator that maintains the sample at test temperature. The spindle is attached to the Brookfield viscometer before the spindle clamping mechanism is released. The clamping mechanism is released and the viscometer is started at 0.01 rpm and torque as a function of time is measured, at least until a maximum reading is obtained. The maximum torque obtained is divided by a vane parameter constant K to obtain the yield stress. The constant K is calculated based on the dimensions of the vanes. (K=36.19 cm³, for a Vane with D=0.75 inch (1.905 cm) and H=2.25 inch (5.715 cm))

Significance and Use

The test method is used for determining the yield point of a cooled crude oil, with or without aging. This determination will be made with vane spindles, which extend horizontally through a sample, minimizing the impact of slippage at the spindle wall. The method will determine the minimum amount of torque necessary to initiate oil movement at low shear, and subsequent viscosity of the fluid after initiation of flow. These data can be directly used in modeling of crude oil behavior in pipelines, during start-up conditions.

Equipment Required

- Certified rotational-type viscometers capable of a minimum rotational speed of 0.01 rpm such as: the Brookfield Viscometer, Model LV DVII+, RV DVII+ or HB DVII+ having the capability of 20 speeds when programmed accordingly.
- Thermometer, Fluke digital thermometer which has been certified to ±0.3"C from −40"C to 100"C and ±2.0"C from −50"C to −100"C, using standards traceable to the National Institute of Standards and Technology or the National Physical Laboratory or using natural physical constants or ratio calibration techniques.
- Westport vane viscometry cup apparatus and vane spindle for the Brookfield viscometer.
- 100 ml glass syringe, and syringe needle equipped with valve.
- Sigma System M26-CC3 environmental chamber, equipped with a liquid nitrogen supply for cooling.
- Temperature controlled refrigerated circulator bath, such as a Julabo FP-50 series.

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PROCEDURES

A. Calibration of Apparatus

Calibration as it is normally understood, carrying out an experimental measurement with a standard material, in the same manner as for the unknown sample, does not apply to vane viscometry: there are no standards. However, as received, from the factory the Brookfield viscometers are certified to give accurate speed and percent torque readings, and it is these two measurements which are critical to vane viscometry.

Tests of consistent viscometer response will be carried out as verification that the viscometer is in good working order. Testing will be carried out for each viscometer when used in each experimental set (an experimental set entails the testing of all samples cooled in one set in an environmental chamber, generally a population size of 24).

1.0 Procedure

1.1 Level the Brookfield viscometer, and the plastic bracket to hold the vane apparatus using the spirit levels attached to them.

1.2 Take an empty vane apparatus, with the top removed but with the spindle locked, and place the apparatus in the refrigerated circulator, temperature is not critical at this step.

1.3 The vane apparatus is held in place by a plastic bracket; make certain the apparatus is placed as far to the right as possible and as far towards the back of the bath as possible.

1.4 Tighten the clamp that holds the vane apparatus in the bracket.

1.5 Attach the alignment rod to the Brookfield viscometer. Move the Brookfield viscometer vertically and horizontally until the alignment rod fits into the top of the vane spindle (without lateral motion).

1.6 Remove the alignment rod and screw on the S hook attachment to the Brookfield viscometer.

1.7 Loosen the clamp that holds the vane apparatus in the bracket.

1.8 Attach the vane spindle to the Brookfield viscometer as shown in Figure 1.

1.9 Raise the Brookfield viscometer until all slack is taken out of the connections to the vane spindle but the vane spindle is not lifted off its plastic support bracket.

1.10 Rotate the vane apparatus until the torque reading is less than 0.05%. It is best not to start with negative torque readings as these are not recorded by the WinGather program.

1.11 Tighten bracket that holds the vane apparatus in place, using the knob in the top right corner of the bracket.

1.12 Start the WinGather program for timed torque readings. Time between readings will be 10 seconds for a speed of 0.01 rpm.

As the Brookfield software does not recognize the vane spindle. spindle input is not necessary. Time, torque and temperature readings. which are independent of spindle, are the only data that will be used.

The WinGather program is Version 1.1 from Brookfield Engineering Laboratories. Inc., located at 11 Commerce Blvd., Middleboro. MA 02346. See referenced manual for further WinGather information. The manual and a copy of the software will be archived at Westport Technology Center.

1.13 Start the Brookfield motor with the locked spindle mechanism in place.

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1.14 Continue the test run until the torque reading goes off scale.

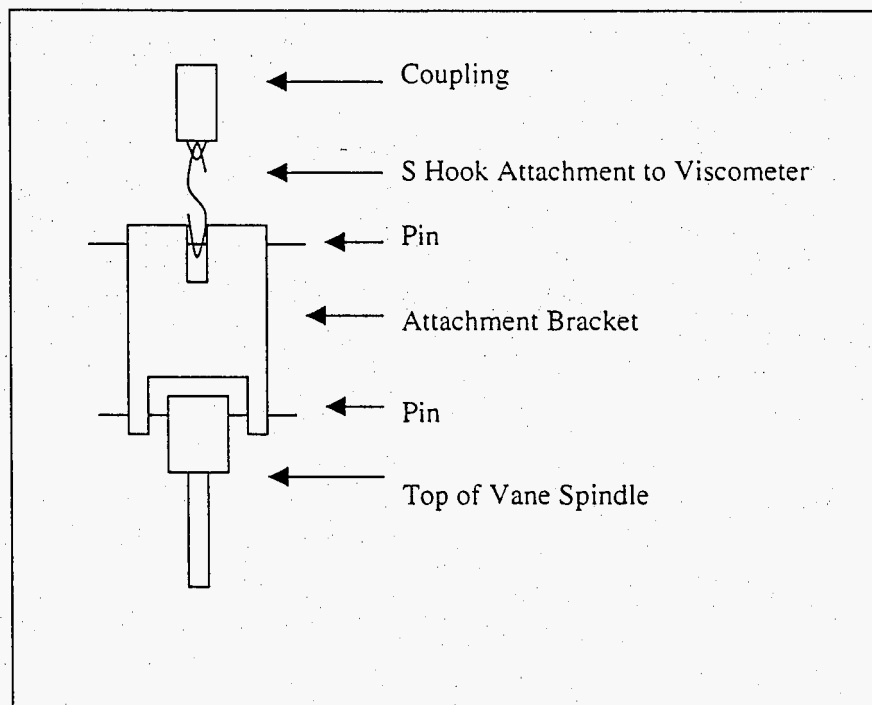1.15 Save the WinGather data file and record the data file name.

Figure 1. Attachment of Vane Apparatus to Viscometer

2.0   Acceptable Calibration

Differences in torque versus time measurements will include variations in viscometer speed and torque measurement. For purposes of measuring yield stress, variances in viscometer speed are not critical. Speed is important however for determining slippage of the sample at the yield point. A torque versus time run will be acceptable if it varies no more than 10% of the maximum torque of the viscometer, at any point in the experimental run.

B.   Verification of Cooling Rate

1.0 Data Recording

Temperatures will be measured by the environmental chamber thermocouple and recorded by a YEW Model 3088 Hybrid Recorder (chart recorder). The chart recorder will print time and temperature at 0:00 and 12:00 each day, in addition to a continuous trace of the temperature. This temperature recording procedure has been verified against a certified Fluke thermometer and found to be within

±1°F. The temperature recording of the environmental chamber and Yew recorder will be re-verified each time the Fluke digital thermocouple meters are re-certified.

1.1   At the end of each vane experimental run, when all samples originally placed in the environmental chamber have been tested or disposed of, remove the chart recorded paper from the recorder. This data is to be saved, and archived with other items for this test method, such as laboratory notebooks.

1.2   The chart recording should be inspected to ascertain if there are any significant anomalies. such as rapid, transitory increases or decreases in temperature.

1.3   Printed temperatures from the chart recorder should be recorded and contrasted with target temperatures as part of final reporting.

Table 1. Times and Target Temperatures

Day (24 hrs)	Target Temperature (°F)
0	90
2.5	70
5.0	50
7.5	35
10	20
12.5	10
15	0
17.5	-10
21	-20

C. Preparation of Sample

The solubility of paraffins in crude oil decreases with decreasing temperature. As an oil cools past its wax appearance temperature, regardless of the rate at which the oil was cooled. significant amounts of paraffin may precipitate. However, the rheological properties of the precipitated wax are highly dependent upon the shear and temperature history of the oil above and below the wax appearance temperature. The initial step in determining yield stress of an oil is to heat the sample to 150°F and hold the oil at that temperature for at least 2 hours, to destroy all temperature and shear histories. It is important to make certain the oil container is tightly closed during this initial conditioning. as loss of light ends through evaporation may significantly increase yield stress.

Following the initial heating of the oil. allow the oil to cool to 90°F in a Sigma environmental chamber. At this point the oil containers, the vane apparatuses and the 100-ml glass syringe for oil transfer are thermally equilibrated in the environmental chamber at 90°F. Allow at least an hour for all materials to reach temperature.

D. Loading of Vane Apparatus

A 100-ml glass syringe equipped with needle and valve is used to load the oil sample into the vane apparatus.

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19. Save the WinGather data file and record the data file name.
20. At test completion, insert certified Fluke thermocouple into vane apparatus and take a final sample temperature.
21. Repeat the test as stated:
    - Under test conditions where (possibly above the oil's WAT) observed torque readings are below 10% full scale on the LV viscometer (lowest torque spring viscometer) only test one sample.
    - If torque readings are above 10% full scale on the LV, then run a second sample for repeatability. If the two test results are not within 10%, run a third sample for precision purposes.
    - Three samples should always be tested at the lower temperatures if test samples are available. If necessary, for precision statements, repeat additional times.

G. Data Recording

Initiate Data Verification and Validation Checklists. Data recording, which will include quality assurance tests such as calibration and data checks, will be achieved using the enclosed vane viscometry data sheets.

Data Sheet 1 (DS1-307) includes recording of data when all the samples, for an entire tests, are prepared, transferred to their respective vane apparatuses, cooled to test temperature and aged.

The data to be recorded include:

- Test Number; Designate the test by number so that subsequent individual vane data sheets (DS2-307) can be traced to the parent data sheet. (i.e. IDC Test 1, Phase 1–Test 1...)
- Sample Description; Identification of sample (i.e. TAPS Mix, PBU, Kuparuk...)
- Sample Benefication; Record temperature treatment information for the samples before transfer to the vane apparatuses.
- Transfer of Samples; Record start and stop times for transfer of all samples from original bottles to vane apparatuses to environmental chambers.
- Cooling/Aging of Sample: Record temperature of environmental chamber when samples are first introduced, also record target temperature (normally –20°F) and the target period of time for cooling samples to that temperature (normally 21 days). Temperature/time measurements will be recorded by the Yew hybrid chart recorder.

Data are to be recorded for each individual sample withdrawn and tested from the environmental chambers on vane viscometry Data Sheet 2 (DS2-307). Record data including:

- Test Number; the same number as used on DS1-307, establishes tracking of DS2-307 sheets.
- Sample Description; Identification of sample (i.e. TAPS Mix, PBU, Kuparuk...)
- Testing; date and time of sample withdrawal, the temperature of the environmental chamber, the name of the WinGather data file where testing data is stored, the type of viscometer (LV, RV or HB) and the speed (rpm) at which the test was carried out (normally 0.01).

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- Data Check; data for the vane testing will be recorded using the WinGather program. The data check section of the data sheet will be used for quality assurance of this WinGather data. During testing (at 5 to 10 minute intervals), record the percent full scale (torque) reading directly from the Brookfield viscometer display, and record the data point of the WinGather program where this data was recorded electronically.
- Maximum Torque; Record the observed highest percent full-scale reading (torque).
- Data Points of Maximum Torque; Record the approximate range of WinGather points during maximum torque (i.e. numbers 73-78).
- Final Oil Temperature; Record the sample temperature after completion of test.

H. Calculation and Interpretation of Results

The experimental test is designed to produce direct readings. temperature and %torque, as data.

Calculation of torque and yield stress.

1. Calculate torque readings from the percent of full-scale readings recorded by the WinGather software by multiplying the percent full-scale reading by 6.733 dyne-cm for the LVDV-II+ viscometer, or 71.87 dyne-cm for the RVDV-II+ viscometer, and 574.96 for the HBDV-II+ viscometer. Torque data may be interpreted by graphing the torque versus time readings obtained during testing. Determine a yield point from this data by observing where a maximum torque reading had been obtained. followed by a decrease in torque reading over time. Calculate the Yield Stress by the following equation;

$$\text{Yield Stress} = \text{Maximum Torque Obtained (dyne-cm)/ K (36.19 cm}^3)$$

I. Reporting

1. Report the following information:
   1.1 Completed and signed Data Verification and Validation Checklists,
   1.2 Date of test,
   1.3 Sample Identification,
   1.4 Cooling time for sample,
   1.5 Aging/testing temperature in degrees Fahrenheit (environmental chamber temp.),
   1.6 Final oil temperature at end of test (measured directly in cup),
   1.7 Viscometer speed.
   1.8 Maximum torque reading.
   1.9 Yield Strength versus Temperature,
   1.10 Locked Spindle Torque versus Time Curve,
   1.11 Time versus torque reading for each test will be reported graphically,
   1.12 Numerical data for the graph,
   1.13 Combined Plot of all Torque vs. Time Curves on one Log Scale Plot,
   1.14 When multiple oils are analyzed, plot all Yield Strength vs. Temperature curves on one Lop Scale Plot.

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J. Precision

Precision – See Section A2 for precision during QC/QA calibration checks. Initial Demonstration of Capability for test resulted in the following:

Determinability (d) – Measurements are performed on individual oil samples, taken at selected temperatures, through a maximum shear condition which destroys any wax structure present. No attempt was made to duplicate measurements on the same oil sample within the test cell; the results would be misleading. Therefore, no statement of determinability can be made on.

Repeatability (r) - The difference between successive results obtained by the same operator in the same laboratory with the same apparatus under constant operating conditions on identical test material would, in the normal and correct operation of this method, have a relative standard deviation at or below 15%.

Reproducibility (R) - The Brookfield viscometers are a very common apparatus for measuring rheological properties. However, with the Vane cup and spindle apparatus design and modifications for low temperature testing of the TAPS/COS samples, no statement of reproducibility by other independent laboratories can be made.

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Data Sheet 1 for Vane Viscometry   Form: DS1-307

Heating, Transfer, Cooling, and Aging for Sample Sets

Test Number:	
Sample Description:	

Sample Beneficiation				Transfer of Samples	
Date:		Start Time:		Start Time:	
Temp.:		Stop Time:		Stop Time:	

Cooling/Aging of Samples

Chamber Temp.:		Target Temp.:	
Ramp Start Time		Target Time:	

Sample Identification

Chamber 1		Chamber 2	
Vane No.	Sample ID	Vane No.	Sample ID

Analyst Signature:_____

Subject:	QA Procedure for Crude Oil Quantification by Capillary Gas Chromatography
Date:	January 4, 2001

Prepared by: Ray Collins	Date: January 4, 2001

Safety Review by: Robert Jaros	Date: January 4, 2001

Scope: This procedure gives quantitative compositions of crude oils and condensates utilizing capillary gas chromatography (CGC).

Safety Precautions: Approved safety glasses with side shield and protective clothing must be worn at all times in the laboratory. Protective gloves must be worn when handling crude oil and solvents. Keep away heat, sparks, and open flame. Use only with adequate ventilation, (i.e. sampling performed within a fume hood if possible). Avoid contact with skin, eyes, and clothing. Avoid breathing mist or vapor. Keep containers closed. Open containers with caution.

Calibration Standards: Prudhoe Bay oil, Identifier: Reference "C".
Colombian oil, Identifier: Reference "W".
D-2887 Reference Gas Oil, Identifier: RGO.

PROCEDURES

Sample Preparation

The sample used in CGC analysis must be free of water and solids. One to 1.3 grams of sample is weighed into a 2ml auto-sampler vial and then a fixed known amount of internal standard is weighed into the sample. The sample is then thoroughly mixed to ensure sufficient mixing of sample and internal standard. Carbon disulfide is added to viscous samples that are too thick to be drawn into a syringe.

Gas Chromatograph Preparation

Prior to analyzing samples, the gas chromatograph (GC) is heated to the columns upper temperature limit to remove contaminants and stabilize the baseline. The inlet septum is changed weekly to reduce septum bleeding.

Calibration of GC Apparatus

The GC is a computer controlled, method-driven gas chromatograph. Internal testing of the instrument functions is carried out upon startup. The standard calibration curve is determined by repeated analyses of Prudhoe Bay (Ref "C") crude oil standard. Accepted values (+/- 3 percent

Subject:	QA Procedure for Crude Oil Quantification by Capillary Gas Chromatography
Date:	January 4, 2001

per component – upper and lower control limits) are plotted versus the results of the Ref "C" analysis that was treated as an unknown sample (see Attachment-1). Similar plots of the D-2887 Reference Gas Oil, Reference "W", and Ref "C" with carbon disulfide standard are plotted when used. If two or more points of the results from the analysis are outside of the control limits, the GC must be undergo maintenance to restore acceptable capabilities.

GC Sequences

A <u>sequence</u> of samples run on the GC consists of a suite of standard blanks and at least one unknown sample. The order of an extended sequence is given on the right. The standards analyzed at the beginning of the sequence are treated as unknowns. If the resultant CGC calculation agrees with the results in Attachment-1, the instrument is deemed to be within calibration specifications and the remainder of the sequence can proceed. The intermediate standards run at the end of each subsequence are similarly treated as unknowns. If they are out of calibration, the sequence is terminated and the preceding subsequence is re-run. If the intermediate standard is acceptable (results within the criteria specified), the sequence proceeds to the next subsequence. The ending standard is treated in the same way as an intermediate standard (as an unknown sample).

```
Table 2: Test Sequence

Blank
Blank
Standard
Subsequence 1
          Sample 1
          Sample 2
          ...
          Sample 10
Standard
Subsequence 2
          Sample 11
          Sample 12
          ...
Ending Standard
```

The ending standard is a discrimination standard, which consists of normal paraffins ranging from C-13 through C-30; this standard is used to verify the GC's suitability for the next sequence of samples by comparing the relative response factors of the normal paraffins that must be between 0.95 and 1.05. If the factors fall out of acceptable region, then corrective maintenance must be performed on the injector system and the GC must be re-tested with the discrimination standard.

Data Acquisition

Agilent Technology's Chemstation is used to control and acquire the gas chromatographic data. The Chemstation software provides accurate pressure control to ensure repeatable retention times from sequence to sequence. To provide additional retention time repeatability, Retention Time Locking software is used to lock the retention time base on a known compound. These capabilities ensure correct identification of compounds.

