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Review of SRC-II Process Product Hydrotreating Data, for Completeness and Applicability to the Demo Plant

G. C. Frazier
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Fossil Energy Program

REVIEW OF SRC-II PROCESS PRODUCT HYDROTREATING DATA,
FOR COMPLETENESS AND APPLICABILITY TO THE DEMO PLANT

Final Report

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REVIEW OF SRC-II PROCESS PRODUCT HYDROTREATING DATA,
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George C. Frazier and Ahsan Faruqi

ABSTRACT

The SRC-II product naphtha, gas oil, and whole liquid product hydrotreating data base was reviewed for completeness and applicability to the design of the SRC-II Demonstration Plant. Primary data sources were the Chevron and the UOP experimental hydrotreating work on SRC-II product blends from the Fort Lewis Pilot Plant and the PNL bioassay investigations on UOP hydrotreated samples. Secondary data important to this task were the hydrotreating data for both the EDS and also the H-Coal liquid products. This study is thus based on the current literature and does not reflect ongoing investigations at Gulf and Battelle, PNL in areas such as toxicology and fuels compatibility.

The SRC-II product hydrotreating data are sparse at this point, but in view of the fact that these data can be supplemented in useful ways through correlation with the corresponding data for the EDS and H-Coal liquid fractions, it is concluded that a sufficient data base exists for the closure of a hydrotreating plant material balance, at least in the hydrotreating severity range of the Phase 0 design goals. However, factors may have to be introduced to account for the data scatter, particularly with respect to the gas yields. Little thermal property data appear in the literature for coal-derived liquids; thus it will be necessary to draw upon correlations developed in the petroleum industry for closure of the plant energy balance unless experimental thermal property data are forthcoming.

An area of uncertainty and concern at this time is the extent to which the Demo Plant product mutagenicity would be reduced by hydrotreatment. Although bioassay tests were conducted on both moderately and severely hydrotreated SRC-II Pilot Plant gas oil, the Demo Plant product is expected to contain an appreciable fraction of 800°F+ material, compared with the Pilot Plant product, which contained essentially no material in this higher boiling range.

Finally, data could not be found which detail the extent to which the storage stability of these or similar liquids is improved with extent of hydrotreatment.

SUMMARY AND RECOMMENDATIONS

The available data on the hydrotreating of the SRC-II product, with particular emphasis upon the naphtha fraction, were examined in order to assess its applicability and completeness for the design of the SRC-II Demo Plant. The primary sources of the data were the Chevron and UOP work on the distillate blends received from the Fort Lewis Pilot Plant. The hydrotreating data at this particular time are sparse and somewhat scattered, but it was found that they could be supplemented in some instances at least by data on similar liquids, such as the EDS and the H-Coal products.

No firm criteria or quantitative guidelines could be found for establishing a useful level of severity of on-site hydrotreatment of the SRC-II Demo Plant naphtha or gas oil. Although it is generally accepted that hydrotreatment will stabilize freshly formed coal-derived liquids, no data could be found in the literature which show the extent to which these liquids are stabilized as a function of the hydrogen added during hydrotreatment. On the other hand, data are available which show the extent to which the SRC-II gas oil mutagenicity is reduced by hydrotreatment. Yet, no criteria are known for setting the level to which the mutagenic response of this or similar liquids should be reduced. It therefore appears that the extent to which the SRC-II naphtha and possibly gas oil fractions will be hydrotreated on site will be established more by the good judgement of the designers than any by other basis, unless additional data are forthcoming.

Given that the decision is taken to hydrotreat the Demo Plant naphtha and possibly the gas oil on site, our assessment is as follows:

Naphtha Hydrotreating

There are sufficient experimental data for the design of a naphtha hydrotreating unit which will remove an estimated 99.2 wt. % of the nitrogen if the hydrogen consumption is 350 SCF/bbl (Phase 0 design), but factors may have to be introduced into the design to account for the sparcity of the data, its scatter, and certain extrapolations which

will be necessary. As much as 1000-1100 SCF/bbl of hydrogen have been consumed during the hydrotreating of SRC-II naphtha, so additional hydrogen beyond that called for in the Phase 0 design could be added to the naphtha without exceeding the range of the available data. Major data weaknesses are in the areas of the limited range of process conditions (temperature, pressure, LHSV), and the scatter in the gas yield data. Insufficient data are available for process optimization, but the SRC-II data are supplemented considerably by the use of the EDS and the H-Coal naphtha hydrotreating data, particularly in the area of nitrogen removal as affected by hydrogen consumption.

Thermal property data from petroleum correlations can be used to estimate the energy requirements of a naphtha hydrotreating process, but data for coal-derived naphthas, particularly heat capacity and latent heat of vaporization data, would allow more precise estimation of the process fuel requirements.

Gas Oil Hydrotreatment

The fact that the SRC-II gas oil samples which were hydrotreated by Chevron and UOP were lighter than the corresponding product expected from the Demo Plant is a troublesome point when it comes to estimating hydrogen consumption, gas yields and processing conditions, should the decision be taken to hydrotreat the gas oil on site. However the experience to date with some similar gas oils, such as the EDS and H-Coal products, can be used to supplement the SRC-II data, especially with respect to gas yields and hydrogen consumption. Missing are the thermal data required for reasonably precise closure of the process heat balance, although data from the petroleum industry can be used for an approximate heat balance design. One must be prepared to accept errors in the hydrotreating plant heat balance as large as 7% unless additional thermal property data on coal liquids are forthcoming.

The PNL bioassay tests on both moderately and severely hydrotreated SRC-II Pilot Plant gas oil samples showed that up to 99% reduction in mutagenicity can be produced by hydrotreatment. However, these tests were conducted on an oil with an end point of 800°F. The response of

the mutagens in the Demo Plant product to hydrotreatment is therefore questionable as it is expected to contain an appreciable fraction of 800°F+ material. This is particularly significant in view of the fact that mutagens tend to be concentrated in the higher boiling fractions of coal-derived liquids.

Whole Liquid Product Hydrotreating

The SRC-II whole liquid product can be hydrotreated by commercial technology up to chemical hydrogen consumptions of about 3,100 SCF/bbl. This level of hydrotreatment can remove as much as 99.999% of the nitrogen and 99.89% of the sulfur from the SRC-II Pilot Plant samples. As these samples were somewhat lighter than the liquid expected from the Demo Plant, exact prediction of the yields, particularly the gas yield, from these data cannot be expected. The heteroatom removal data provide reasonably good correlation with hydrogen consumption as well as with the change in API gravity. The lack of thermal property data may introduce errors in the process heat balances by as much as about 7%, according to a previous study. Mutagenic response test data for the whole liquid were not found, but it should be possible to make at least approximate estimates based on the PNL data on the SRC-II gas oil.

Based upon these findings, we make the following recommendations:

- Hydrotreating data correlation and analyses. The data correlations put forward in this work can be considered no more than preliminary. Yet this effort does show that data from similar liquids such as the EDS and H-Coal products can supplement and fill data gaps in the SRC-II hydrotreating data base. It would be useful to continue this data compilation, analysis, and interpretation in terms of basic kinetic models of the hydrotreating process, incorporating the remaining experimental data to be generated by the UOP and Chevron programs as they become available. The generation of additional hydrotreating data beyond those currently planned is probably not warranted at this time. However, this situation should be reviewed after

the completion of the analysis and interpretation of the data produced by the current experimental programs.

- Thermal property data. Reasonably precise closures of the hydro-treating plant heat balance and fuel requirement estimates cannot be made with confidence until thermal property data are available on coal-derived liquids. It is recommended that an experimental program designed to obtain these data be implemented forthwith.
- Storage stability data. An experimental program should be organized to generate the necessary data to show the extent to which SRC-II product, particularly the naphtha, is stabilized by level of hydrotreatment.
- Tolerable mutagenic response levels. A group composed of experts from a wide variety of disciplines should be organized and charged with the responsibility of establishing criteria and guidelines for tolerable mutagenicity levels for the SRC-II and similar coal-derived naphtha and gas oils.

1. INTRODUCTION

A decision has been taken jointly by the Department of Energy and the Pittsburgh and Midway Coal Mining Company (P&M) to demonstrate the SRC-II process technology in a plant to be constructed on a site near Morgantown, West Virginia. The Phase 0 design study for this plant was completed in 1979.¹ The Phase I design study is currently under way, with the objective of firming up the plant design in detail.

This demonstration plant would process about 6,000 t/d of a Pittsburgh seam coal to about 16,200 bbl/d of syncrude equivalent, including about 3,850 bbl/d of naphtha and 8,350 bbl/d of fuel oil.² The details of the product slate and the associated product specifications have not been finalized at this time, but the Phase 0 design calls for a moderately light hydrotreatment of the naphtha, presumably for stabilization purposes. Any treatment of the fuel oil would presumably be accomplished at existing off-site facilities. The

hydrotreated naphtha would also be transported to existing refining facilities for further processing.¹

The Demonstration Project provides for hydrotreatment of the naphtha, and a Phase I design evaluation on hydrotreatment of the light gas oil and the heavy gas oil.³ This study will address the hydrotreating of naphtha, which is an approved process for the Demonstration Plant configuration, and the hydrotreating of light gas oil and heavy gas oil, which are considerations for commercial plants. A question remaining at this time is the extent (e.g., moderate or severe) to which the naphtha, in particular, is to be hydrotreated. The level of hydrotreatment is important because hydrotreatment and its level of severity will impact on the Demonstration Plant design in areas such as the overall plant hydrogen balance including gasification requirements, the plant fuel balance, pumping requirements, utilities, gas production and cleanup, and emissions control facilities.

The study reported on here was therefore undertaken with the objective of reviewing the SRC-II hydrotreating data base in order to determine whether or not there are sufficient data in those areas such as hydrogen requirements, gas yields, heteroatom removal, product stability, and others, to provide a sound basis for design. Particular attention was also given to determining whether or not there are gaps in the data and, if so, how they might be bridged.

In view of the fact that the Demonstration Plant product slate and its specifications are no more than preliminary at this time,² the approach taken here was to consider a set of plausible directions, or cases, which the project might take with respect to the processing of the syncrude to final products, and then to assess whether or not the data base is adequate for the design of the required hydrotreating area of the plant, and the prediction of required product properties, such as extent of heteroatom removal and desired stability characteristics. Cases which were considered are as follows:

1. Hydrotreating of only the naphtha at the Demonstration Plant.
2. Hydrotreating of the gas oil as well as the naphtha at the Demonstration Plant.
3. Hydrotreating of the entire syncrude liquid at the Demonstration Plant.

Each of these cases was considered at two levels of severity: moderate and also high severity. To assess whether or not the current SRC-II hydrotreating data base is adequate for plant design and product characterization, the following possible reasons for hydrotreatment of the Demonstration Plant syncrude were identified:

- To stabilize the liquid against deterioration during storage or transport. Freshly formed syncrudes are known to be unstable, but their stability is improved with hydrogen treatment.
- To prepare the liquid for further downstream processing, such as reforming or hydrocracking or possibly fluid catalytic cracking, especially with respect to increased hydrogen content and reduced metals content.
- To reduce the heteroatom content for one or more of the following reasons:
 - odor improvement (sulfur removal). There is a risk of off-site spills if the syncrude is transported to other processing locations. Reduction of the material's sulfur content by hydrotreatment at the Demonstration Plant would make such spills less objectionable.
 - to reduce the mutagenicity of the liquid. Due to the lack of spills, this question becomes of increased concern if the syncrude is to be transported from its source to other sites for processing. The bioactivity of syncrudes is known to be due primarily to certain organic nitrogen compounds, which can be reduced by hydrotreatment.
 - to meet certain product specifications, such as sulfur content.
 - to protect the catalyst in a downstream processing step, such as reforming. Reforming catalysts, especially, are highly sensitive to nitrogen. Reformer feedstocks in the petroleum industry are generally limited to those materials in which the nitrogen content has been reduced to no more than a few tenths of a part per million.