Westport Technology Center   **Standard Laboratory Procedure**

Subject:	QA Procedure for Crude Oil Quantification by Capillary Gas Chromatography
Date:	January 4, 2001

Attachment 1

WESTPORT
TECHNOLOGY CENTER INTERNATIONAL
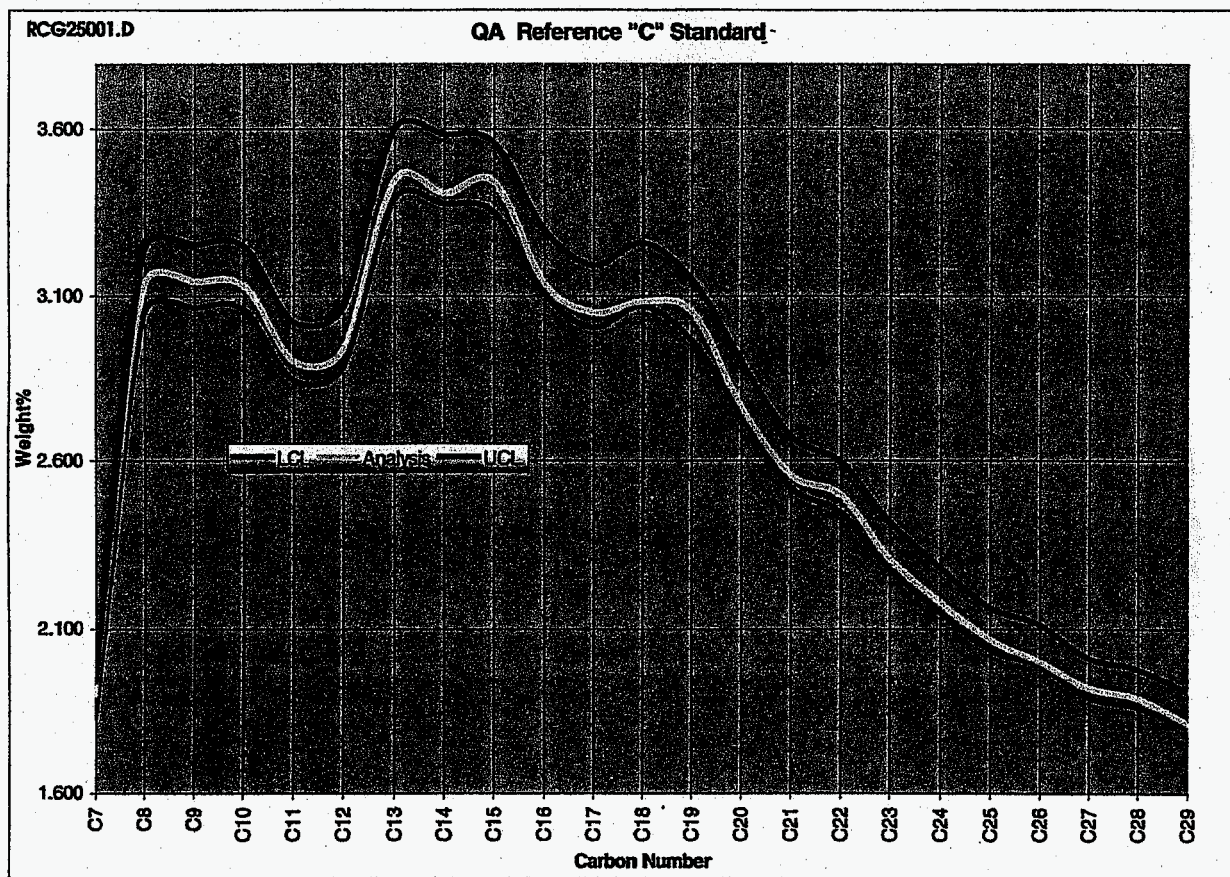
ATTACHMENT 2

Evaluation of GTL #2 and Crude Oil Blends
For Yield Stress Values

A REPORT BY WESTPORT TEHCNOLOGY CENTER

WESTPORT
TECHNOLOGY CENTER INTERNATIONAL

RT-01-038

November 2001

Evaluation of GTL #2 and Crude Oil Blends
For Yield Stress Values

Final Report

Work By:
S. Brown, X. Zhou

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Table of Contents

Introduction

The gel strength of various GTL (gas to liquid samples) and North Slope crude oil blends were determined by the rotating vane method. The tests for determining the yield stress, or yield stress value, of the cooled crude oil blends, were performed following Westport's Standard Laboratory Procedure (SLP) 307, "Crude Oil Yield Stress value Determination by Vane Viscometry". This determination was made with Brookfield rotary viscometers and vane spindles, which extend horizontally through a sample, minimizing the impact of slippage at the spindle wall. This method determines the minimum amount of torque necessary to initiate oil movement at low shear, and subsequent gel breakdown after initiation of flow. These data can be directly used in modeling of crude oil behavior in pipelines, during start-up conditions.

Test Results

Twenty-four test samples were prepared by weight for testing, eight each at the three ratios listed below:

(a). 100% Light Hydrocarbon GTL #2
(b). 25% Light Hydrocarbon GTL #2 + 75% TAPS Mix Crude at PS-1
(c). 20% Light Hydrocarbon GTL #2 + 80% TAPS Mix Crude at PS-1

The client supplied the light hydrocarbon GTL samples. The crude oil sample was supplied by the Alyeska Pipeline Service Company, taken from the flowing TAPS mixed stream at Pump Station 1. All blend mixes were carried out by weight to weight measurements.

Samples were tested at selected temperatures as the crude oil blends were cold ramped from 90°F to -20°F over a twenty-one day period. The maximum recorded torque obtained during vane rotation at a constant speed of 0.01 rpm was converted into a yield stress value. The summary of vane test results is presented in Table 1 and Figure 1.

Initially, test temperatures were set at 20°F, 0°F and –20°F. However, simple bottle testing of the GTL #2 sample indicated possible gel strength onset at earlier temperatures. Therefore, tests were performed on the 100% GTL at 27°F and a yield stress of 171 dynes/sqcm were recorded.

At a temperature of 20°F the GTL #2 sample had reached it's pour point value and gel strength was beyond the limits of the RV viscometer. Averaged yield stress values of 2.91 dynes/sqcm for the 3:1 ratio blend and 1.13 dynes/sqcm for the 4:1 ratio blend were measured. The 100% GTL produces high gel strength, but when blended with TAPS mix crude a significant reduction occurred. It is also evident that ratio blending also contributes to lower yield stress values.

At a temperature of 0°F the 3:1 ratio blend with TAPS mix crude produced an average yield stress value of 694 dynes/sqcm. The 4:1 ratio blend with TAPS mix crude produced an average yield stress value of 389 dynes/sqcm. This again supports that ratio blending may be effective in lowering the ultimate yield stress values of GTL/crude oil blends.

The effect on yield stress values from ratio blending was not apparent in prior testing performed with the GTL #1 sample. At present time, testing has not been performed with alternative crude oil samples to determine if the effect of ratio blending is reproducible with varying crude composition.

All testing performed at a temperature of –20°F produced yield stress values beyond the measurable limits of the Vane test equipment, or values greater than 1589 dynes/sqcm.
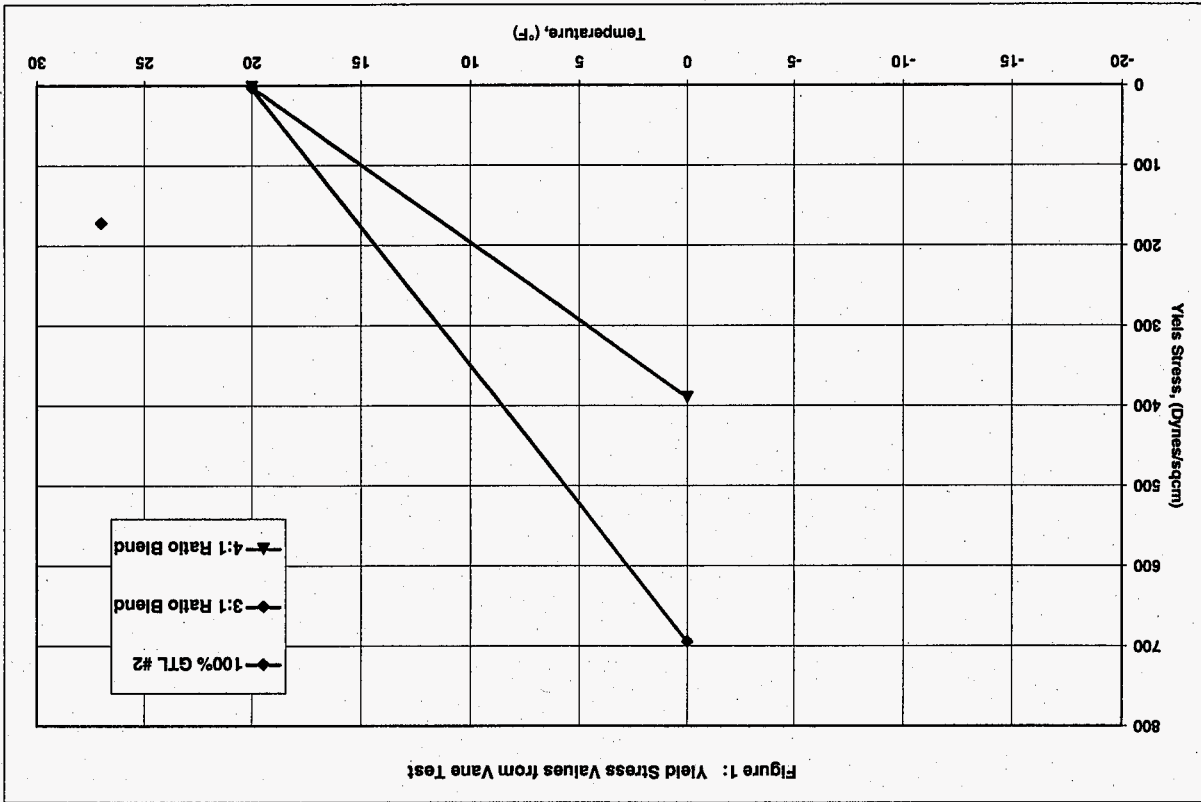
Figure 1: Yield Stress Values from Vane Test

Table 1: Yield Stress by Vane Viscometer

Test Sample (ID #)	Brookfield Viscometer Model	Sample Temperature (°F)	Maximum Torque Response (% full scale)	Yield Point Torque (dyne-cm)	Yield Stress (dynes/sqcm)
GTL-01	RV	27	failed	-	-
GTL-02	RV	27	86.3	6202	171
GTL-03	RV	20	>100	>7187	>199
GTL-04	RV	0	>100	>7187	-
GTL-05	HB	0	>100	>57496	>1589
GTL-06	HB	0	>100	>57496	>1589
GTL-07	HB	-20	>100	>57496	>1589
GTL-08	HB	-20	<100	>57496	>1589
3:1-01	RV	20	1.5	108	2.98
3:1-02	RV	20	1.3	93.4	2.58
3:1-03	RV	20	1.6	115	3.18
3:1-04	LV	0	>100	>674	-
3:1-05	HB	0	41.3	23746	656
3:1-06	HB	0	46.1	26506	732
3:1-07	HB	-20	>100	>57496	>1589
3:1-08	HB	-20	>100	>57496	>1589
4:1-01	RV	20	0.4	28.7	0.79
4:1-02	RV	20	0.9	64.7	1.79
4:1-03	RV	20	0.4	28.7	0.79
4:1-04	LV	0	>100	>674	-
4:1-05	RV	0	>100	>7187	-
4:1-06	HB	0	21.9	12592	348
4:1-07	HB	0	27.1	15581	431
4:1-08	HB	-20	>100	>57496	>1589

100% GTL #2

Temperature (°F)	Yield Stress (average) (dynes/sqcm)
27	171
20	>199
0	>1589
-20	>1589

3:1 Ratio Blend

Temperature (°F)	Yield Stress (average) (dynes/sqcm)
20	2.91
0	694
-20	>1589

4:1 Ratio Blend

Temperature (°F)	Yield Stress (average) (dynes/sqcm)
20	1.13
0	389
-20	>1589

SECTION 1

Yield Stress versus Elapsed Time
For
GTL #2 & TAPS Crude Oil Blends

Summary of Test Method

Westport's SLP-307 consists of determining the yield stress value of a crude oil by measuring the torque on a spindle, using a Brookfield viscometer, rotating at 0.01 rpm in the material. The spindle to be used consists of four rectangular vanes dimensioned (0.75"w x 2.25"h) and oriented at 90 degree increments around the central axis. The sample cup is dimensioned (1.5" id x 4.0"h). Vertical orientation of vanes within the sample cup is dimensioned (1.00" from top and 0.75" from bottom).

The crude oil blends were initially heated to 150°F to destroy all temperature and shear histories and then cooled to 100°F at which stress value they were loaded into the vane closed-cup apparatus. The closed-cup apparatus holds the vanes rigidly during cooling and aging and prevents loss of light ends through evaporation. After loading into the cup apparatus the samples were cooled in an environmental chamber at a controlled rate to below 0°F. The cooling rate mimics the expected rate of cooling of the Trans-Alaska pipeline oil in the case of shut-in.

Samples were withdrawn from the environmental chamber at five test temperatures (approximately 60, 40, 20, 9 and 0°F) and transferred to a refrigerated circulator that maintains the sample at test temperature. The spindle was attached to the Brookfield viscometer (LV, RV or HB) before the spindle clamping mechanism was released. The clamping mechanism was released and the viscometer was started at 0.01 rpm and torque as a function of time was measured, until a maximum reading was obtained. The maximum torque (dyne-cm) obtained is divided by a vane parameter constant K to obtain the yield stress (dynes/sqcm). The constant K is calculated based on the dimensions of the vanes. ($K=36.19$ cm^3, for a Vane with D=0.75 inch (1.905 cm) and H=2.25 inch (5.715 cm). For further detailed information Westport's SLP-307 is attached in Appendix B.

Temperature Ramping Profile

The temperature decay curve used for test sample preparation was taken from Trans-Alaska Pipeline cold restart data supplied by the Alyeska Pipeline Service Company. Based on this curve, selective temperatures were entered into the program menu of the environmental cooling chamber, a cryogenic chamber cooled by liquid Nitrogen vapor. Temperatures are recorded on strip chart display with digital inscriptions at 12-hour intervals. The programmed temperature ramp is presented in Table 2 and Figure 2.

TABLE 2, TEST TEMPERATURE PROFILE
VANE AND ROTARY VISCOMETRY

Environmental Chamber Temperature Ramp Program				
Step #	Days	Hours	Temp °F	Temp °C
0	0	0	90	32.2
1	2.5	60	70	21.1
2	5	120	50	10.0
3	7.5	180	35	1.7
4	10	240	20	-6.7
5	12.5	300	10	-12.2
6	15	360	0	-17.8
7	17.5	420	-10	-23.3
8	21	504	-20	-28.9
9	Hold	576	-20	-28.9

Figure 2: Test Temperature Profile
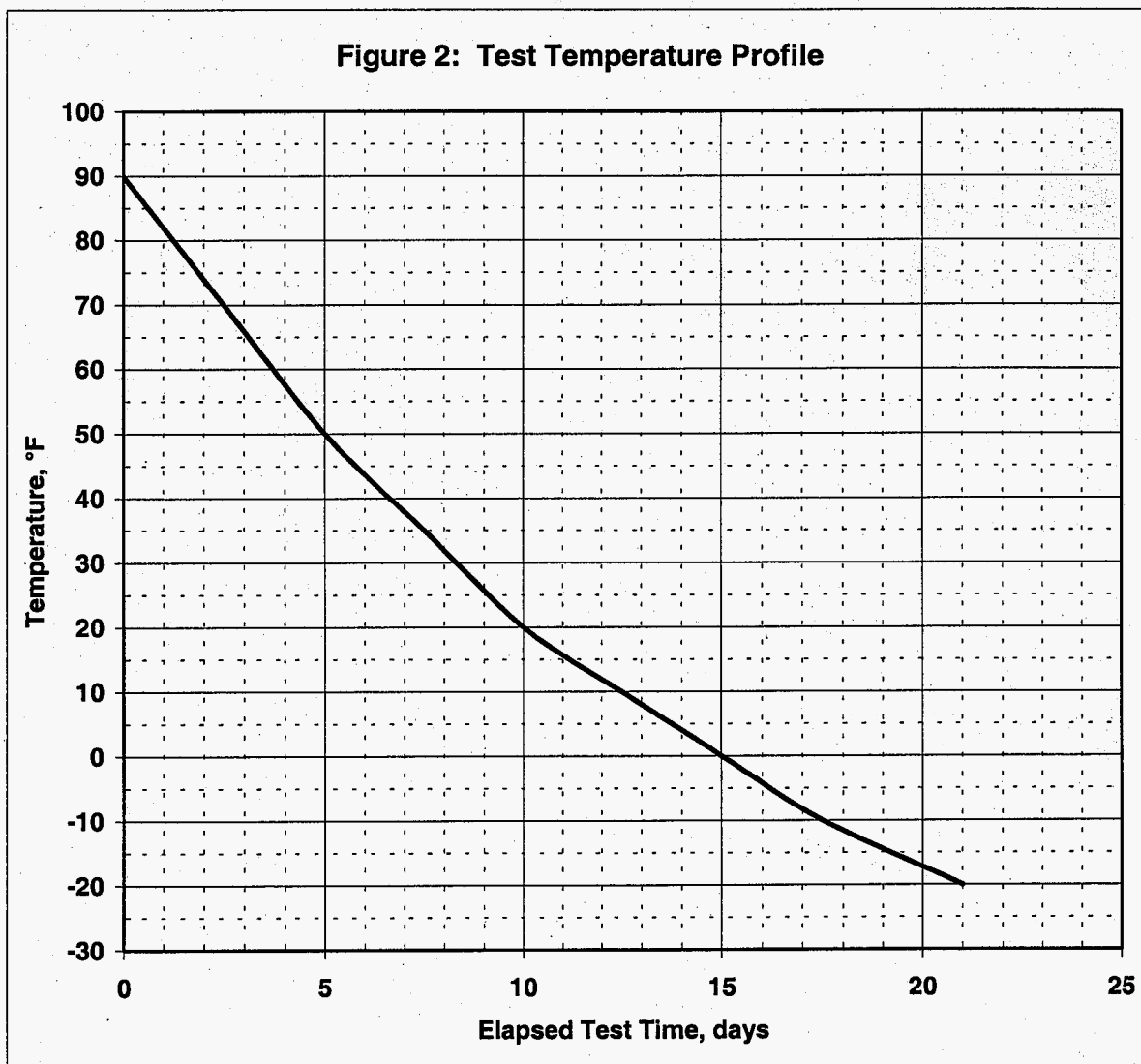
DETERMINATION OF YIELD STRESS VALUE

Figures 3 through 25 on the following pages present torque buildup versus elapsed test time. The yield stress value is determined from the maximum torque response, usually followed by a decline indicating any gel structure present was broken by the applied stress and degrades under continued shearing. In most tests, several minutes of "no torque" response at test initiation are recorded; this response is associated with the time for the 'S'-hook connections to "tighten" before movement, or stress, is applied to the vane shaft. One test sample (GTL-01) was not recorded during testing due to data acquisition failure. Numerical data for the remaining twenty-three tests are presented in Appendix A.

100% Light Hydrocarbon GTL #2

Figure 3: Test Sample GTL-02

Figure 3 presents torque response (dyne-cm) versus elapsed test time at a test temperature of 27°F. The test was performed on the Brookfield RV viscometer. A maximum torque of 6202 dyne-cm was recorded giving a yield stress value of 171 dynes/sqcm.

Figure 4: Test Sample GTL-03

Figure 4 presents torque (dyne-cm) versus elapsed test time at a test temperature of 20°F. The test was performed on the Brookfield RV viscometer. A maximum torque limit of the RV viscometer was reached (7187 dyne-cm); therefore the yield stress value was greater than 199 dynes/sqcm. The HB viscometer was under service warranty calibration and unavailable for use at the time of this test.

Figure 5: Test Sample GTL-04

Figure 5 presents torque (dyne-cm) versus elapsed test time at a test temperature of 0°F. The test was performed on the Brookfield RV viscometer. A maximum torque limit of the RV viscometer was reached (7187 dyne-cm); therefore the yield stress value was greater than 199 dynes/sqcm. A second sample was tested on the HB viscometer.

Figure 6: Test Sample GTL-05

Figure 6 presents torque (dyne-cm) versus elapsed test time at a test temperature of 0°F. The test was performed on the Brookfield HB viscometer. A maximum torque limit of the HB viscometer was reached (57496 dyne-cm); therefore the yield stress value was greater than 1589 dynes/sqcm. The HB viscometer has the highest rated spring torque available for this testing.

Figure 7: Test Sample GTL-06

Figure 7 presents torque response (dyne-cm) versus elapsed test time at a test temperature of 0°F. The test was performed on the Brookfield HB viscometer as a check for repeatability. A maximum torque limit of the HB viscometer was reached (57496 dyne-cm); therefore the yield stress value was greater than 1589 dynes/sqcm. The HB viscometer has the highest rated spring torque available for this testing.

Figure 8: Test Sample GTL-07

Figure 8 presents torque (dyne-cm) versus elapsed test time at a test temperature of -20°F. The test was performed on the Brookfield HB viscometer. A maximum torque limit of the HB viscometer was reached (57496 dyne-cm); therefore the yield stress value was greater than 1589 dynes/sqcm. The HB viscometer has the highest rated spring torque available for this testing.

Figure 9: Test Sample GTL-08

Figure 9 presents torque (dyne-cm) versus elapsed test time at a test temperature of -20°F. The test was performed on the Brookfield HB viscometer as a check for repeatability. A maximum torque limit of the HB viscometer was reached (57496 dyne-cm); therefore the yield stress value was greater than 1589 dynes/sqcm. The HB viscometer has the highest rated spring torque available for this testing.

3:1 Ratio Blend   (25% GTL #2 + 75% TAPS Crude Oil at PS-1)

Figure 10: Test Sample 3:1 - 01

Figure 10 presents torque (dyne-cm) versus elapsed test time at a test temperature of 20°F. The test was performed on the Brookfield RV viscometer. A maximum torque of 108 dyne-cm was recorded giving a yield stress value of 2.98 dynes/sqcm.

Figure 11: Test Sample 3:1 - 02

Figure 11 presents torque (dyne-cm) versus elapsed test time at a test temperature of 20°F. The test was performed on the Brookfield RV viscometer as a check for repeatability. A maximum torque of 93.4 dyne-cm was recorded giving a yield stress value of 2.58 dynes/sqcm.

Figure 12: Test Sample 3:1 - 03

Figure 12 presents torque (dyne-cm) versus elapsed test time at a test temperature of 20°F. The test was performed on the Brookfield RV viscometer as a check for repeatability. A maximum torque of 115 dyne-cm was recorded giving a yield stress value of 3.18 dynes/sqcm.

Figure 13: Test Sample 3:1 - 04

Figure 13 presents torque (dyne-cm) versus elapsed test time at a test temperature of 0°F. The test was performed on the Brookfield LV viscometer. A maximum torque limit of the LV viscometer was reached (674 dyne-cm); therefore the yield stress value was greater than 18.6 dynes/sqcm. A second sample was tested on the HB viscometer.

Figure 14: Test Sample 3:1 - 05

Figure 14 presents torque (dyne-cm) versus elapsed test time at a test temperature of 0°F. The test was performed on the Brookfield HB viscometer. A maximum torque of 23,746 dyne-cm was recorded giving a yield stress value of 656 dynes/sqcm.

Figure 15: Test Sample 3:1 - 06

Figure 14 presents torque (dyne-cm) versus elapsed test time at a test temperature of 0°F. The test was performed on the Brookfield HB viscometer as a check for repeatability. A maximum torque of 26,506 dyne-cm was recorded giving a yield stress value of 732 dynes/sqcm.

Figure 16: Test Sample 3:1 - 07

Figure 16 presents torque (dyne-cm) versus elapsed test time at a test temperature of -20°F. The test was performed on the Brookfield HB viscometer. A maximum torque limit of the HB viscometer was reached (57496 dyne-cm); therefore the yield stress value was greater than 1589 dynes/sqcm. The HB viscometer has the highest rated spring torque available for this testing.

Figure 17: Test Sample 3:1 - 08

Figure 17 presents torque (dyne-cm) versus elapsed test time at a test temperature of -20°F. The test was performed on the Brookfield HB viscometer as a check for repeatability. A maximum torque limit of the HB viscometer was reached (57496 dyne-cm); therefore the yield stress value was greater than 1589 dynes/sqcm. The HB viscometer has the highest rated spring torque available for this testing.

4:1 Ratio Blend   (20% GTL #2 + 80% TAPS Crude Oil at PS-1)

Figure 18: Test Sample 4:1 - 01

Figure 18 presents torque (dyne-cm) versus elapsed test time at a test temperature of 20°F. The test was performed on the Brookfield RV viscometer. A maximum torque of 28.7 dyne-cm was recorded giving a yield stress value of 0.79 dynes/sqcm.

Figure 19: Test Sample 4:1 - 02

Figure 19 presents torque (dyne-cm) versus elapsed test time at a test temperature of 20°F. The test was performed on the Brookfield RV viscometer as a check for repeatability. A maximum torque of 64.7 dyne-cm was recorded giving a yield stress value of 1.79 dynes/sqcm. The variance between 4:1-01 and 4:1-02 was greater than 15%, therefore a third sample was tested.

Figure 20: Test Sample 4:1 - 03

Figure 20 presents torque (dyne-cm) versus elapsed test time at a test temperature of 20°F. The test was performed on the Brookfield RV viscometer as a check for repeatability. A maximum torque of 28.7 dyne-cm was recorded giving a yield stress value of 0.79 dynes/sqcm.

Figure 21: Test Sample 4:1 – 04

Figure 21 presents torque (dyne-cm) versus elapsed test time at a test temperature of 0°F. The test was performed on the Brookfield LV viscometer. A maximum torque limit of the LV viscometer was

reached (674 dyne-cm); therefore the yield stress value was greater than 18.6 dynes/sqcm. A second sample was tested on the RV viscometer.

Figure 22: Test Sample 4:1 – 05

Figure 22 presents torque (dyne-cm) versus elapsed test time at a test temperature of 0°F. The test was performed on the Brookfield RV viscometer. A maximum torque limit of the RV viscometer was reached (7187 dyne-cm); therefore the yield stress value was greater than 199 dynes/sqcm. A third sample was tested on the HB viscometer.

Figure 23: Test Sample 4:1 – 06

Figure 23 presents torque (dyne-cm) versus elapsed test time at a test temperature of 0°F. The test was performed on the Brookfield HB viscometer. A maximum torque of 12592 dyne-cm was recorded giving a yield stress value of 348 dynes/sqcm.

Figure 24: Test Sample 4:1 – 07

Figure 24 presents torque (dyne-cm) versus elapsed test time at a test temperature of 0°F. The test was performed on the Brookfield HB viscometer as a check for repeatability. A maximum torque of 15581 dyne-cm was recorded giving a yield stress value of 431 dynes/sqcm.

Figure 25: Test Sample 4:1 – 08

Figure 25 presents torque (dyne-cm) versus elapsed test time at a test temperature of -20°F. The test was performed on the Brookfield HB viscometer. A maximum torque limit of the HB viscometer was reached (57496 dyne-cm); therefore the yield stress value was greater than 1589 dynes/sqcm. The HB viscometer has the highest rated spring torque available for this testing.
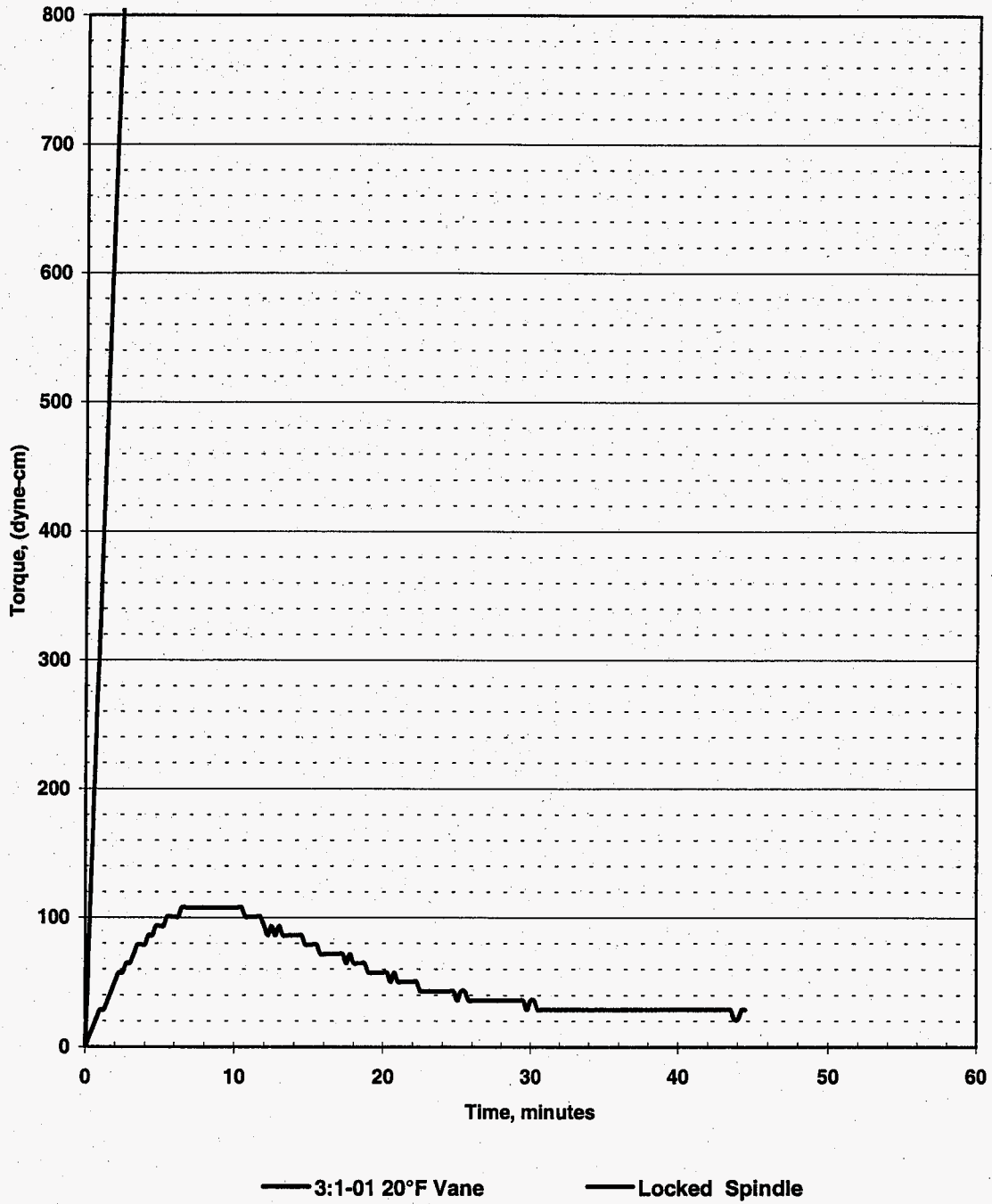
Figure 3: Sample GTL-02 at 27°F   RV Viscometer

Yield Point by Vane Viscometry
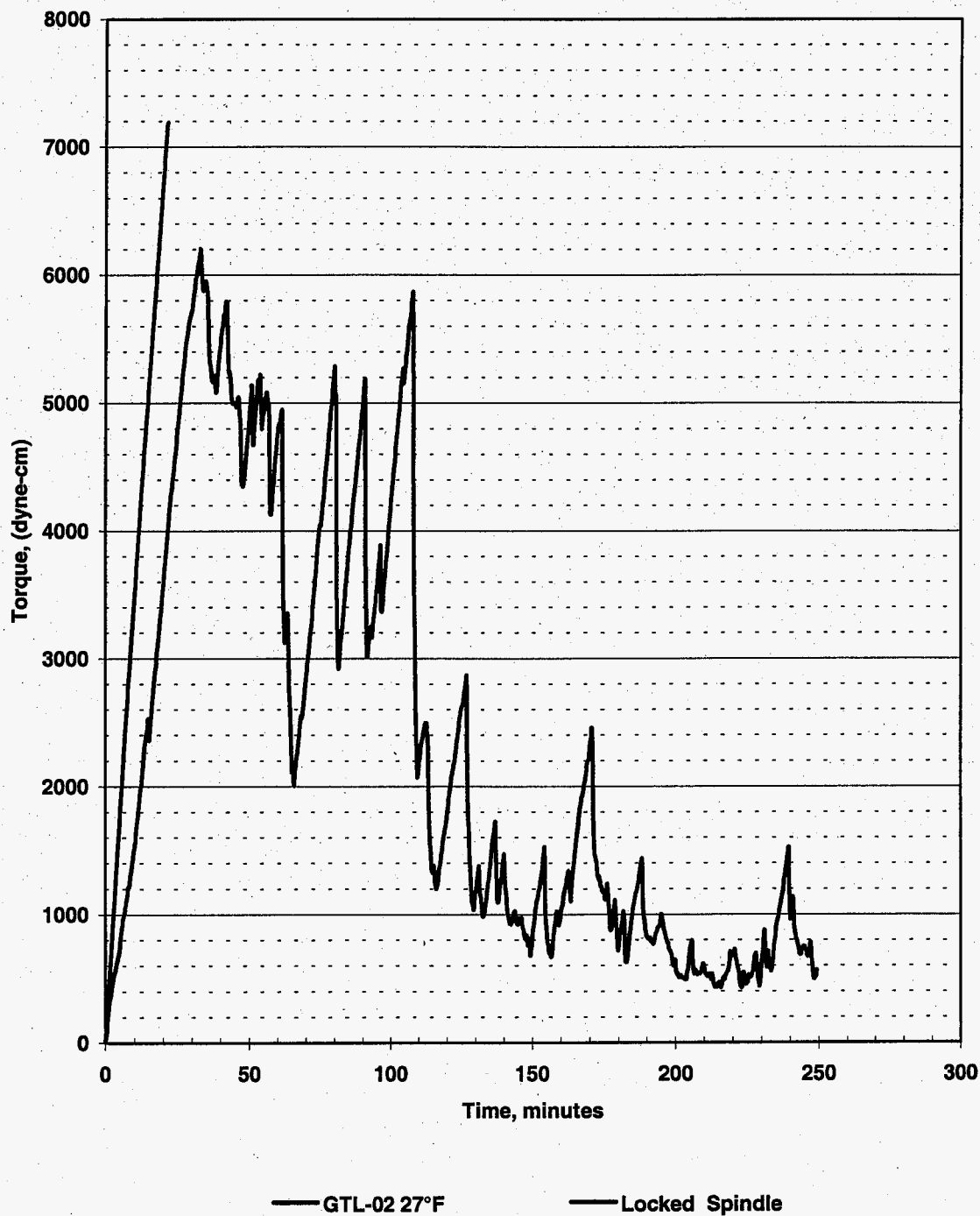
Figure 4: Sample GTL-03 at 20°F   RV Viscometer
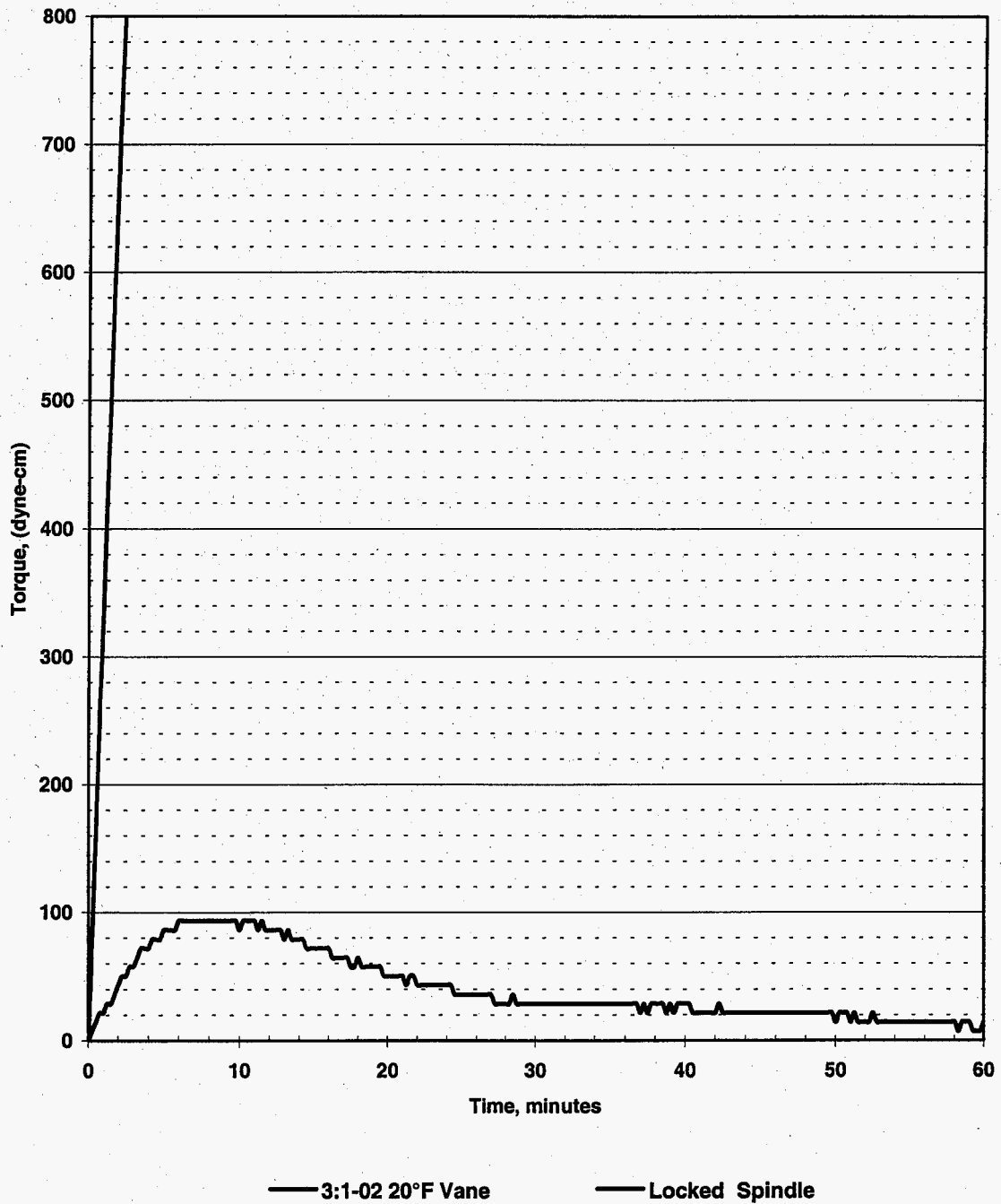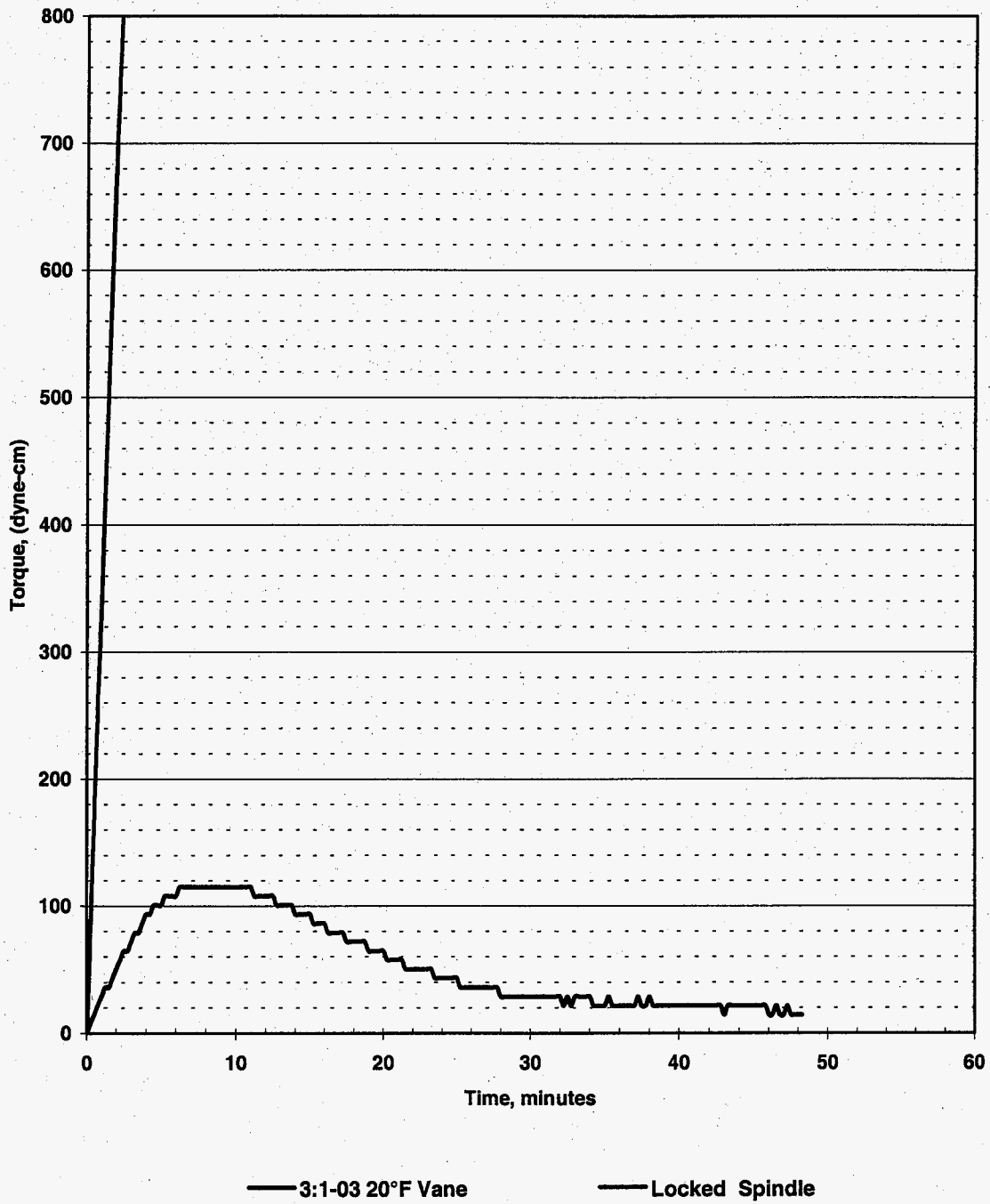
Yield Point by Vane Viscometry