- To reduce the aromatic content of the material in the light gas oil range, especially if the production of jet fuel becomes of interest.
- Relative hydrogen cost and availability at the Demonstration Plant vis-a-vis cost and availability at alternate upgrading locations such as refineries. The impact of the relatively small amount of syncrude from the Demonstration Plant (no more than about 12-13 Mbbl/d) on the hydrogen balance of a reasonably large petroleum refinery would be small, which suggests it might be more economical to conduct the major portion of the hydrotreating operation in an existing facility, especially in view of the fact that the hydrogen recovered from the naphtha reforming process can provide a significant part of the hydrogen required for hydrotreatment.

Thus there are some reasons for conducting the syncrude hydrotreating at both the Demonstration Plant site and at existing refinery locations, and it is in part the task of the designers to strike this balance. The task here was to attempt to determine by review of the existing data whether or not the designer is constrained in this task due to lack of, or gaps in, the hydrotreating data base.

Thinking beyond the SRC-II Demonstration Plant to subsequent commercialization, one needs to establish product quality goals for hydrotreating individual products. Part of this task is that of establishing product boiling range definitions. An acceptable set, for discussion purposes, might be as follows:

Naphtha	100 - 400°F
Kerosene-jet fuel	400 - 550°F
Light gas oil	400 - 750°F
Heavy gas oil	750 - 950°F

This does not imply that a plant must produce streams of these specific boiling ranges. However, such a classification method is essential to establish the hydrotreating requirements of dissimilar products.

For naphtha, it can be assumed that the ultimate objective is to produce a suitable feedstock for catalytic reforming. A nitrogen level

of less than one ppm must be achieved. If this is done, the data in this report show that the sulfur content will automatically be low enough. Further, this operation is feasible with conventional technology.

For streams above the gasoline boiling range, the product quality goals are more complex. Several products are involved (jet fuel, kerosene, distillate fuel oils) and the level of acceptability of sulfur, nitrogen, and other specifications (including possible future specifications) is not well established. However, the feasibility of meeting such specifications and establishing the process conditions and hydrogen consumptions needed is a topic which should be addressed in the near future.

Finally, it will be noted in the subsequent sections of this report that the product boiling ranges of the material hydrotreated in the UOP and Chevron laboratories do not always fall within the boiling range definitions set out above. However, the experimental results show that in many cases of interest at least the data from feedstocks of relatively different boiling ranges correlate with respect to hydrogen requirements. These findings, and others, need to be verified as the hydrotreating data base for coal-derived liquids expands.

2. HYDROTREATING DATA BASE

The hydrotreating data sources for the SRC-II syncrude are the work by Chevron (the FE-2315-series) and by UOP (the FE-2566-series). These groups hydrotreated both the naphtha and the gas oil, and in the case of Chevron, the whole liquid product, supplied from the Fort Lewis Pilot Plant. These data form the primary data base for this study. In addition to the primary data, additional data for liquids with some similarities to the SRC-II product were also examined, such as the EDS and the H-Coal syncrude hydrotreating data. These data are termed the secondary data for this study. Properly correlated, these secondary data can supplement the primary data base, in some cases at least.

The assembled data were reviewed both for applicability to the task of hydrotreating the Demo Plant product liquid and its naphtha and gas oil fractions, and for completeness with respect to:

- Hydrogen consumption as a function of degree of hydrotreating severity (temperature, pressure, and LHSV).
- Hetroatom removal, especially N and S, as a function of processing conditions and hydrogen consumption.
- Yields, especially gas yields, as a function hydrotreating conditions.
- Reduction in aromatic content, as a function of processing conditions and hydrogen consumption.
- Hydrotreated product storage stability, as a function of extent of hydrotreatment.
- Extent to which the product mutagenicity is reduced, as a function of degree of hydrotreatment.

A summary of the findings of this study is presented in the following sections.

2.1 Comparison of SRC-II Pilot Plant and Demo Plant Syncrude Inspections

The SRC-II syncrude supplied to Chevron and UOP for hydrotreating studies was derived from a Pittsburg seam coal.⁴ The Demo Plant is also reportedly being designed for a similar feed coal.⁵ However, there are some differences between the ultimate analyses of the Pilot and Demo Plant coals, particularly with respect to the nitrogen content. For example, the coal from which the UOP syncrude samples were derived contained 0.83% nitrogen, as compared with a Demo Plant projected range of 1.12 to 1.32% N, as shown by the analyses of Table A.1. It is therefore expected that the liquid produced in the Demo Plant will require more severe hydrotreatment than the Pilot Plant syncrude in order to achieve a given nitrogen level in the product. The sulfur contents of the pilot plant coal and the projected Demo Plant coal are comparable.

The inspections of the syncrude samples sent to Chevron and UOP are shown in Appendix A. Projected inspections of the Demo Plant

syncrude apparently are not available, so a complete comparison between the Pilot Plant and the Demo Plant syncrudes cannot be made at this time. However, projected distillation curves for the Demo Plant syncrude have been made,^{4,6} and are reproduced in Appendix A, Figures A.1 and A.2, and Table A.7. Based on these projected distillation curves, it appears that the Demo Plant syncrude will contain an appreciable amount of 800°F+ material, as compared with the syncrude from the Pilot Plant, which contained a negligible amount of 800°F+ material. The 800°F+ fraction of the projected Demo Plant liquid appears to be in the 10-15 vol % range. The response of this high boiling material to hydrotreatment is unknown at this time. However, it may be possible to extend the Chevron and UOP SRC-II hydrotreatment data by use of data for some heavier liquids, as pointed out at appropriate points in this study.