Legend: GTL-03 20°F   Locked Spindle
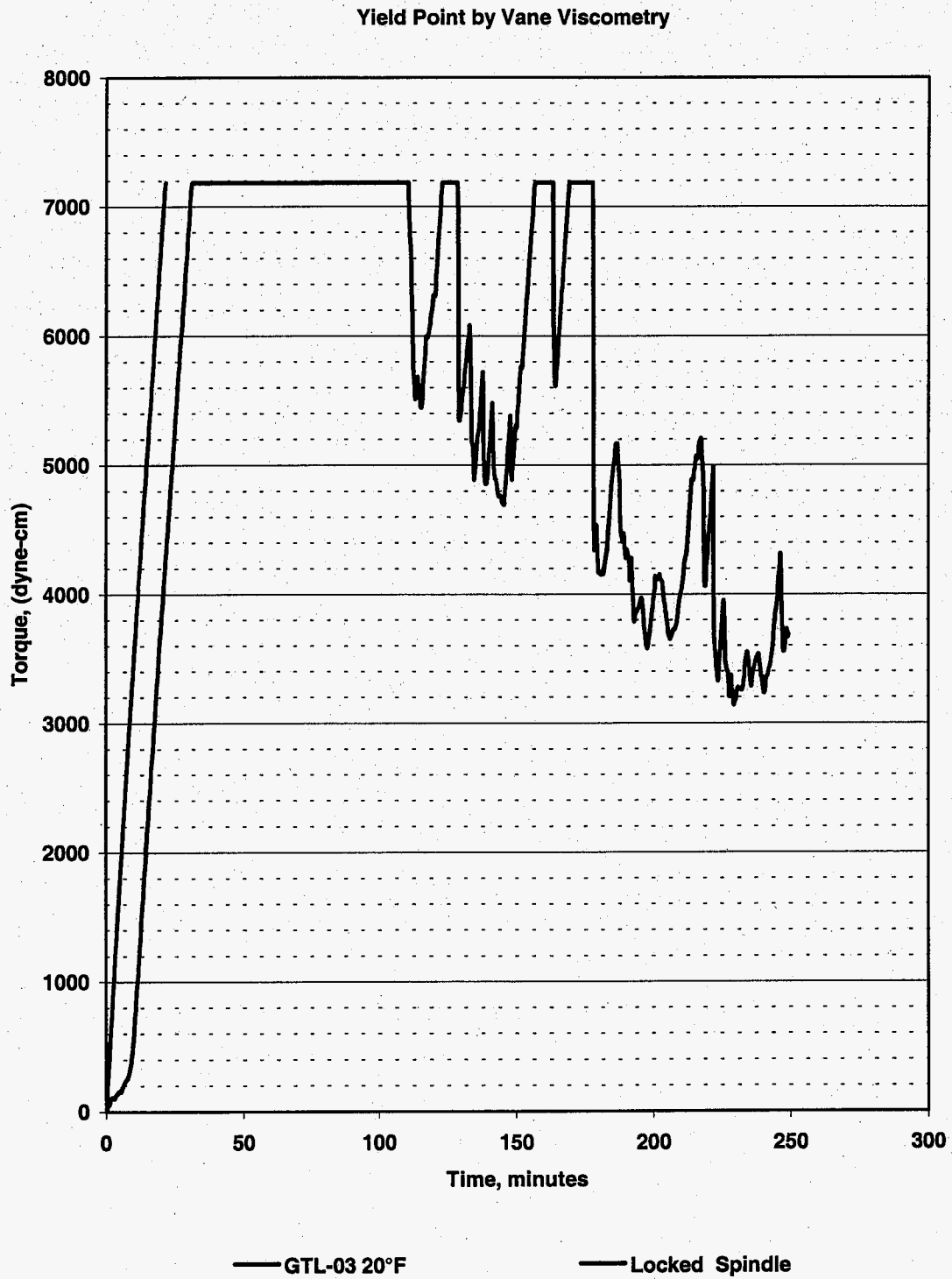
Figure 5: Sample GTL-04 at 0°F   RV Viscometer

Yield Point by Vane Viscometry

GTL-04 0°F   Locked Spindle
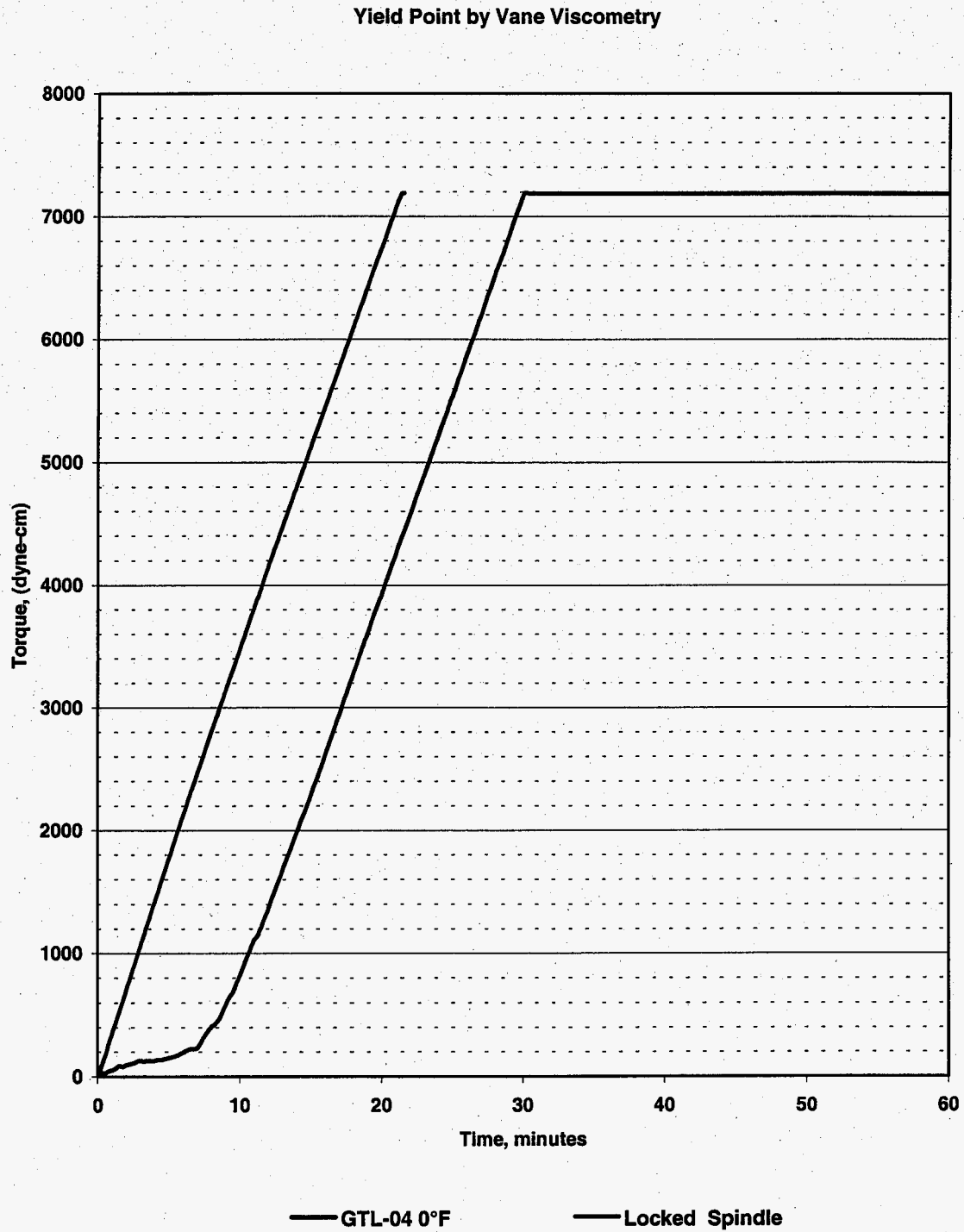
Figure 6: Sample GTL-05 at 0°F   HB Viscometer

Yield Point by Vane Viscometry
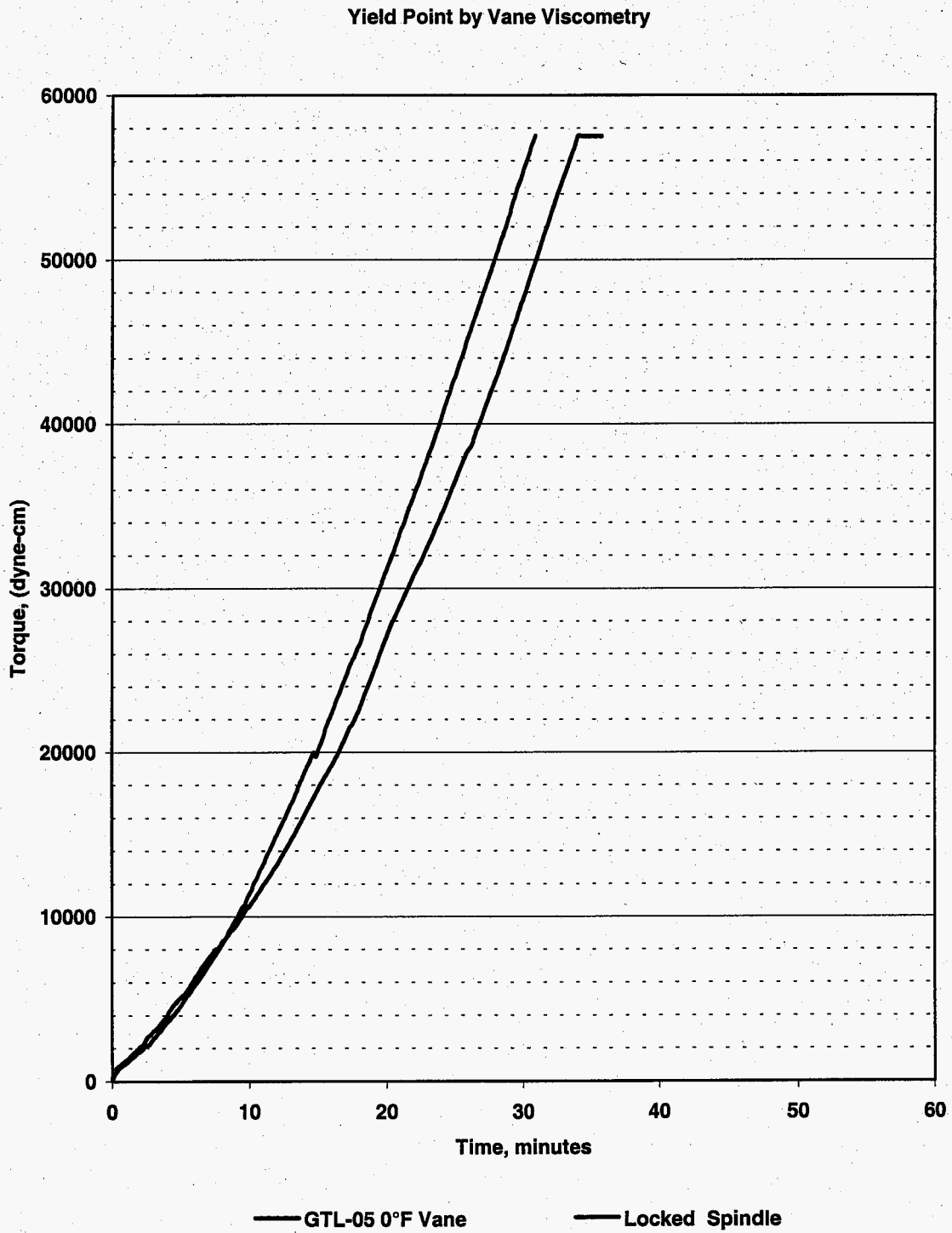
Figure 7: Sample GTL-06 at 0°F   HB Viscometer

Yield Point by Vane Viscometry
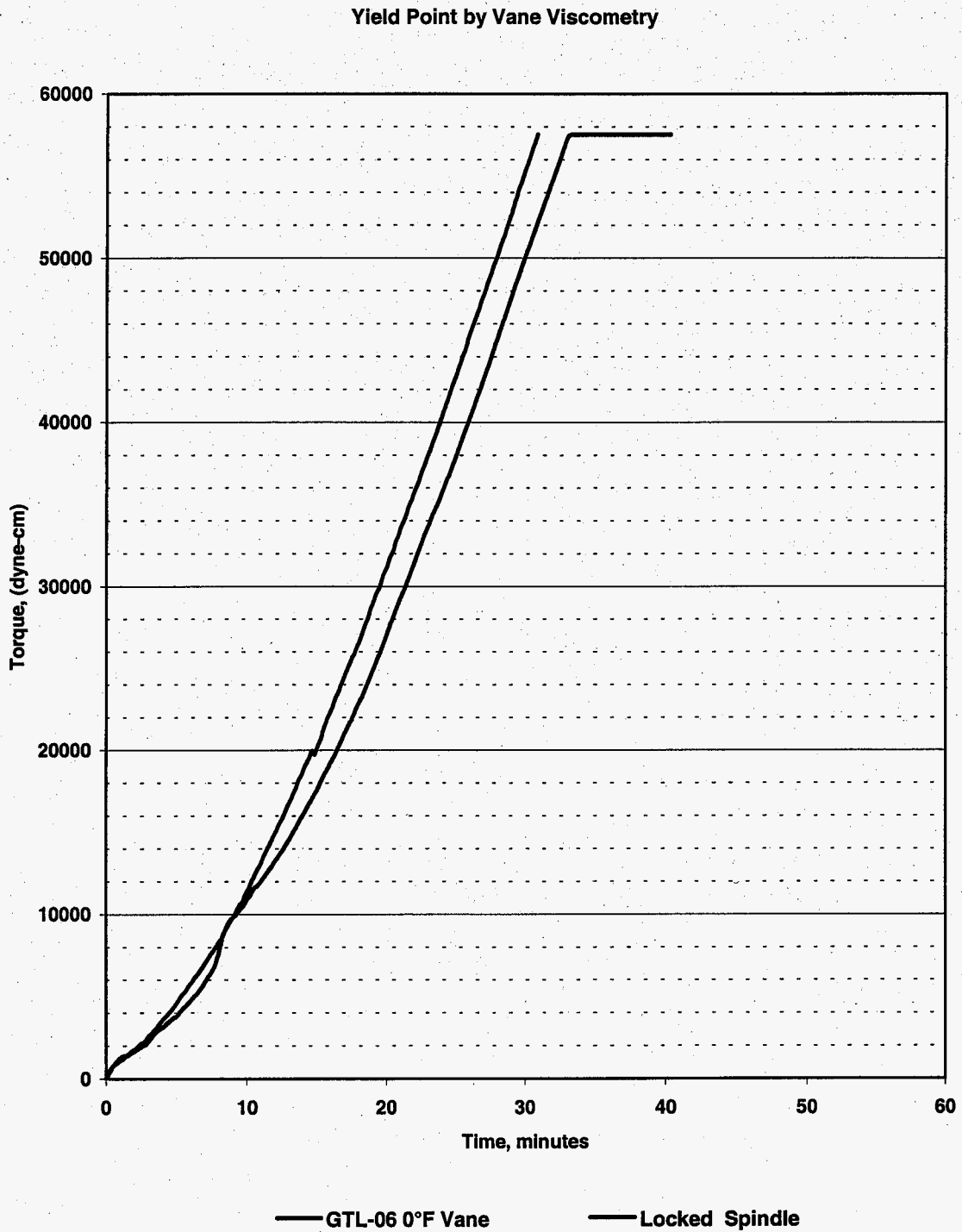
Figure 8: Sample GTL-07 at -20°F   HB Viscometer

Yield Point by Vane Viscometry

GTL-07 -20°F Vane   Locked Spindle
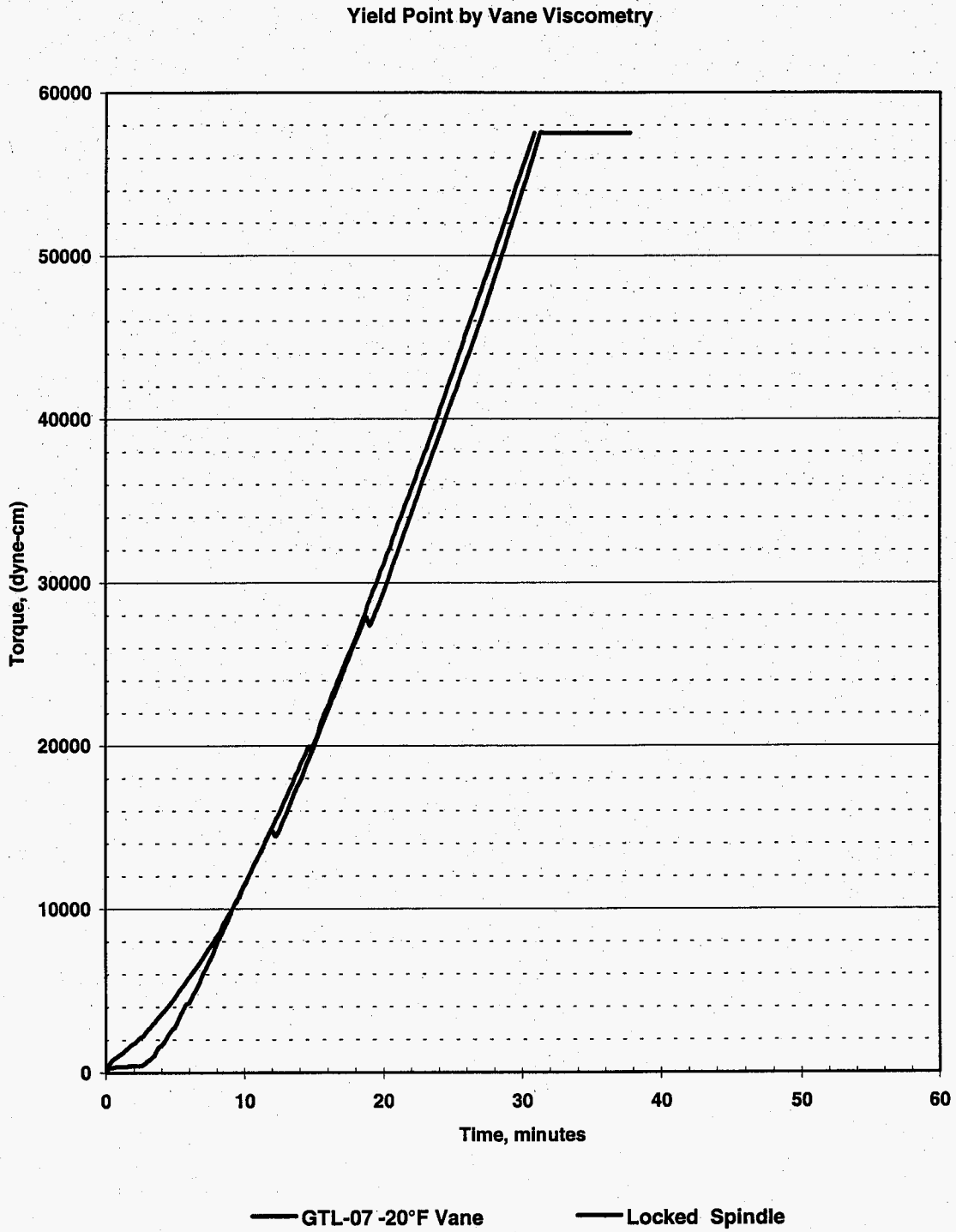
Figure 9: Sample GTL-08 at -20°F   HB Viscometer