In summary, it appears the major differences to be expected between the Pilot and Demo Plant syncrudes are:

- The Demo Plant syncrude may contain an appreciably higher nitrogen content, depending on the coal used.
- The API gravity of the Demo Plant syncrude is expected to be appreciably lower than the Pilot Plant liquid, due to the presence of 10-15 vol % of 800°F+ material, which is not present in the Pilot Plant product.

Both of these factors make the Demo Plant whole liquid product a more difficult hydrotreating feedstock than the Pilot Plant product. If only the Demo Plant naphtha is to be hydrotreated, however, the possibly greater nitrogen content should be the only factor to be taken into account in attempting to use the Pilot Plant hydrotreating data as a basis for design of the Demo Plant.

It should be pointed out that there appear to be some inconsistencies in the data. For example, the nitrogen content in the Fort Lewis Powhatan No. 5 coal shipment is reported as 0.83% (Table A.1). With a coal nitrogen content in this range, it is difficult to see how the nitrogen content of the raw UOP liquid samples can be in the range of 8100-9040 ppm (0.81-0.904%) (Table A.5), as the coal liquefaction process

itself generally converts part of the coal nitrogen to volatile products such as ammonia. The Chevron whole liquid sample contained 0.85 wt. % N (8500 ppm) (Table A.2).

One final factor which could cause some difficulty in applying the available hydrotreating data for the prediction of Demo Plant results is that the Chevron and the UOP samples had aged for a considerable period of time (up to about one year in some cases) before they were processed through the hydrotreating reactors. There are little if any hydrotreating data available on fresh syncrude, so the extent to which aging affects the hydrotreating processing conditions required to achieve a given result is not known.

2.2 SRC-II Raw Naphtha Hydrotreating

Both Chevron and UOP have hydrotreated SRC-II raw naphtha.^{4,7} The ranges in process conditions used by each are shown in Table 2.2.1. The complete processing data are available from Chevron, but the processing temperature, pressure, and LHSV ranges used by UOP are available only in coded form. In addition to raw naphtha, Chevron also hydrotreated naphtha in two stages. The Chevron SRC-II naphtha hydrotreating data are therefore probably the most comprehensive data on this naphtha at this time. However, the second stage hydrotreating data may be of little use for the Demo Plant design unless the decision is taken to effect a severe naphtha hydrotreatment on site. The hydrotreatment of raw naphtha is considered to be the primary focus of this review. However, some of the Chevron results for hydrotreating of hydrotreated SRC-II naphtha are shown in Appendix B.

2.2.1 Naphtha hydrotreating data

The data shown in Table 2.2.1 indicate that the nitrogen content of the SRC-II naphtha can be reduced sufficiently for reformer feedstock (less than one ppm) by the use of commercial technology and process condition ranges. However, there are only a few data points available at this time for raw SRC-II naphtha, so a designer would likely be unable to optimize a hydrotreating process for this feed by consideration of

Table 2.2.1. SRC-II process raw naphtha hydrotreating conditions

Processing condition or property	Range	
	Chevron	UOP
Temperature range, °F	692-699	
T-T (base), °C		24
Pressure range, psig	793-799	
P-P (base), psi		450
Hydrogen pressure range, psig	642-645	Not reported
LHSV range	0.95-1.03	
LHSV/LHSV (base)		0.125
Recycle gas rate range, SCF/bbl	3,282-3,449	Not reported
Hydrogen consumption range, SCF/bbl	1,056-1,121	560
Boiling range of feed, °F	186-465	107-367
Nitrogen content range of product, ppm	0.1-0.21	0.8
Catalyst	ICR-113 (Ni, W, Si, Al)	DCB (Groups VI B and VIII elements)

Source: refs. 4 and 7.

Fig. 2.2.1. Hydrotreating of coal-derived naphthas for the production of reformer feedstock: hydrogen requirements as a function of the heteroatom content of the feed (from FE-2315-47; FE-2566-12, -25, and -27).

Fig. 2.2.2. Nitrogen removal from coal-derived naphthas during hydrotreating (data from FE-2315-47; FE-2566-12, -25, and -27).

these data alone. This constraint is relaxed somewhat, however, by the inclusion of the data for coal-derived naphtha produced by the EDS and the H-Coal processes, as can be seen in Figures 2.2.1 through 2.2.7. Keep in mind that data shown in these figures were generated with the primary objective of showing that these naphthas could be reduced in nitrogen content sufficiently for reformer feedstock. This means that the nitrogen of the SRC-II naphtha would have to be reduced by about 99.9+ wt.%, more or less, depending on the nitrogen content of the coal from which naphtha is derived.

The chemical hydrogen consumption required to reduce the nitrogen content to reformer feedstock levels is shown as a function of heteroatom content of the raw naphtha feed in Figure 2.2.1, for three coal-derived naphthas: SRC-II, H-Coal, and EDS. Though limited, these data appear to correlate over the range of about 0.95 to 4.2 wt. % feed heteroatom content. This suggests that the hydrogen consumption may be independent of the process used to generate the naphtha. One could therefore interpolate to predict the required hydrogen consumption for intermediate heteroatom contents, with some confidence. For example, for a naphtha of these types with a heteroatom content of 3.5 wt. %, a hydrogen consumption of about 960 SCF/bbl of feed is indicated. However, the data should be extrapolated outside the data range with caution, if at all.

The extent of the data correlation between nitrogen removal and hydrogen consumption is shown in Figure 2.2.2. These data suggest an essentially linear relationship between log nitrogen removal and hydrogen consumption, in the range of about 480 to 1120 SCF/bbl. Again one should be able to interpolate with some confidence to hydrogen consumption levels within this range. The data in the Phase 0 design report for the SRC-II Demo Plant¹ can be used to calculate a hydrogen consumption of about 350 SCF/bbl for the naphtha hydrotreatment. This is a relatively mild hydrotreatment and the data correlation of Figure 2.2.2 must be extrapolated in order to estimate the extent of nitrogen removal which would result from such a treatment. As pointed out above, extrapolations must be executed with caution, so the nitrogen removal value of 99.2 wt.% corresponding to an extrapolation of the

Fig. 2.2.3. Gas yield from coal-derived naphthas during hydro-treatment (data from FE-2315-47; FE-2566-12, -25, and -27).