Yield Point by Vane Viscometry

——GTL-08 -20°F Vane   ——Locked Spindle
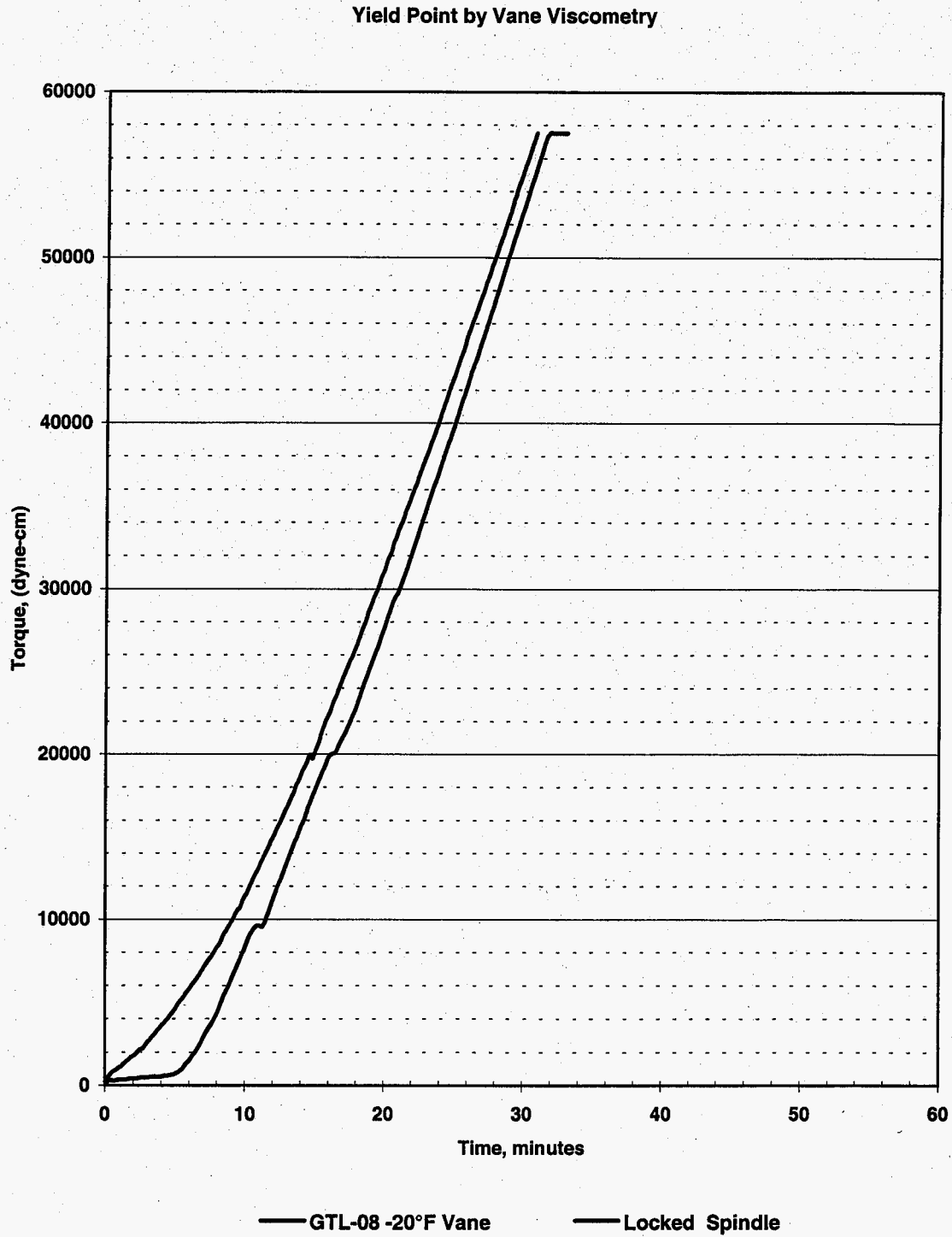
Figure 10: Sample 3:1 - 01 at 20°F   RV Viscometer

Yield Point by Vane Viscometry

3:1-01 20°F Vane   Locked Spindle

Figure 11: Sample 3:1 - 02 at 20°F   RV Viscometer

Yield Point by Vane Viscometry

Legend: ——— 3:1-02 20°F Vane   ——— Locked Spindle

X-axis: Time, minutes
Y-axis: Torque, (dyne-cm)

Figure 12: Sample 3:1 - 03 at 20°F   RV Viscometer

Yield Point by Vane Viscometry

3:1-03 20°F Vane   Locked Spindle

Figure 13: Sample 3:1 - 04 at 0°F   LV Viscometer

Yield Point by Vane Viscometry

Legend: ———3:1-04 0°F Vane   ———Locked Spindle
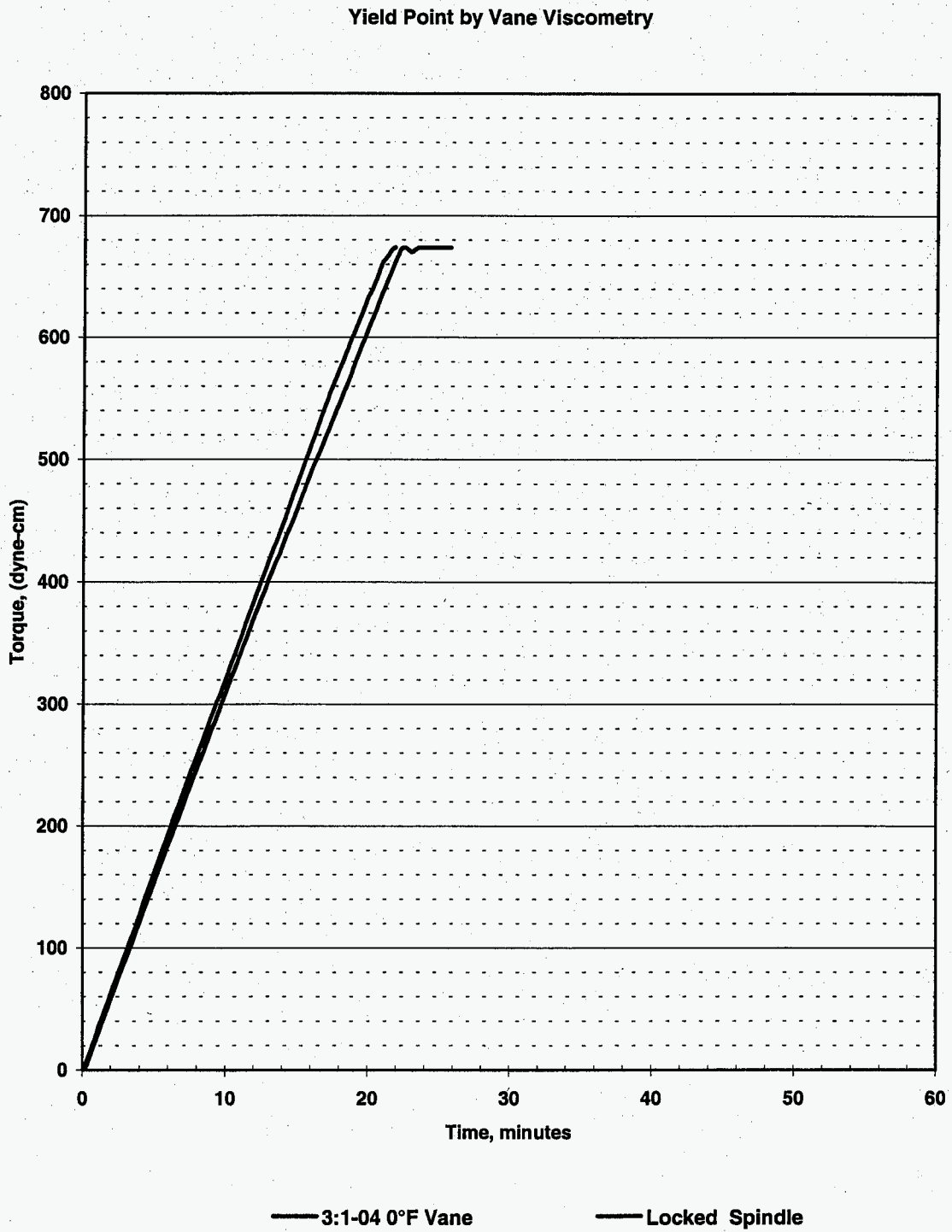
Figure 14: Sample 3:1 - 05 at 0°F   HB Viscometer

Yield Point by Vane Viscometry
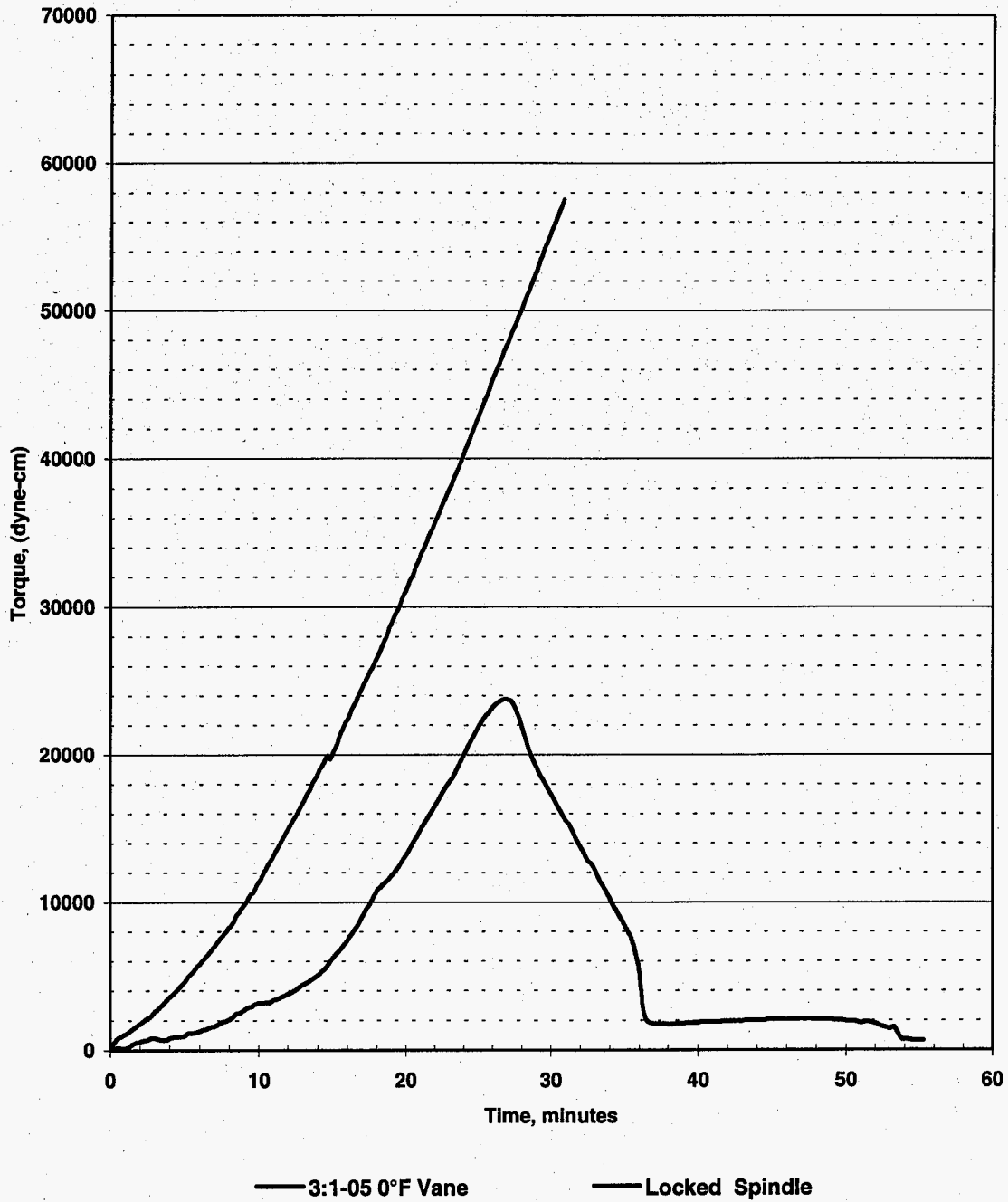
Figure 15: Sample 3:1 - 06 at 0°F   HB Viscometer

Yield Point by Vane Viscometry
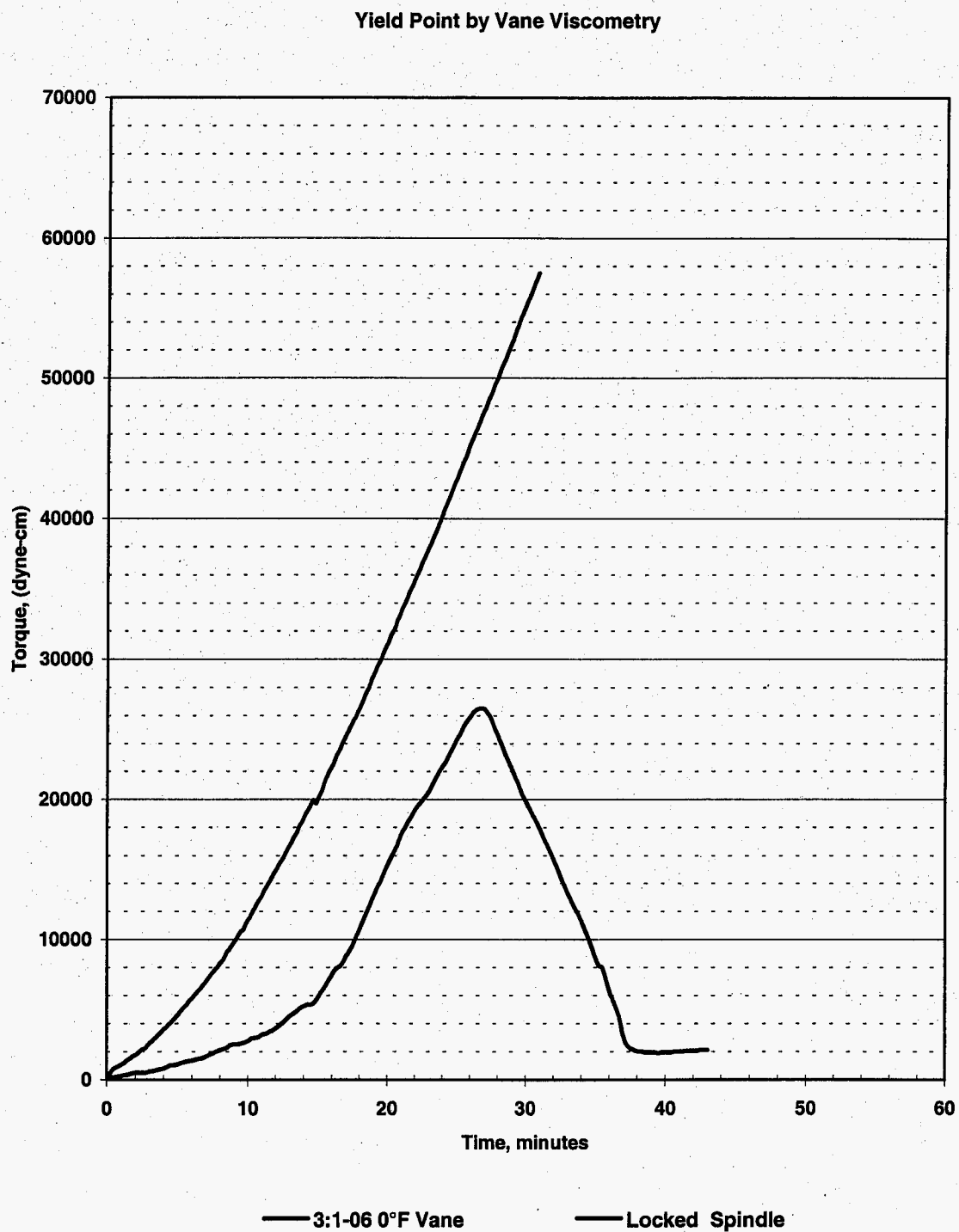
Figure 16: Sample 3:1 - 07 at -20°F   HB Viscometer

Yield Point by Vane Viscometry

Legend: 3:1-07 -20°F Vane   Locked Spindle

X-axis: Time, minutes
Y-axis: Torque, (dyne-cm)
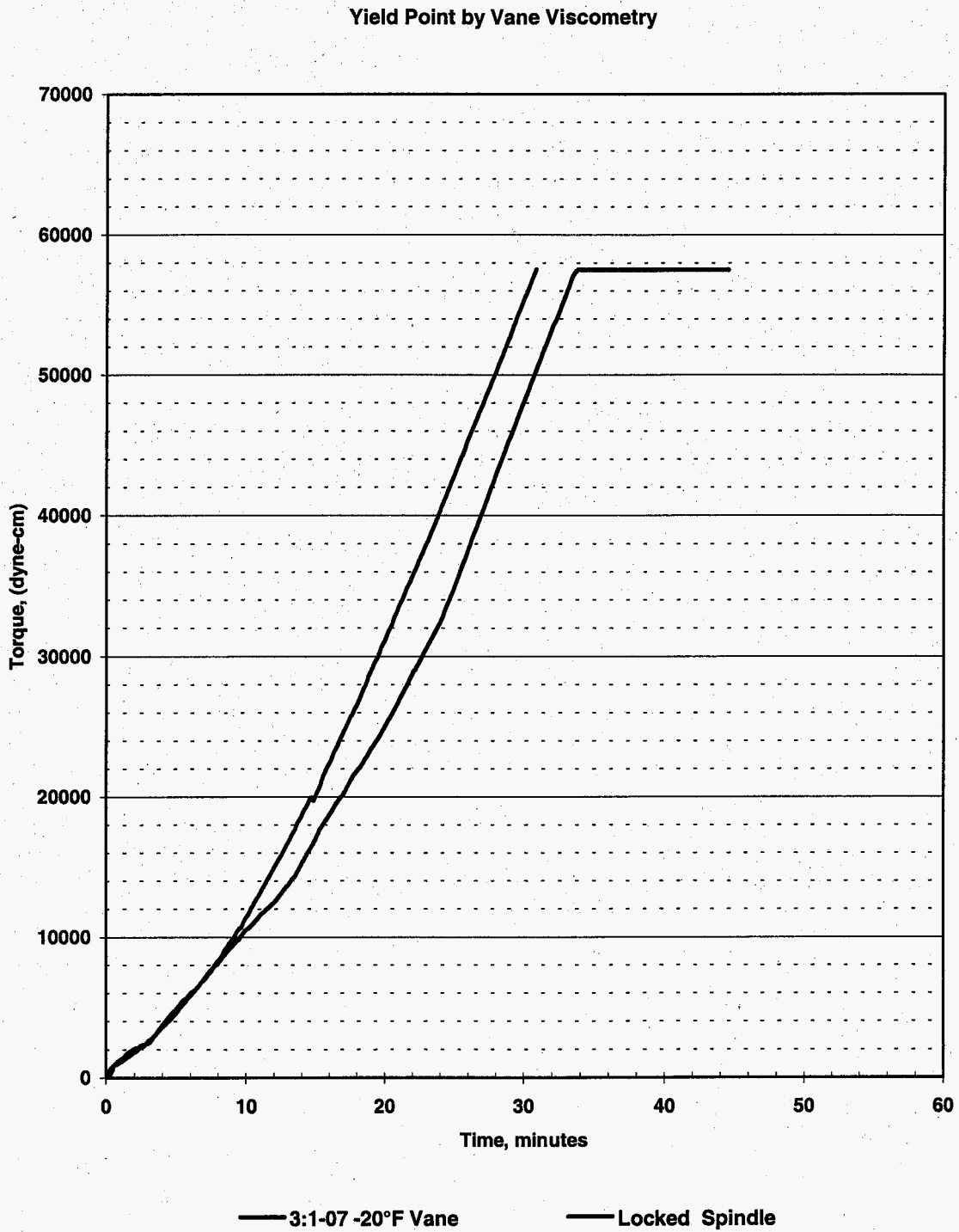
Figure 17: Sample 3:1 - 08 at -20°F   HB Viscometer

Yield Point by Vane Viscometry

Legend: 3:1-08 -20°F Vane   Locked Spindle

Axes: Torque, (dyne-cm) vs Time, minutes
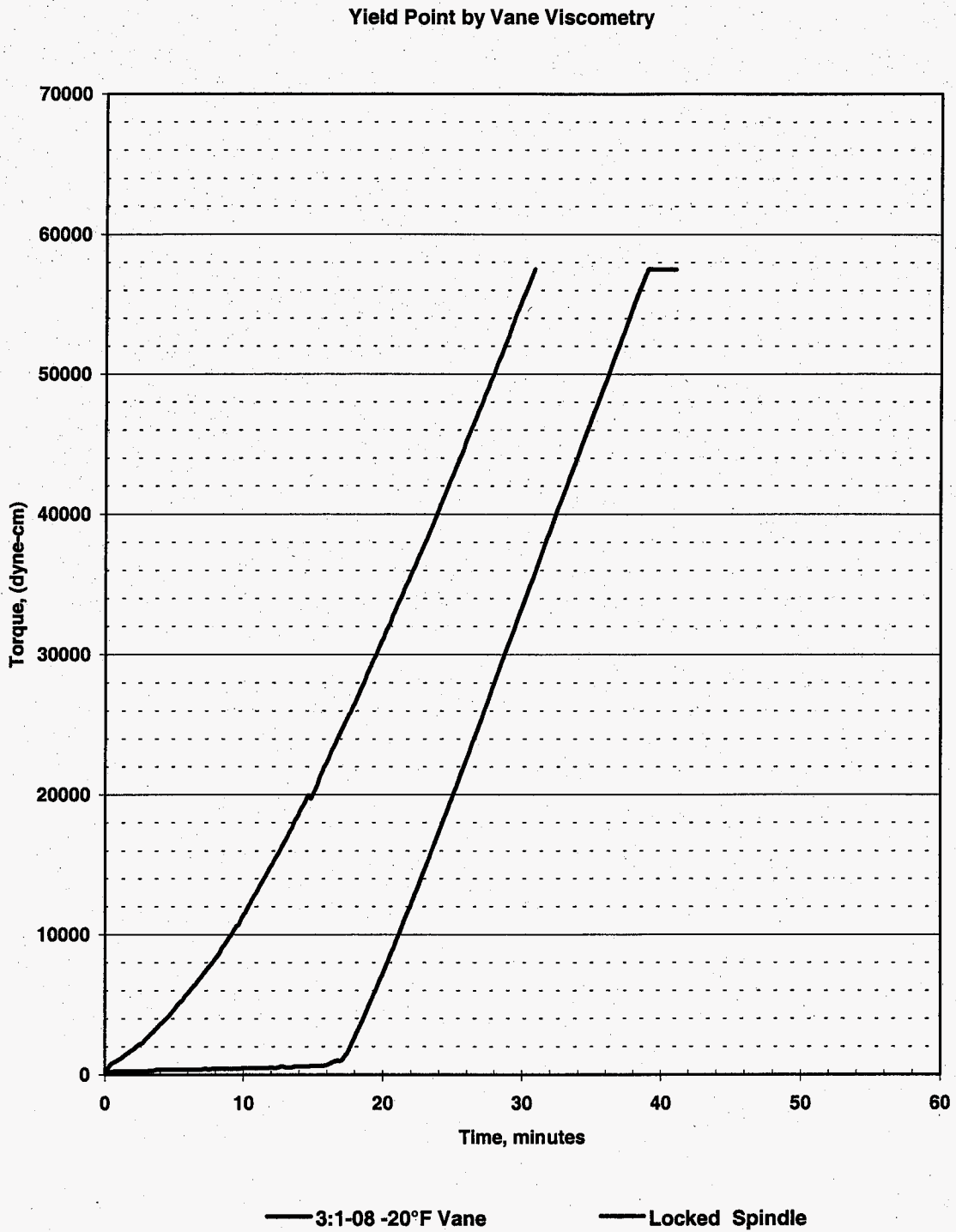
Figure 18: Sample 4:1 - 01 at 20°F   RV Viscometer

Yield Point by Vane Viscometry
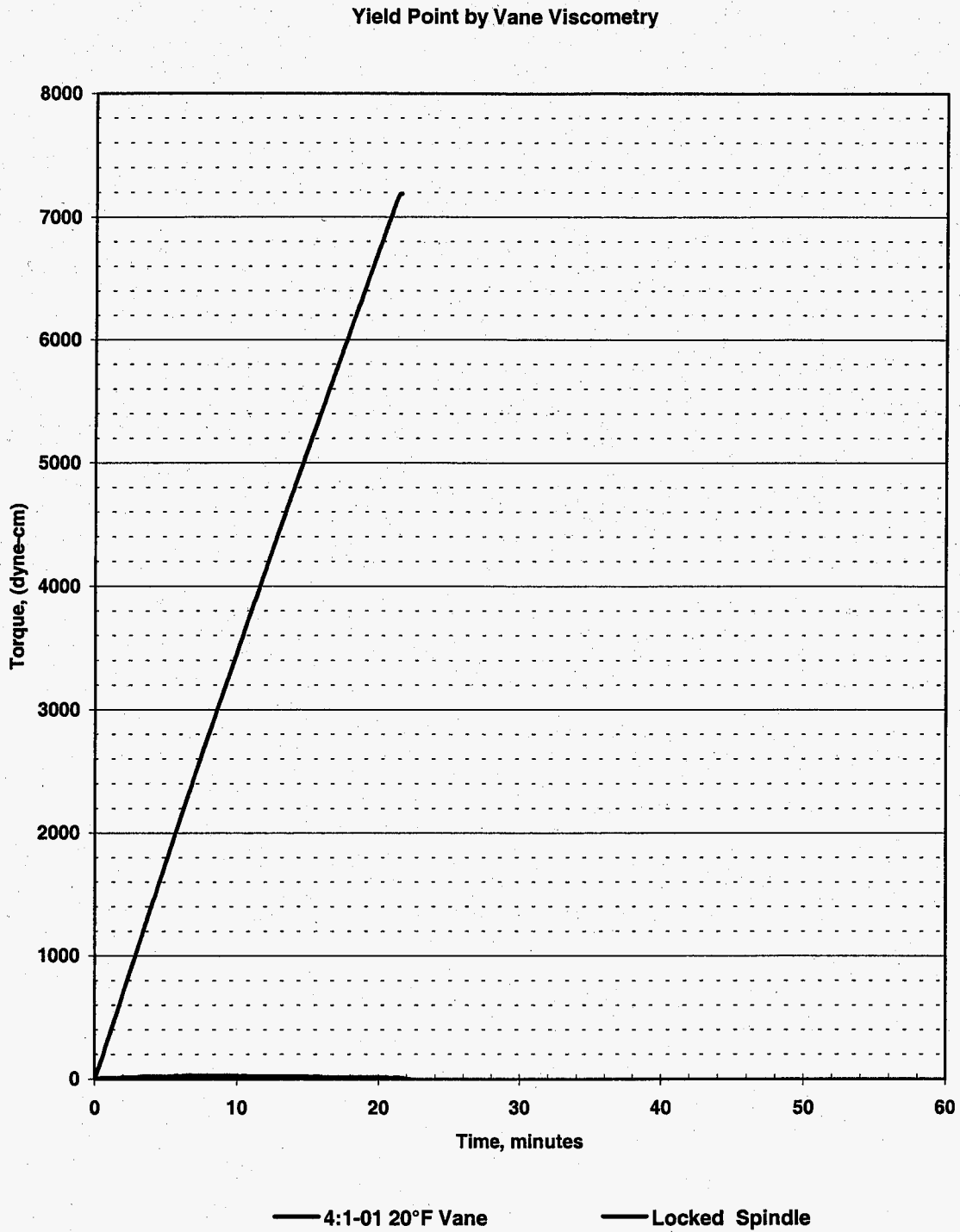
Figure 19: Sample 4:1 - 02 at 20°F   RV Viscometer

Yield Point by Vane Viscometry

4:1-02 20°F Vane   Locked Spindle
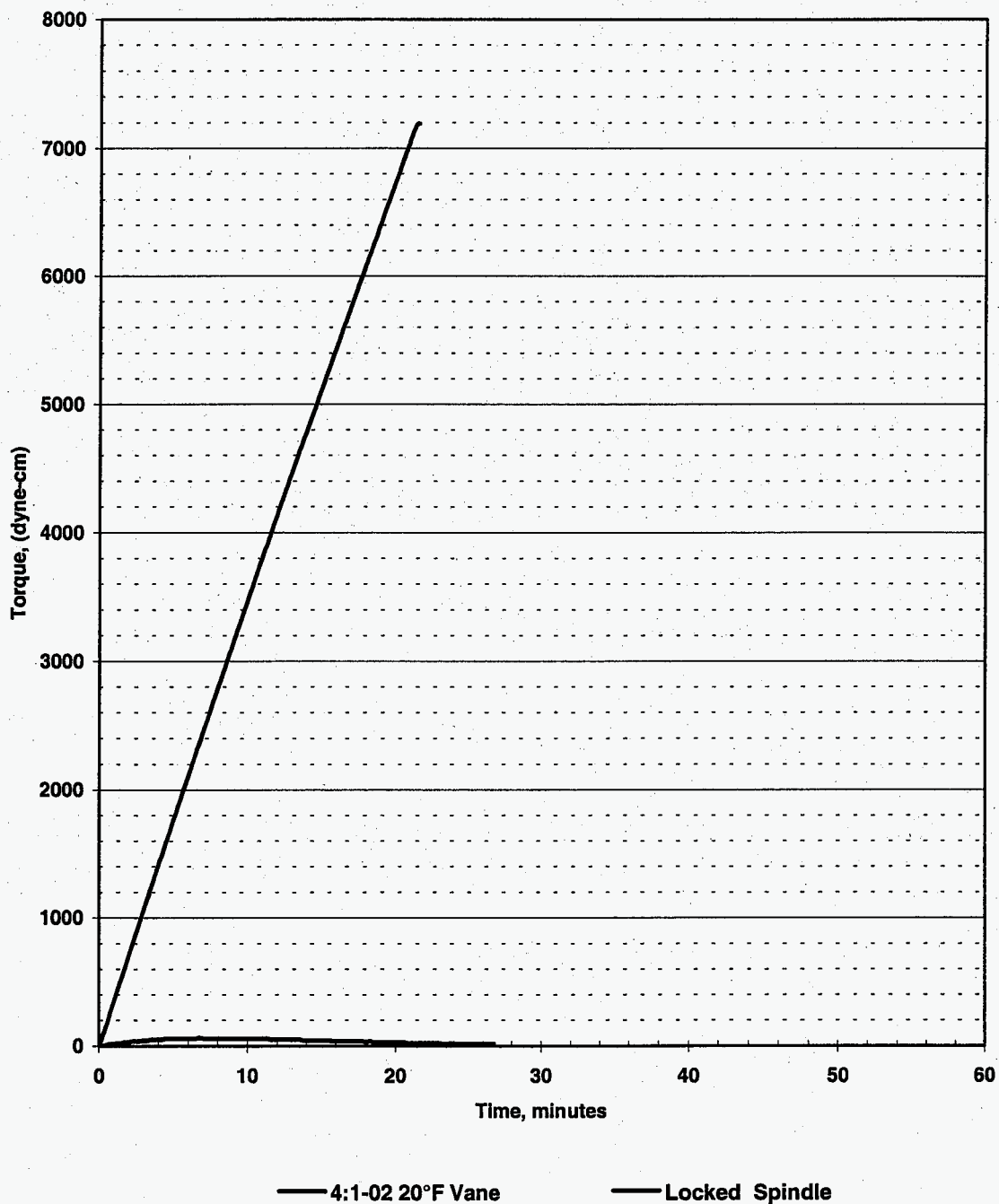
Figure 20: Sample 4:1 - 03 at 20°F   RV Viscometer

Yield Point by Vane Viscometry
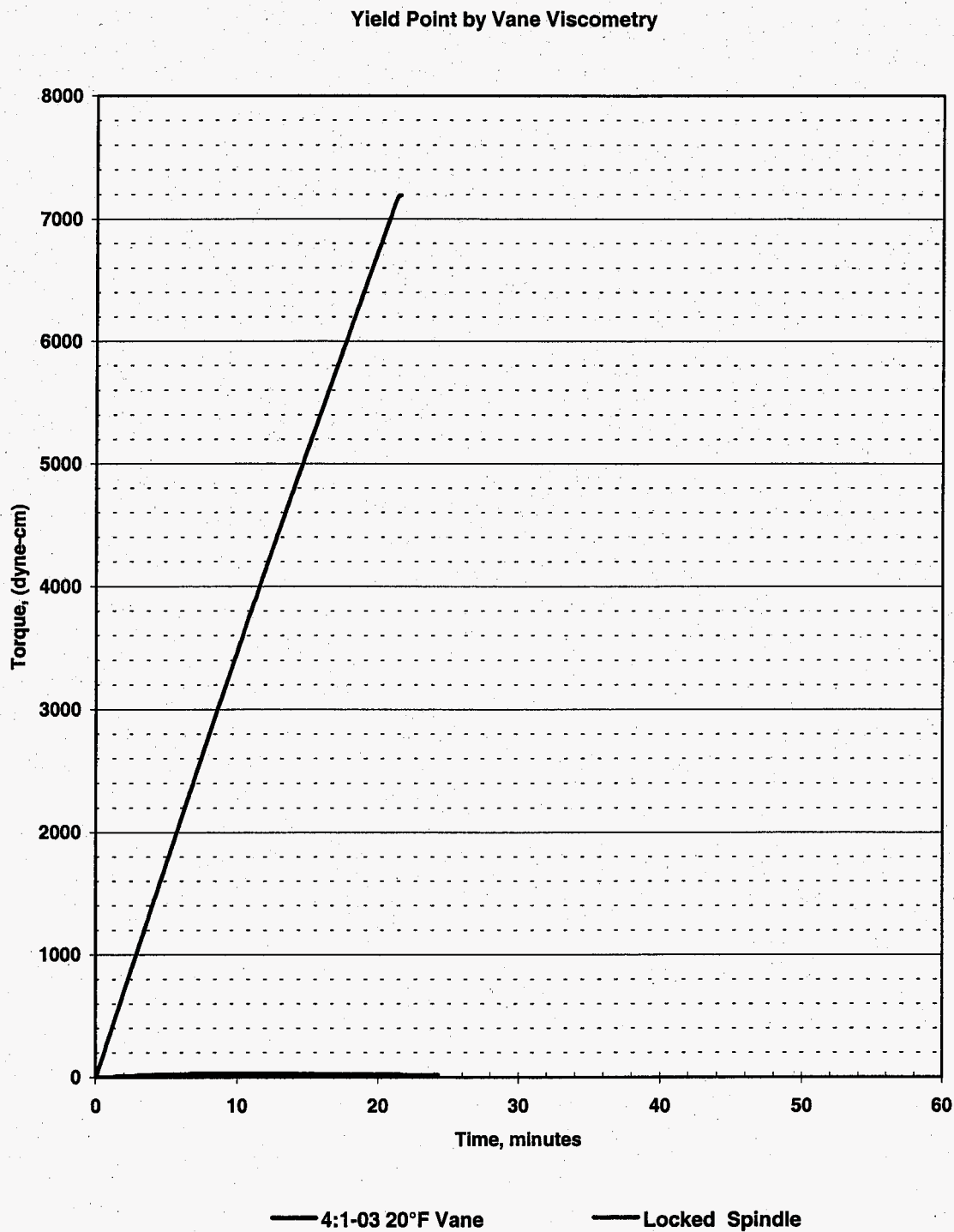
Figure 21: Sample 4:1 - 04 at 0°F   LV Viscometer

Yield Point by Vane Viscometry
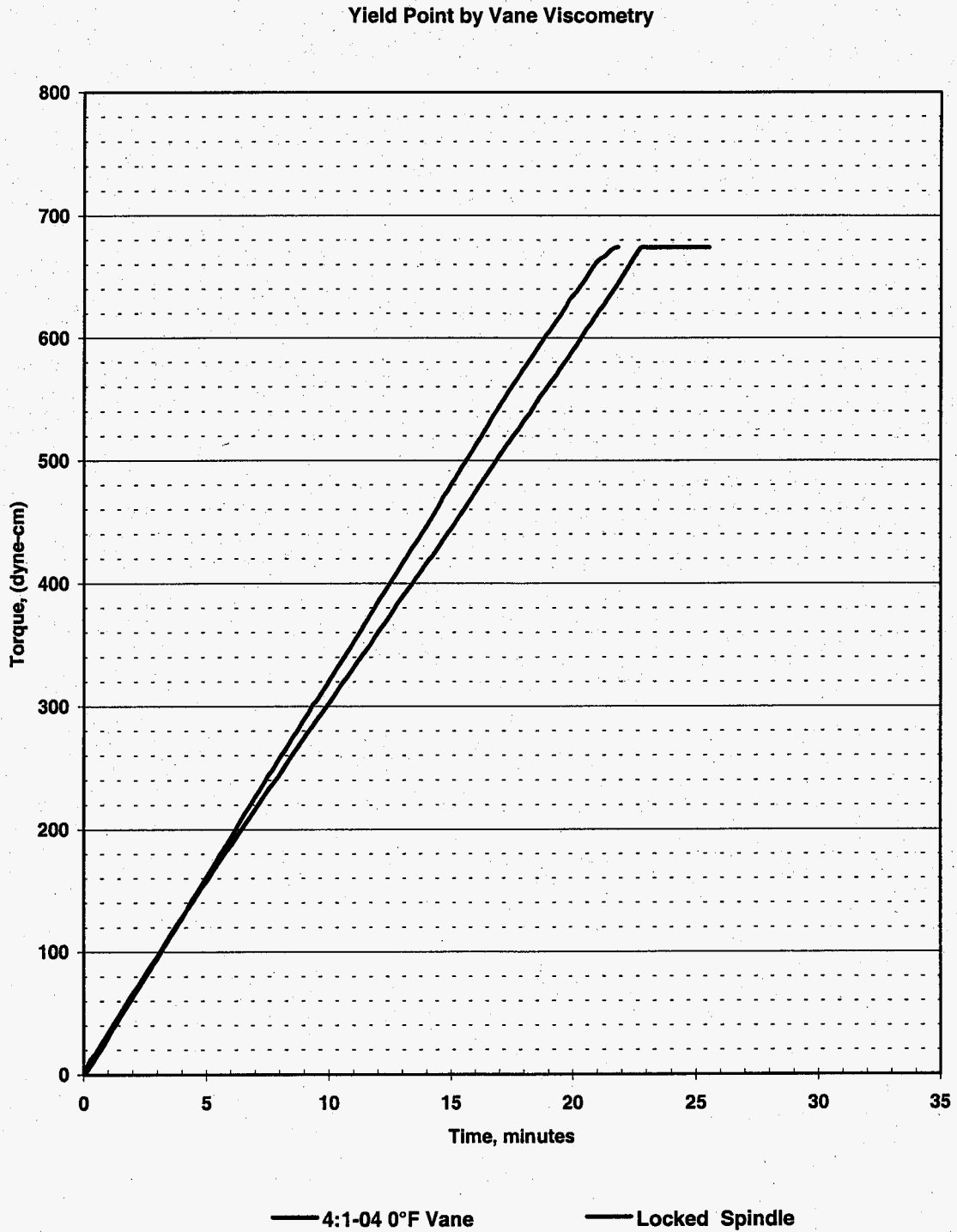
Figure 22: Sample 4:1 - 05 at 0°F   RV Viscometer