Fig. 2.2.4. Effect of hydrotreatment on the API gravity of some coal-derived naphthas (data from FE-2315-47; FE-2566-12, -25, and -27).

hydrogen consumption to 350 SCF/bbl in Figure 2.2.2 is taken as an estimate, only, of the extent to which the SRC-II naphtha nitrogen content would be reduced by the Phase 0 hydrotreating specification.

The data of Figures 2.2.1 and 2.2.2 exhibit surprisingly little scatter in view of the fact that they were produced from three different naphthas and in different laboratories under somewhat different conditions. However, the gas yield data for naphtha hydrotreating, Figure 2.2.2, are somewhat more widely scattered. These data, when extrapolated to a hydrogen consumption of 350 SCF/bbl of naphtha feed, suggest the gas yield should be in the range of about 0.05-0.1 wt. % of the feed.

The change in the naphtha API gravity with hydrogen consumption is shown in Figure 2.2.4, for three naphthas: SRC-II, EDS, and H-Coal. These data, if extrapolated to the Phase 0 Design hydrogen consumption of about 350 SCF/bbl, suggest that the API gravity of the SRC-II Demo naphtha would not be changed appreciably.

The task of establishing precisely the hydrotreating processing conditions (temperature, pressure, LHSV) required to effect a naphtha hydrogen consumption in the range of, say, 350 SCF/bbl is difficult at this time, given only the published data, as these data have been generated for somewhat more severe conditions. As pointed out above, a hydrogen consumption of about 350 SCF/bbl by SRC-II naphtha should reduce the Demo naphtha nitrogen content by about 99.2 wt.%. This value is outside the range of the processing condition parameters, as shown in Figures 2.2.5 and 2.2.6, but on the "safe" side. Hence, one could use the available data to "over design" the hydrotreater, and then back off on the operating temperature or pressure, or both, after plant start-up, until the design hydrogen consumption is reached.

Finally the question arises as to what fraction of the sulfur would be removed by hydrotreatment, given a particular hydrogen consumption level or nitrogen removal percent. The available data are shown in Figures 2.2.7 and 2.2.8. There appears to be no particular pattern or correlation in the data. However, it seems safe to say that naphtha hydrotreating conditions which would consume 350 SCF/bbl of feed and reduce the nitrogen content to the range of 99.2%, as discussed above, would certainly remove 99+ wt. % of the sulfur.

Fig. 2.2.5. Nitrogen removal from raw SRC-II naphtha as affected by LHSV during hydrotreating (from FE-2315-47).

Fig. 2.2.6. Nitrogen removal from some coal-derived naphthas, as affected by LHSV (data from FE-2566-12, -25, and -27).

Fig. 2.2.7. Sulfur removal from some coal-derived naphthas during hydrotreating (data from FE-2315-47; FE-2566-12 and -27).

Fig. 2.2.8. Nitrogen and sulfur removal from some coal-derived naphthas during hydrotreatment (data from FE-2315-47; FE-2566-12 and -27).

2.2.2 Effect of hydrotreating on naphtha bioactivity

Battelle at Pacific Northwest Laboratories (PNL) performed chemical and biological characterization work on SRC-II Process gas oil both before and after hydrotreatment.⁶ The results of this work are summarized in Section 2.3. However, there appear to be no similar studies on SRC-II naphtha.

The bioactivity of coal-derived liquids is known to be due primarily to the presence of nitrogen compounds. The mild naphtha hydrotreating conditions suggested in the Phase 0 Demo Plant design (~350 SCF/bbl) along with the data in Figure 2.2.2 indicate that about 99.2% of the nitrogen should be removed from the naphtha by the proposed hydrotreating process. The extent to which the bioactivity of this liquid would be reduced by this extent of nitrogen removal is apparently not known at this time. This question should therefore be put to a qualified laboratory such as PNL for an opinion as to whether or not the testing of this naphtha and its hydrotreated product for bio-activity is warranted.

2.2.3 Effect of hydrotreating on naphtha storage stability

Lin, Anderson, and Wiser;⁹ Krishna and Ehsan;¹⁰ and Brown and Karn¹¹ have investigated the aging effects of coal-derived liquids in various oxygen-containing atmospheres. Viscosity and its change in time have been taken as the primary measure of the aging process. The most recent study, by Brown and Karn, was conducted on a fresh liquid produced by the Synthoil process. They concluded that the rate of product degradation was related to the initial viscosity of the sample, the storage temperature, and the concentration of oxygen in the gaseous environment over the samples. Some of the Brown and Karn data are reproduced in Figure 2.2.9. These data show that the rate of change of the liquid viscosity is lowest when stored under pure nitrogen, and that the viscosity can change sharply with time as the oxygen content of the atmosphere is increased.

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Fig. 2.2.9. Viscosity change with storage time of a freshly formed synthoil sample, showing effects of temperature and gaseous environment (from Brown and Karn).

No similar data are known for SRC-II-derived liquids. Further, there appear to be no data showing the extent to which the storage stability of coal-derived liquids is improved with increased levels of hydrotreatment. The designer is therefore unable at this time to specify quantitatively the level of hydrogen consumption required to stabilize an SRC-II naphtha, for example, for a given storage period. Unless data are forthcoming, it appears the extent to which the SRC-II naphtha should be hydrotreated on site will be up to the best judgment of the designers.

2.2.4 Physical and thermal property data

Data such as liquid heat capacities and latent heats of vaporization are required for precise firm-up of the design of an SRC-II naphtha hydrotreating process. Little if any thermal property data appear to be available for liquids of this type.