Yield Point by Vane Viscometry
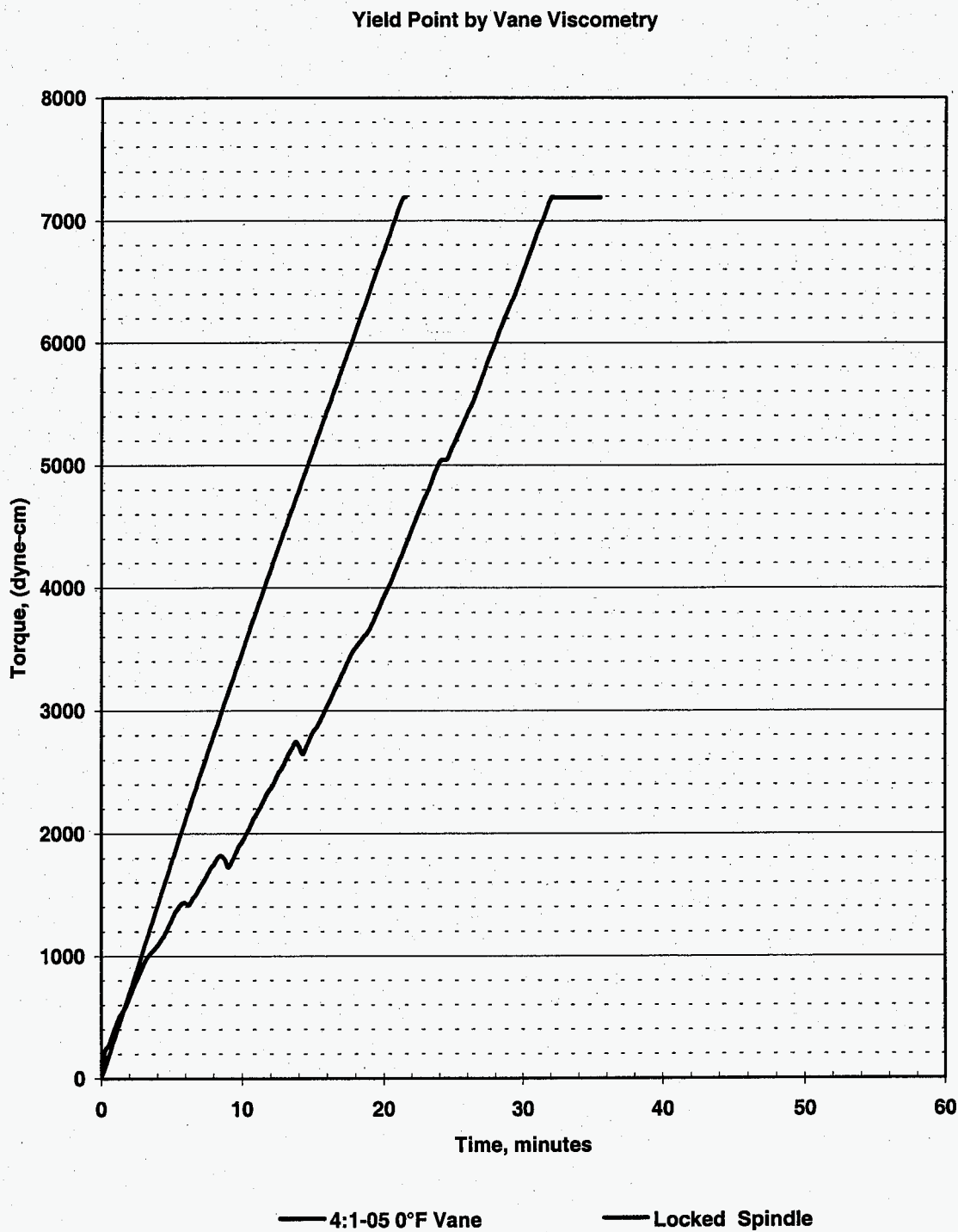
Figure 23: Sample 4:1 - 06 at 0°F   HB Viscometer

Yield Point by Vane Viscometry
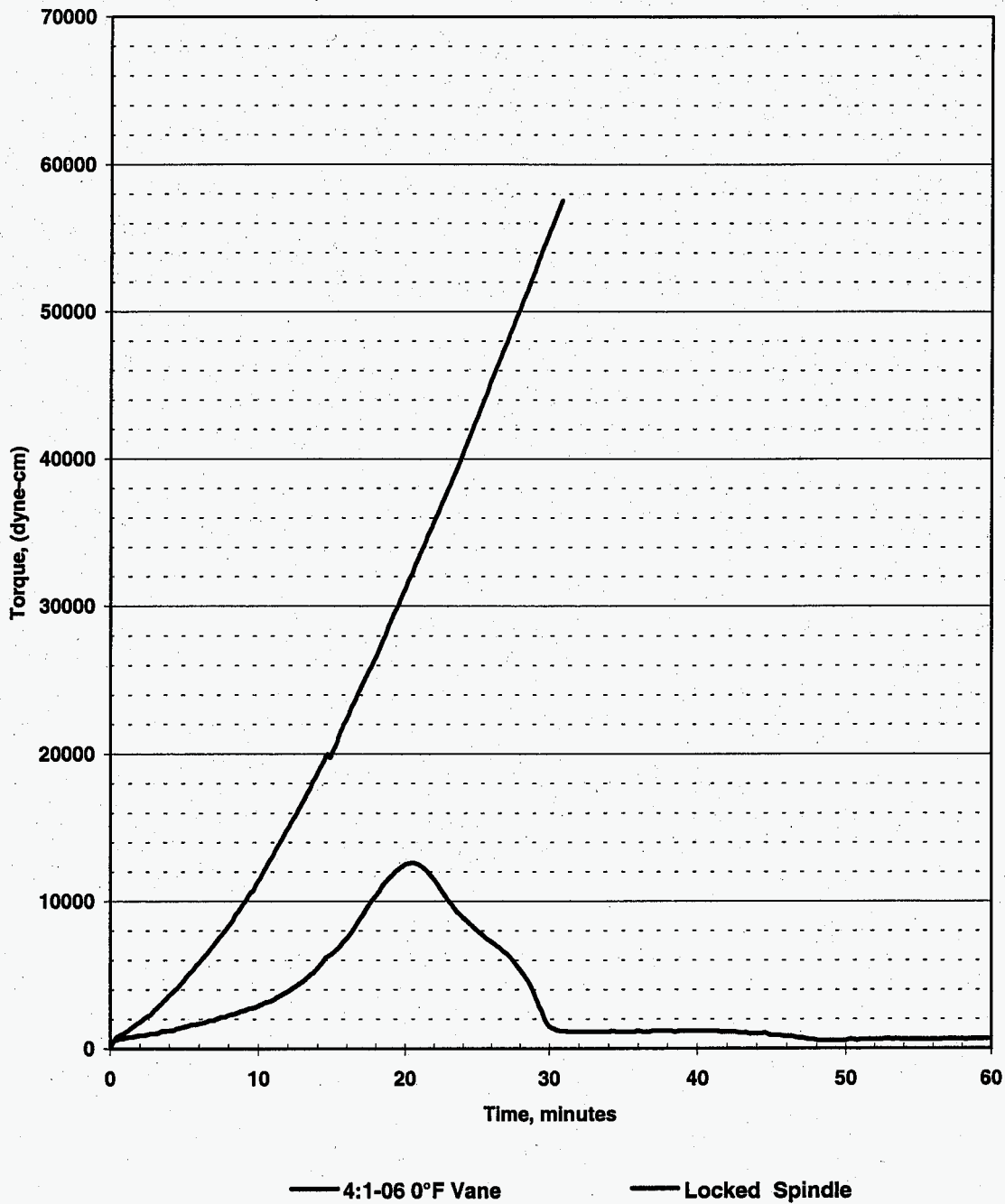
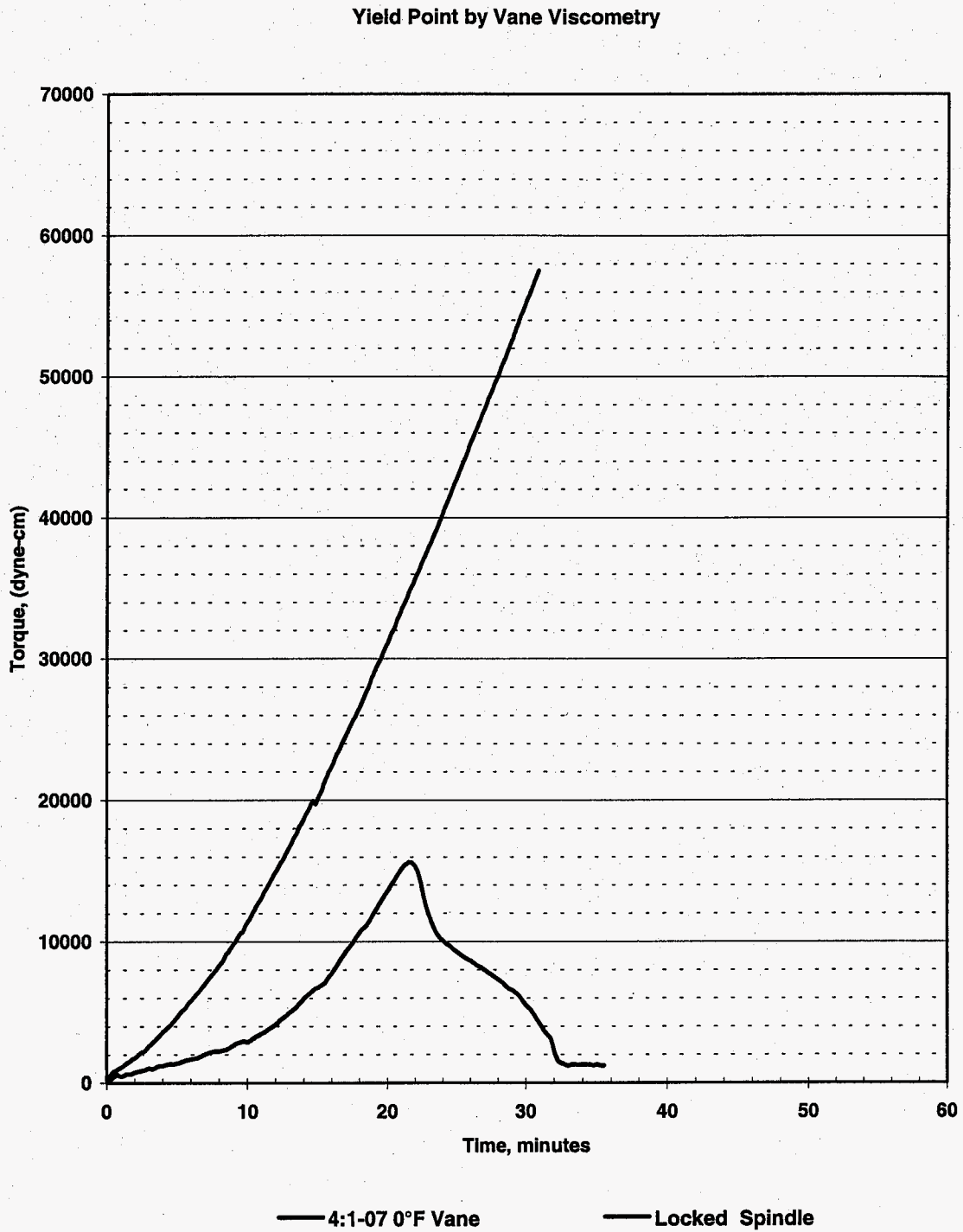**Figure 24: Sample 4:1 - 07 at 0°F   HB Viscometer**

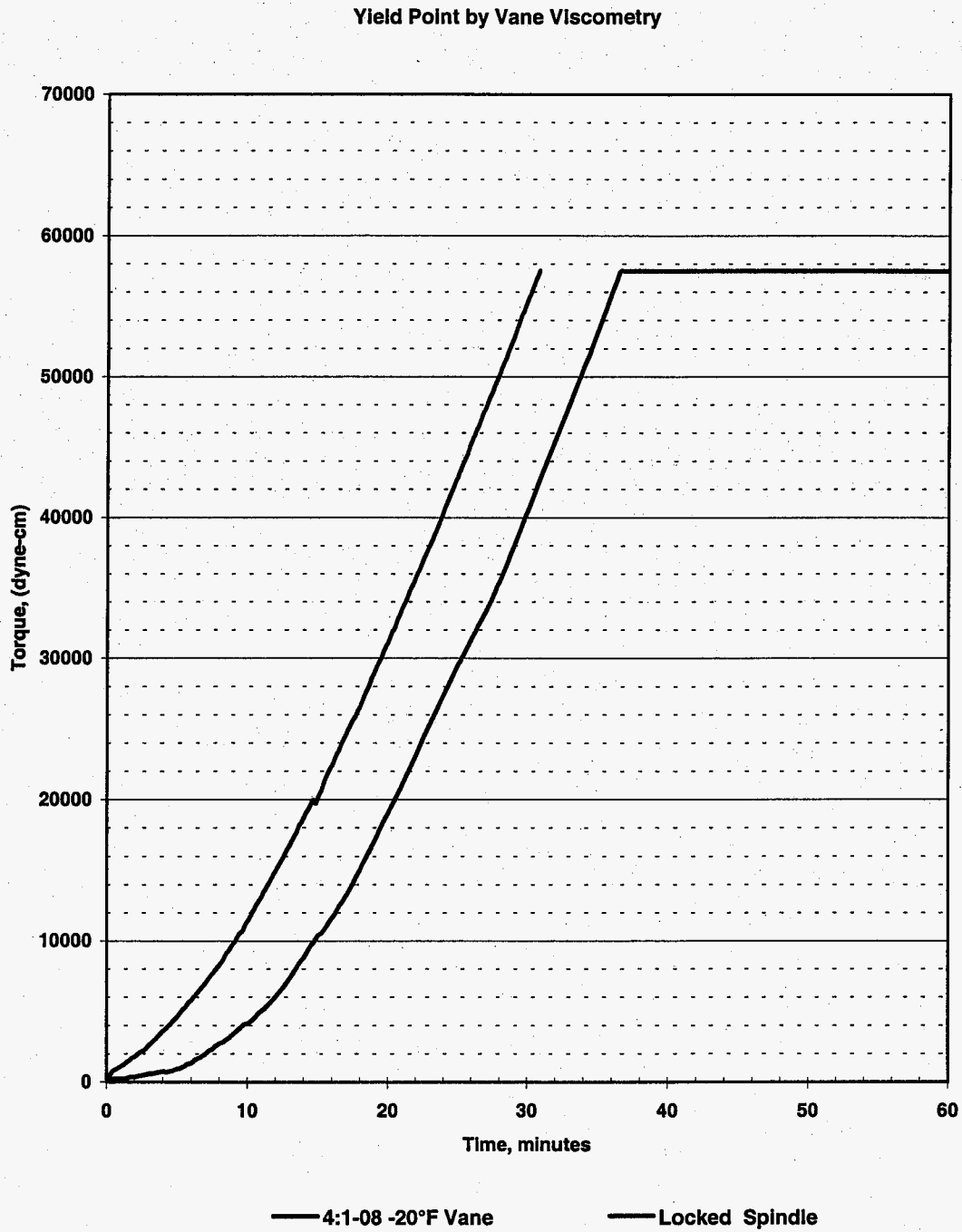**Yield Point by Vane Viscometry**

Figure 25: Sample 4:1 - 08 at -20°F   HB Viscometer

Yield Point by Vane Viscometry

Appendix A

Vane Viscometer - Yield Stress Data
For
GTL & Crude Oil Blends

100% GTL #2

3:1 Ratio Blend

25% GTL #2 + 75%TAPS Crude Oil at PS-1

4:1 Ratio Blend

20% GTL #2 + 80%TAPS Crude Oil at PS-1

Appendix B

Standard Laboratory Procedure

(SLP-307)

"Crude Oil Yield Stress Value Determination By Vane Viscometry"

Subject: Crude Oil Yield Point Determination by Vane Viscometry	Document No: SLP-307
Date: July 31, 2000	Revision 3

Prepared by: Neal Magri
Applicable to: Westport Technology Center International

Technical Review by: Bayram Kalpakci	Date: July 31, 2000
Safety Review by: Robert Jaros	Date: July 31, 2000
Quality Assurance Review: John Shillinglaw	Date: July 31, 2000

Scope: This test method describes the use of the Brookfield viscometer for the determination of the yield stress value of crude oils.

Safety Precautions: Approved safety glasses with side shield and protective clothing must be worn at all times in the laboratory. Protective gloves are required to be worn when handling crude oil and solvents. Keep away heat, sparks and open flame. Use only with adequate ventilation, (i.e. sampling performed within a fume hood if possible). Avoid contact with skin, eyes and clothing. Avoid breathing mist or vapor. Keep containers closed. Open containers with caution.

Important: Crude oil and container will be hot after the initial heating during the beneficiation process. Handle and dispose of syringes and needles properly. Empty containers may contain toxic, flammable/combustible or explosive residue or vapors. Do not cut, grind, drill, weld, reuse or dispose containers unless adequate precautions are taken against these hazards. Observe ALL PRECAUTIONARY LABELING.

Hazard: CRUDE OIL, Vapors may be harmful. Possible aspiration hazard if swallowed, can enter lungs and cause damage. May be irritating to the skin, eyes and respiratory tract. May release toxic hydrogen sulfide vapors. Skin cancer hazard based on tests with laboratory animals. Contains BENZENE—a cancer hazard. Extremely flammable liquid. Vapor may cause flash fire.

Reference Documents: ASTM D2983 Standard Test Method for Low-Temperature Viscosity of Automotive Fluid Lubricants; Westport Standard Procedure SLP-305 Rheological Properties of Crude Oils by Rotational Viscometer; Brookfield Digital Viscometer Model DV-II+ Version 2.0, Operating Instructions, Manual No. M/92-161-F1193; Brookfield WinGather Software, Operating Instructions, Manual No. M/95-320-C398; Operations and Programming Manual for Sigma Systems M26-C3 Environmental Chamber with Programmable Temperature Controller / Model CC-3. Dzuy, N.Q., Boger, D.V., "Yield Stress Measurement for Concentrated Suspensions", J. Rheology, 27 (4), 321-9 (1983).

Subject: Crude Oil Yield Point Determination by Vane Viscometry	**Document No: SLP-307**

Date: July 31, 2000	**Revision 3**

Subject: Crude Oil Yield Point Determination by Vane Viscometry	Document No: SLP-307
Date: July 31, 2000	Revision 3

Summary of Test Method

This test method consists of determining the yield stress value of a crude oil by measuring the torque on a spindle, using a Brookfield viscometer, rotating at 0.01 rpm in the material. The spindle to be used consists of four rectangular vanes dimensioned (0.75"w x 2.25"h) and oriented at 90 degree increments around the central axis. The sample cup is dimensioned (1.5" id x 4.0"h). Vertical orientation of vanes within the sample cup is dimensioned (1.00" from top and 0.75" from bottom). The crude oil is initially heated to 150°F to destroy all temperature and shear histories and then cooled to 90°F at which stress value it is loaded into the cup apparatus. The cup apparatus holds the vanes rigidly during cooling and aging and prevents loss of light ends through evaporation. After loading into the cup apparatus the sample is cooled in an environmental chamber at a controlled rate to -20°F. The cooling rate mimics the expected rate of cooling of the pipeline oil in the case of shut-in.