An approach to the design of a hydrotreating process, in the absence of such data, is to use the petroleum data and correlations. This was the approach taken by Econergy Associates¹² in their preliminary design of a complete coal liquid upgrading facility. According to their results, the overall plant heat balance closed to within 3.1%, with a maximum deviation of 7.0% for light gas oil hydrotreating. It is therefore concluded here that an SRC-II naphtha hydrotreating process designed on the basis of thermal property data taken from petroleum correlations should not be in serious error. However, the implementation of an experimental program designed to obtain these data for coal-derived liquids is recommended.

2.2.5 SRC-II naphtha hydrotreating assessment

The naphtha hydrotreating data are somewhat spotty and in some instances, nonexistent. However, based upon these data we make the following assessments:

- There are sufficient experimental data for the design of a naphtha hydrotreating unit which will remove an estimated 99.2 wt. % of the nitrogen if the hydrogen consumption is 350 SCF/bbl (Phase 0 design), but factors may have to be

introduced into the design to account for the sparcity of the data, its scatter, and certain extrapolations which will be necessary. As much as 1000-1100 SCF/bbl of hydrogen have been consumed during the hydrotreating of SRC-II naphtha, so additional hydrogen beyond that called for in the Phase 0 design could be added to the naphtha without exceeding the range of the available data. Major data weaknesses are in the areas of the limited range of process conditions (temperature, pressure, LHSV) and the scatter in the gas yield data. Insufficient data are available for process optimization, but the SRC-II data are supplemented considerably by the use of the EDS and the H-Coal naphtha hydrotreating data, particularly in the area of nitrogen removal as affected by hydrogen consumption.

- There appears to be no data on the bioactivity of SRC-II naphtha particularly as affected by hydrotreatment, so a study in this area would appear warranted, based on the judgment of PNL.
- Similarly, there appear to be no data showing the extent to which SRC-II naphtha is stabilized by level of hydrotreatment, so, unless data are forthcoming, the extent to which the naphtha should be hydrotreated on site will be a matter of the designer's judgment.
- Thermal property data from petroleum correlations can be used to estimate the energy requirements of a naphtha hydrotreating process, but data for coal-derived naphthas, particularly heat capacity and latent heat of vaporization data, would allow more precise estimation of the process fuel requirements.

2.3 SRC-II Gas Oil Hydrotreating

As pointed out in Section 2.1, the SRC-II Demo Plant gas oil is expected to contain an appreciable fraction of 800°F+ material, compared with that produced in the Pilot Plant. It is therefore of interest to see whether or not the available SRC-II gas oil hydrotreating data can be supplemented by the data from the hydro-treating of heavier coal-derived gas oils. Candidate oils for this purpose are the EDS and H-Coal process products. Inspections of these gas oils (Appendix C) indicate that these materials are somewhat heavier than the SRC-II gas oil (Appendix A), and that the EDS product in particular contains an appreciable fraction which boils above 800°F+. The data presented in the following section shows the extent to which the data from the different liquids are complementary.

Unfortunately, the UOP and Chevron hydrotreating results, particularly for the EDS product, are not all available at this time, so this aspect of this study can only be considered preliminary.

2.3.1 Gas oil hydrotreating data

The hydrotreating processing conditions employed by Chevron and UOP in the hydrotreating of some coal-derived gas oils are shown in Table 2.3.1. The Chevron experiments were at a nominal temperature of 750°F and a nominal total pressure of 2500 psig. The liquid hourly space velocity range was 0.43 to 1.01. The UOP processing conditions are reported in coded form.

A collection of pertinent gas oil hydrotreating results are shown in Figures 2.3.1 through 2.3.12. Complete inspections of the hydro-treated products are to be found in the original reports.^{4,14,15,16}

Figure 2.3.1 shows the response of the SRC-II and H-Coal gas oils to heteroatom removal, as a function of hydrogen consumption. These data suggest the SRC-II gas oil product may be a somewhat more difficult feed than the H-Coal product, so far as heteroatom removal is concerned. The nitrogen removal data are shown in Figures 2.3.2 and 2.3.3. Figure 2.3.3 shows that nitrogen removals in the range of 99.99% can be achieved, but with a hydrogen consumption of about 2600 SCF/bbl. Nitrogen removal as a function of reciprocal LHSV is shown in Figures 2.3.4 and 2.3.5. A pseudo first-order reaction model result would plot as a straight line on these graphs. Even though there is considerable scatter in the data, these results suggest such a simple model is not appropriate for this class of reactions (nitrogen removal).

A correlation between the nitrogen removal and sulfur removal data is shown in Figure 2.3.6. These results indicate that 85%+ of the sulfur has been removed from these liquids by the time only 30% of the nitrogen has been removed.

The change in the gas oil API gravity with hydrogen consumption is shown in Figures 2.3.7 and 2.3.8. The UOP data, Figure 2.3.8, suggests the SRC-II liquid may be more responsive to °API change than

Table 2.3.1. Coal-derived gas oil hydrotreating process conditions

Processing Condition or Property	Range	
	Chevron	UOP
Temperature, °F	746-752	
T-T(base), °C		-59 to 33
Total pressure, psig	2495-2501	
P-P(base), psig		750 to 1050
LHSV range	0.43-1.01	
LHSV/LHSV (base)		0.36 to 1.22
Recycle gas rate, SCF/bbl	12,776-16,368	Not reported
Hydrogen consumption, SCF/bbl	2549-4061	
wt. %		1.26-3.94
Nitrogen content of product, ppm	0.22-21	0.1-5970
Catalyst	ICR-106(Ni, W, Si, Al)	DCB (Group VI B and Group VIII elements)
Oils processed to date	SRC-II	SRC-II, H-Coal, EDS

Source: refs. 4, 14, 15 and 16.

Fig. 2.3.1. Heteroatom removal by hydrotreating of some coal-derived gas oils: UOP results (data from FE-2566-20, -25, and -35)

Fig. 2.3.2. Nitrogen removal by hydrotreating of some coal-derived gas oils: UOP results (data from FE-2566-20, -30, and 35).