Samples are withdrawn from the environmental chamber at 10 test temperatures (approximately 80, 65, 50, 40, 20, 10, 0, -10, -15 and -20°F) and transferred to a refrigerated circulator that maintains the sample at test temperature. The spindle is attached to the Brookfield viscometer before the spindle clamping mechanism is released. The clamping mechanism is released and the viscometer is started at 0.01 rpm and torque as a function of time is measured, at least until a maximum reading is obtained. The maximum torque obtained is divided by a vane parameter constant K to obtain the yield stress. The constant K is calculated based on the dimensions of the vanes. (K=36.19 cm^3, for a Vane with D=0.75 inch (1.905 cm) and H=2.25 inch (5.715 cm))

Significance and Use

The test method is used for determining the yield stress value of a cooled crude oil, with or without aging. This determination will be made with vane spindles, which extend horizontally through a sample, minimizing the impact of slippage at the spindle wall. The method will determine the minimum amount of torque necessary to initiate oil movement at low shear, and subsequent viscosity of the fluid after initiation of flow. These data can be directly used in modeling of crude oil behavior in pipelines, during start-up conditions.

Equipment Required

- Certified rotational-type viscometers capable of a minimum rotational speed of 0.01 rpm such as: the Brookfield Viscometer, Model LV DVII+, RV DVII+ or HB DVII+ having the capability of 20 speeds when programmed accordingly.
- Thermometer, Fluke digital thermometer which has been certified to ±0.3°C from –40°C to 100°C and ±2.0°C from –50°C to –100°C, using standards traceable to the National Institute of Standards and Technology or the National Physical Laboratory or using natural physical constants or ratio calibration techniques.
- Westport vane viscometry cup apparatus and vane spindle for the Brookfield viscometer.
- 100 ml glass syringe, and syringe needle equipped with valve.
- Sigma System M26-CC3 environmental chamber, equipped with a liquid nitrogen supply for cooling.
- Temperature controlled refrigerated circulator bath, such as a Julabo FP-50 series.

Subject: Crude Oil Yield Point Determination by Vane Viscometry	Document No: SLP-307

Date: July 31, 2000	Revision 3

PROCEDURES

A. Calibration of Apparatus

Calibration as it is normally understood, carrying out an experimental measurement with a standard material, in the same manner as for the unknown sample, does not apply to vane viscometry; there are no standards. However, as received, from the factory the Brookfield viscometers are certified to give accurate speed and percent torque readings, and it is these two measurements which are critical to vane viscometry.

Tests of consistent viscometer response will be carried out as verification that the viscometer is in good working order. Testing will be carried out for each viscometer when used in each experimental set (an experimental set entails the testing of all samples cooled in one set in an environmental chamber, generally a population size of 24).

1.0   Procedure

1.1 Level the Brookfield viscometer, and the plastic bracket to hold the vane apparatus using the spirit levels attached to them.

1.2 Take an empty vane apparatus, with the top removed but with the spindle locked, and place the apparatus in the refrigerated circulator, temperature is not critical at this step.

1.3 The vane apparatus is held in place by a plastic bracket; make certain the apparatus is placed as far to the right as possible and as far towards the back of the bath as possible.

1.4 Tighten the clamp that holds the vane apparatus in the bracket.

1.5 Attach the alignment rod to the Brookfield viscometer. Move the Brookfield viscometer vertically and horizontally until the alignment rod fits into the top of the vane spindle (without lateral motion).

1.6 Remove the alignment rod and screw on the S hook attachment to the Brookfield viscometer.

1.7 Loosen the clamp that holds the vane apparatus in the bracket.

1.8 Attach the vane spindle to the Brookfield viscometer as shown in Figure 1.

1.9 Raise the Brookfield viscometer until all slack is taken out of the connections to the vane spindle but the vane spindle is not lifted off its plastic support bracket.

1.10 Rotate the vane apparatus until the torque reading is less than 0.05%. It is best not to start with negative torque readings as these are not recorded by the WinGather program.

1.11 Tighten bracket that holds the vane apparatus in place, using the knob in the top right corner of the bracket.

1.12 Start the WinGather program for timed torque readings. Time between readings will be 10 seconds for a speed of 0.01 rpm.

As the Brookfield software does not recognize the vane spindle, spindle input is not necessary. Time, torque and temperature readings, which are independent of spindle, are the only data that will be used.

The WinGather program is Version 1.1 from Brookfield Engineering Laboratories, Inc., located at 11 Commerce Blvd., Middleboro, MA 02346. See referenced manual for further WinGather information. The manual and a copy of the software will be archived at Westport Technology Center.

1.13 Start the Brookfield motor with the locked spindle mechanism in place.

1.14 Continue the test run until the torque reading goes off scale.
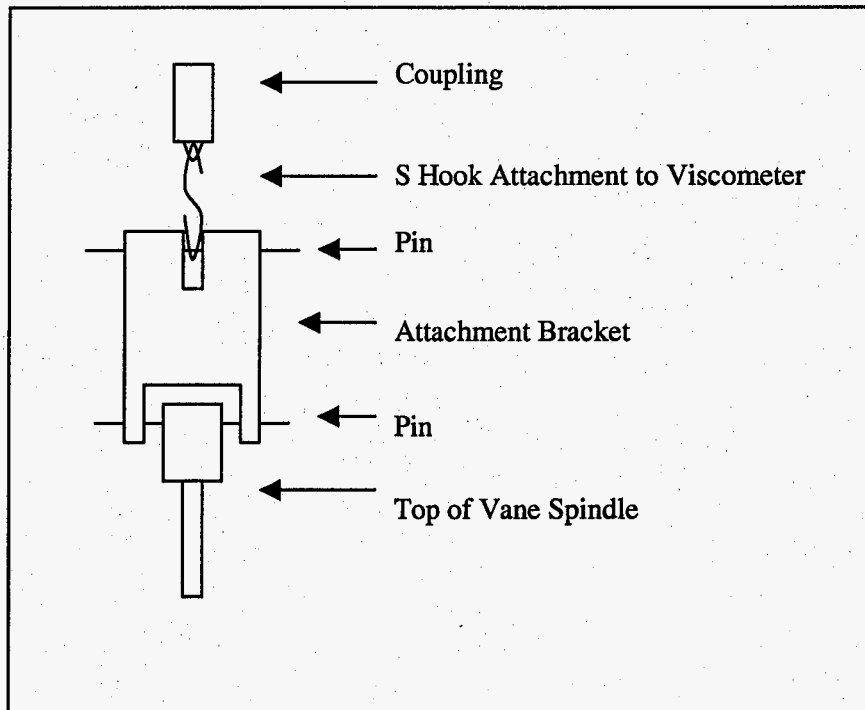1.15 Save the WinGather data file and record the data file name.

Figure 1. Attachment of Vane Apparatus to Viscometer

2.0   Acceptable Calibration

Differences in torque versus time measurements will include variations in viscometer speed and torque measurement. For purposes of measuring yield stress, variances in viscometer speed are not critical. Speed is important however for determining slippage of the sample at the yield stress value. A torque versus time run will be acceptable if it varies no more than 10% of the maximum torque of the viscometer, at any stress value in the experimental run.

B.   Verification of Cooling Rate

1.0 Data Recording

Temperatures will be measured by the environmental chamber thermocouple and recorded by a YEW Model 3088 Hybrid Recorder (chart recorder). The chart recorder will print time and temperature at 0:00 and 12:00 each day, in addition to a continuous trace of the temperature. This temperature recording procedure has been verified against a certified Fluke thermometer and found to be within ±1°F. The temperature recording of the environmental chamber and Yew recorder will be re-verified each time the Fluke digital thermocouple meters are re-certified.

1.1　At the end of each vane experimental run, when all samples originally placed in the environmental chamber have been tested or disposed of, remove the chart recorded paper from the recorder. This data is to be saved, and archived with other items for this test method, such as laboratory notebooks.

1.2　The chart recording should be inspected to ascertain if there are any significant anomalies, such as rapid, transitory increases or decreases in temperature.

1.3　Printed temperatures from the chart recorder should be recorded and contrasted with target temperatures as part of final reporting.

Table 1.　Times and Target Temperatures

Day (24 hrs)	Target Temperature (°F)
0	90
2.5	70
5.0	50
7.5	35
10	20
12.5	10
15	0
17.5	-10
21	-20

C. Preparation of Sample

The solubility of paraffins in crude oil decreases with decreasing temperature. As an oil cools past its wax appearance temperature, regardless of the rate at which the oil was cooled, significant amounts of paraffin may precipitate. However, the rheological properties of the precipitated wax are highly dependent upon the shear and temperature history of the oil above and below the wax appearance temperature. The initial step in determining yield stress of an oil is to heat the sample to 150°F and hold the oil at that temperature for at least 2 hours, to destroy all temperature and shear histories. It is important to make certain the oil container is tightly closed during this initial conditioning, as loss of light ends through evaporation may significantly increase yield stress.

Following the initial heating of the oil, allow the oil to cool to 90°F in a Sigma environmental chamber. At this stress value the oil containers, the vane apparatuses and the 100-ml glass syringe for oil transfer are thermally equilibrated in the environmental chamber at 90°F. Allow at least an hour for all materials to reach temperature.

D. Loading of Vane Apparatus

A 100-ml glass syringe equipped with needle and valve is used to load the oil sample into the vane apparatus.

1. Take the syringe, one sample bottle (120-ml) and one vane apparatus out of the environmental chamber that is set at 95°F to 100°F.
2. Remove the plunger from the syringe, invert the syringe and close the needle valve.
3. Pour 100 ml of crude oil into the syringe and replace the plunger.
4. Turn the syringe upright, open the needle valve and expel the gas bubble.
5. The syringe should contain at least 95 ml of oil but no more than 98 ml. If necessary add more oil or expel some of the oil into a waste container.
6. Inject the oil sample through the loading port of the vane apparatus.
7. Set the top-sealing chamber of the cup apparatus in place and place the vane apparatus back into the environmental chamber.
8. Place a Styrofoam block on top of the vane between the vane and environmental chamber roof, to hold the top down.
9. Repeat steps 1-8 until all samples have been loaded.

E. Controlled Cooling and Aging of Samples in Environmental Chambers

The environmental chamber's programmable temperature controller allows up to 100 program steps. Each step has 5 sub-steps (0-4). To program ramp and soak functions select a step, and input setting specific parameters in the corresponding sub-steps.

The sub-step parameters and ranges are as follows:

0	+/- TTT.T	Set stress value temperature displayed to the nearest 0.1°C (+200 to −100°C)
1	HH.MM	Ramp time hours (00-99), minutes (00-59)
2	HH.MM	Hold time hours (00-99), minutes (00-59)
3	NNN	Next step (00-99, 100=end)
4	P	Active temperature control probe (1 or 2)

As described in section B cooling will be carried out in 8 steps with a final hold time of 99 hours to allow for aging if required.

F. Measurement of Yield Stress

1. If an approximation of the yield stress for the sample under test conditions is not available make the initial measurement with the Brookfield RV viscometer. Prior measurements, at warmer temperatures, will dictate selection of appropriate Brookfield viscometer for future testing, at colder temperatures.
2. Level the Brookfield viscometer, and the plastic bracket to hold the vane apparatus using the spirit levels attached to them.
3. If the viscometer has not been previously aligned during calibration do so following steps 1.2 through 1.7 in section A.
4. Equilibrate the Brookfield temperature bath at −2°F below test temperature, as the test sample will be warmer than the bath temperature due to heat picked up from the top of the vane apparatus.

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5. Remove a vane apparatus from the environmental chamber along with the insulated container of ethylene glycol. Place the vane apparatus in the insulated container.

Important: Even though the vane spindle is locked in position all movements of the vane apparatus should be made as gently as possible to reduce the possibility of disrupting wax networks present.

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6. Gently carry the vane apparatus to the Brookfield viscometer.
7. Remove the top of the vane apparatus.
8. Place the apparatus in the refrigerated circulator at temperature. Move the apparatus as far to the right and toward the back of the bracket as possible.
9. Pin the vane apparatus spindle to the Brookfield spindle as shown in Figure 1.
10. Adjust the height of the viscometer so that all slack is taken out of attachments but the vane apparatus is not lifted from the plastic bracket
11. Rotate the vane apparatus so that the torque reading is as close to zero percent as possible.
12. Tighten the knob that holds the vane apparatus in place.

Important: At this stress value the vane spindle will be released from its locking mechanism. Careful handling of the apparatus from this stress value onward is especially critical to avoid uncontrolled breakdown of any wax networks formed.

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13. Start the WinGather program for timed torque readings. Time between readings will be 10 seconds for a speed of 0.01 rpm.

As the Brookfield software does not recognize the vane spindle, spindle input is not necessary. Time, torque and temperature readings, which are independent of spindle, are the only data that will be used.

The WinGather program is Version 1.1 from Brookfield Engineering Laboratories, Inc., located at 11 Commerce Blvd., Middleboro, MA 02346. See referenced manual for further WinGather information. The manual and a copy of the software will be archived at Westport Technology Center.

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14. Gently remove the locking pin from the vane apparatus.
15. Gently cut the band holding the locking clamps in place.
16. Gently separate the locking clamps slightly.
17. Start the Brookfield motor.
18. Continue the test run until a stable torque reading has been obtained or until the torque reading goes off-scale.

If maximum torque reading on the Brookfield RV is <9% of full scale (647 dyne-cm), repeat steps F1 through F18 with a new sample using the LV Brookfield viscometer.

If torque reading goes off-scale, it will be necessary to repeat steps F1 through F18 with the next strongest Brookfield viscometer (HB). If yield stress is beyond the full-scale capabilities of the Brookfield HB viscometer, stop testing and report accordingly.

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19. Save the WinGather data file and record the data file name.
20. At test completion, insert certified Fluke thermocouple into vane apparatus and take a final sample temperature.
21. Repeat the test as stated:
    - Under test conditions where (possibly above the oil's WAT) observed torque readings are below 10% full scale on the LV viscometer (lowest torque spring viscometer) only test one sample.
    - If torque readings are above 10% full scale on the LV, then run a second sample for repeatability. If the two test results are not within 10%, run a third sample for precision purposes.
    - Three samples should always be tested at the lower temperatures if test samples are available. If necessary, for precision statements, repeat additional times.

G. Data Recording

Initiate Data Verification and Validation Checklists. Data recording, which will include quality assurance tests such as calibration and data checks, will be achieved using the enclosed vane viscometry data sheets.

Data Sheet 1 (DS1-307) includes recording of data when all the samples, for an entire tests, are prepared, transferred to their respective vane apparatuses, cooled to test temperature and aged.

The data to be recorded include:

- Test Number; Designate the test by number so that subsequent individual vane data sheets (DS2-307) can be traced to the parent data sheet. (i.e. IDC Test 1, Phase 1–Test 1...)
- Sample Description; Identification of sample (i.e. TAPS Mix, PBU, Kuparuk...)
- Sample Benefication; Record temperature treatment information for the samples before transfer to the vane apparatuses.
- Transfer of Samples; Record start and stop times for transfer of all samples from original bottles to vane apparatuses to environmental chambers.
- Cooling/Aging of Sample; Record temperature of environmental chamber when samples are first introduced, also record target temperature (normally –20°F) and the target period of time for cooling samples to that temperature (normally 21 days). Temperature/time measurements will be recorded by the Yew hybrid chart recorder.

Data are to be recorded for each individual sample withdrawn and tested from the environmental chambers on vane viscometry Data Sheet 2 (DS2-307). Record data including:

- Test Number; the same number as used on DS1-307, establishes tracking of DS2-307 sheets.
- Sample Description; Identification of sample (i.e. TAPS Mix, PBU, Kuparuk...)
- Testing; date and time of sample withdrawal, the temperature of the environmental chamber, the name of the WinGather data file where testing data is stored, the type of viscometer (LV, RV or HB) and the speed (rpm) at which the test was carried out (normally 0.01).

- Data Check; data for the vane testing will be recorded using the WinGather program. The data check section of the data sheet will be used for quality assurance of this WinGather data. During testing (at 5 to 10 minute intervals), record the percent full scale (torque) reading directly from the Brookfield viscometer display, and record the data stress value of the WinGather program where this data was recorded electronically.
- Maximum Torque; Record the observed highest percent full-scale reading (torque).
- Data Stress values of Maximum Torque; Record the approximate range of WinGather stress values during maximum torque (i.e. numbers 73-78).
- Final Oil Temperature; Record the sample temperature after completion of test.

H. Calculation and Interpretation of Results

The experimental test is designed to produce direct readings, temperature and %torque, as data.

Calculation of torque and yield stress.

1. Calculate torque readings from the percent of full-scale readings recorded by the WinGather software by multiplying the percent full-scale reading by 6.733 dyne-cm for the LVDV-II+ viscometer, or 71.87 dyne-cm for the RVDV-II+ viscometer, and 574.96 for the HBDV-II+ viscometer. Torque data may be interpreted by graphing the torque versus time readings obtained during testing. Determine a yield stress value from this data by observing where a maximum torque reading had been obtained, followed by a decrease in torque reading over time. Calculate the Yield Stress by the following equation;

$$\text{Yield Stress} = \text{Maximum Torque Obtained (dyne-cm)} / K \ (36.19 \ cm^3)$$

I. Reporting

1.   Report the following information:
      1.1   Completed and signed Data Verification and Validation Checklists,
      1.2   Date of test,
      1.3   Sample Identification,
      1.4   Cooling time for sample,
      1.5   Aging/testing temperature in degrees Fahrenheit (environmental chamber temp.),
      1.6   Final oil temperature at end of test (measured directly in cup),
      1.7   Viscometer speed,
      1.8   Maximum torque reading,
      1.9   Yield Strength versus Temperature,
      1.10 Locked Spindle Torque versus Time Curve,
      1.11 Time versus torque reading for each test will be reported graphically,
      1.12 Numerical data for the graph,
      1.13 Combined Plot of all Torque vs. Time Curves on one Log Scale Plot,
      1.14 When multiple oils are analyzed, plot all Yield Strength vs. Temperature curves on one Lop Scale Plot.

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J. Precision

Precision – See Section A2 for precision during QC/QA calibration checks. Initial Demonstration of Capability for test resulted in the following;

Determinability (d) – Measurements are performed on individual oil samples, taken at selected temperatures, through a maximum shear condition which destroys any wax structure present. No attempt was made to duplicate measurements on the same oil sample within the test cell; the results would be misleading. Therefore, no statement of determinability can be made on.

Repeatability (r) - The difference between successive results obtained by the same operator in the same laboratory with the same apparatus under constant operating conditions on identical test material would, in the normal and correct operation of this method, have a relative standard deviation at or below 15%.

Reproducibility (R) - The Brookfield viscometers are a very common apparatus for measuring rheological properties. However, with the Vane cup and spindle apparatus design and modifications for low temperature testing of the TAPS/COS samples, no statement of reproducibility by other independent laboratories can be made.

Data Sheet 1 for Vane Viscometry Form: DS1-307

Heating, Transfer, Cooling, and Aging for Sample Sets

Test Number:	
Sample Description:	

Sample Beneficiation

Date:		Start Time:	
Temp.:		Stop Time:	

Transfer of Samples

Start Time:
Stop Time:

Cooling/Aging of Samples

Chamber Temp.:		Target Temp.:	
Ramp Start Time		Target Time:	

Sample Identification

Chamber 1		Chamber 2	
Vane No.	Sample ID	Vane No.	Sample ID

Analyst Signature:_____

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Data Sheet 2 for Vane Viscometry	Form: DS2-307		
Withdrawal and Testing of Individual Samples			
Test Number:			
Sample Description:			
Sample Testing			
Withdrawal Date and Time:		Viscometer:	
Chamber Temp.:		Viscometer Speed:	
WinGather File Name:			
Data Check			
WinGather Data Stress value Number	Percent Full Scale (Torque)	WinGather Data Stress value Number	Percent Full Scale (Torque)
Maximum Percent Full-Scale (Torque):			
Data Stress values of Maximum Torque:			
Final Oil Temperature:			

Analyst Signature:_____