Fig. 2.3.3. Nitrogen removal by hydrotreating of SRC-II gas oil (data from FE-2315-47).

Fig. 2.3.4. Nitrogen removal from SRC-II gas oil as a function of processing conditions during hydrotreating: Chevron results (data from FE-2315-47).

Fig. 2.3.5. Nitrogen removal from some coal-derived gas oils as a function of processing conditions during hydrotreating: UOP results (data from FE-2566-20, -30, and -35).

Fig. 2.3.6. Nitrogen-sulfur removal correlation for hydrotreating of some coal-derived gas oils (data from FE-2513-47; FE-2566-20, -30, and -35).

Fig. 2.3.7. Effect of hydrogen consumption on change of API gravity of some coal-derived gas oils during hydrotreating (data from FE-2315-47 and FE-2566-30).

Fig. 2.3.8. Effect of wt. % hydrogen consumption on change of API gravity of some coal-derived gas oils during hydrotreating (data from FE-2566-20, -30, and -35).

Fig. 2.3.9. Gas yield from some coal-derived gas oils during hydrotreating (data from FE-2315-47; FE-2566-20, -30, and -35).

Fig. 2.3.10. Light naphtha yield from hydrotreating of SRC-II gas oil (data from FE-2315-47).

Fig. 2.3.11. Light naphtha yield from some coal-derived gas oils during hydrotreating (data from FE-2566-20, -30, and -35).

Fig. 2.3.12. Heavy naphtha yield from SRC-II gas oil during hydrotreating (data from 2315-47).

the H-Coal product for a given value of hydrogen consumption, which may be due to the fact that the SRC-II sample was lighter at the outset (see Appendices A and C).

The gas, light and heavy naphtha yields due to gas oil hydrotreating are shown in Figures 2.3.9 through 2.3.12. The gas yield, Figure 2.3.9, tends to suggest a lower gas yield from SRC-II gas oil than the H-Coal product for a given change in API gravity, but this may be misleading, due to the fact that the SRC-II sample is of higher gravity than anticipated for the Demo Plant. Additional data or further analysis of these results would be useful for clarification of this point.

2.3.2 Effect of hydrotreating on SRC-II gas oil bioactivity

Battelle at Pacific Northwest Laboratories determined the Ames assay mutagenic response of SRC-II raw and moderately and severely hydrotreated gas oil samples supplied by UOP.⁶ Inspections of the samples, Table A.5, reveal little material in the 800°F+ boiling range. Hence, the extent to which the results apply to the Demo Plant gas oil is not known at this time.

The results of the PNL mutagenic response tests are shown in Table 2.3.2. The data in this table and in Table A.5 may be used to produce Table 2.3.3, which shows the percent reduction of the SRC-II gas oil mutagenicity with increasing percent removal of nitrogen. These data show that the mutagenicity of this oil can be reduced by 99%+ by severe hydrotreatment. The questions remaining at this time are:

- To what extent is the 800°F+ material expected in the Demo Plant product responsive to mutagenic reduction by hydrotreatment?
- How are the mutagenic factors remaining in the hydrotreated gas oil distributed with respect to boiling range? That is, are they more heavily concentrated in the light or the heavy gas oil fractions?
- To what extent should the mutagenicity of the Demo Plant product be reduced on site?

There appear to be little, if any, data or discussion in the literature which can be used to develop definitive answers to these questions.

Table 2.3.2. The effect of hydrotreating on the mutagenicity of SRC-II distillate blend gas oil

Material	Chemical fraction ^a	% of total wt.	Revertants (TA98) per µg fraction ^b	Weighted response ^c	Total revertants fractionated material	Total revertants unfractionated material
Feedstock	Basic	5.3	16.2	0.86	Σ = 1.20	2.54
	Acid-induced tar	5.9	2.7	0.16		
	Base-induced tar	1.4	5.6	0.08		
	Isooctane in-soluble tar	1.2	8.9	0.10		
Moderately hydrotreated	Basic	1.4	2.2	0.03	Σ = 0.03	None detected above background
	Acid-induced tar	0.06	0	0		
	Base-induced tar	0.03	0	0		
	Isooctane in-soluble tar	0.44	0	0		
Severely hydrotreated	Basic	0.99	0.79	0.01	Σ = 0.01	None detected above background
	Acid-induced tar	1.3	0	0		
	Base-induced tar	0.03	0	0		
	Isooctane in-soluble tar	0.44	0	0		

^a Fractions not shown were not active.

^b Zero values are below the limit of detection of approximately 0.05 rev/µg.

^c (Revertants per µg) x (percent total weight)/100.

Source: ref. 6.

Table 2.3.3. Relation between the SRC-II gas oil nitrogen content and its mutagenicity

Sample	Nitrogen content ppm	Nitrogen reduction wt. %	Reduction in revertants, %
Raw gas oil	9040	0	0
Moderate hydrotreatment	6000	34	97+
Severe hydrotreatment	1730	81	99+

Source: ref. 6.

2.3.3 Effect of hydrotreating on gas oil storage stability

There appear to be no data in the literature showing the extent to which hydrotreating stabilizes the SRC-II gas oil. For a brief discussion of the type of data and experiments which would be useful in this connection, see Section 2.2.3 above and also Brown and Karn.¹¹

2.3.4 Physical and thermal property data

Among the findings of the Econergy Associates study¹² was the result that the heat balance of a coal-derived light gas oil, such as from the SRC-II product hydrotreating process, might not close within 7% if designed by the use of thermal property correlations developed in the petroleum industry. Hence, additional experimental data on the thermal properties of coal-derived gas oils will be required before designers can proceed with confidence in the prediction of the fuel requirements for coal-derived gas oil hydrotreating plants.

2.3.5 SRC-II gas oil hydrotreating assessment

The fact that the SRC-II gas oil samples which were hydrotreated by Chevron and UOP were lighter than the corresponding product expected from the Demo Plant is a troublesome point when it comes to estimating hydrogen consumption, gas yields and processing conditions, should the decision be taken to hydrotreat the gas oil on site. However the experience to date with some similar gas oils, such as the EDS and H-Coal products, can be used to supplement the SRC-II data, especially with respect to gas yields and hydrogen consumption. Missing are the thermal data required for reasonably precise closure of the process heat balance, although data from the petroleum industry can be used for an approximate heat balance design.

There appears to be little guidance in the literature to assist the designer to establish the extent, if any, to which the gas oil should be hydrotreated on site. The PNL results indicate that the mutagenicity of SRC-II gas oil can be reduced 99%+ by severe hydrotreating, but do not suggest the extent to which the mutagenicity should be reduced.

No data could be found which would allow a quantitative estimate of the degree of hydrotreating required to provide a given level of storage stability to the product.

The available hydrotreating data show that these coal-derived gas oils can be upgraded by the use of commercial technology. However, the extent to which it is useful to hydrotreat the SRC-II Demo Plant gas oil on site, if any, will be determined by the judgment of the designer unless additional data, particularly with respect to storage stability and allowable mutagenicity levels in these products, are forthcoming.

2.4. SRC-II Whole Liquid Hydrotreating

In addition to the hydrotreatment of the SRC-II naphtha and gas oil fractions, Chevron also hydrotreated the SRC-II whole liquid product. When viewing these results, keep in mind that this whole liquid sample contained very little 800°F+ material compared with that anticipated from the Demo Plant. In contrast to gas oil hydrotreating, summarized in the preceding section, no comparable whole liquid product hydrotreating data could be found for heavier liquids, such as the EDS product, for supplementing the Chevron SRC-II whole liquid product data.

2.4.1 Whole liquid hydrotreating data

The range in the hydrotreating processing conditions used by Chevron are shown in Table 2.4.1. These data show that hydrotreating nominal temperatures and pressures in the range of 750-775°F and 2000-2500 psi, respectively, can remove up to 99.999% of the nitrogen and up to 99.89% of the sulfur from this liquid. Space velocities required are in the range of 0.5 - 1.50.

The whole liquid hydrotreating data of Chevron are shown in Figures 2.4.1 through 2.4.12. The heteroatom removal data correlate well with chemical hydrogen consumption, as can be seen in Figures 2.4.1 through 2.4.3. Oxygen removal can be predicted, within limits, from nitrogen removal requirements, Figure 2.4.4. Figure 2.4.5 shows

Table 2.4.1. SRC-II whole liquid product hydrotreating
processing conditions

Processing condition or property	Range or Value
Temperature, °F	749-776
Pressure: Total, psia	1995-2506
Hydrogen, psia	1700-2300
LHSV	0.49-1.52
Recycle gas rate, SCF/bbl	7,504-15,811
Hydrogen consumption, SCF/bbl	
Gross	1,741-3,301
Chemical	1,651-3,109
Nitrogen content of the product, ppm	0.1-560
Nitrogen removal, wt. %	94.28-99.999
Sulfur content of the product, ppm	3-100
Sulfur removal, wt. %	99.27-99.89
Catalysts	ICR-106 (Ni, W, Si, Al) ICR-113 (Ni, Mo, Si, Al)

Source: ref. 4.

Fig. 2.4.1. Heteroatom removal from SRC-II whole liquid product by hydrotreating (data from FE-2315-47).

Fig. 2.4.2. Nitrogen removal from SRC-II whole liquid product by hydrotreating (data from FE-2315-47).

Fig. 2.4.3. Sulfur removal from SRC-II whole liquid product by hydrotreating (data from FE-2315-47).

Fig. 2.4.4. Nitrogen-oxygen removal correlation for SRC-II whole liquid product hydrotreating (data from FE-2315-47).

Fig. 2.4.5. Nitrogen-sulfur removal correlation for SRC-II whole liquid product hydrotreating (data from FE-2315-47).

Fig. 2.4.6. Hydrogen consumption as a function of processing conditions for SRC-II whole liquid product hydrotreating (data from FE-2315-47).

Fig. 2.4.7. Nitrogen removal as a function of processing conditions for SRC-II whole liquid product hydrotreating (data from FE-2315-47).

Fig. 2.4.8. Effect of hydrogen consumption on change in API gravity of SRC-II whole liquid product during hydrotreating (data from FE-2315-47).

Fig. 2.4.10. Light naphtha yield from SRC-II whole liquid product during hydrotreating (data from FE-2315-47).

Fig. 2.4.11. Heavy naphtha yield from SRC-II whole liquid product during hydrotreating (data from FE-2315-47).

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Fig. 2.4.12. Reduction in aromatics in SRC-II whole liquid products during hydrotreating (from FE-2315-47).

time 99.5%+ of the sulfur has been removed from this liquid by the time 94% of the nitrogen has been removed.

The processing conditions required to achieve a given hydrogen consumption and nitrogen removal are shown in Figures 2.4.6 and 2.4.7, respectively. The data are scattered as is usual in these experiments, but can be used to establish values for the temperature, pressure, and LHSV required for a given level of nitrogen removal in the range of about 94 to 99.999 wt. %.

The change in API gravity of the liquid correlates reasonably well with hydrogen consumption, Figure 2.4.8. The gas yield for a given change in API gravity can be estimated from Figure 2.4.9. Naphtha yields are shown in Figures 2.4.10 and 2.4.11. The volume percent reduction in the whole liquid aromatics is shown in Figure 2.4.12, as a function of the chemical hydrogen consumption.

Although these data appear to exhibit a fair amount of scatter, the scatter is probably no worse than usual in a newly developing technology or in the application of an existing technology to a new area. Factors which can be reduced as additional data becomes available can be introduced in the design process to account for the data scatter.

2.4.2 Effect of hydrotreating on SRC-II whole liquid product bioactivity

No data could be found on the mutagenic response of the SRC-II whole liquid product. However, experts in this area might be able to make an assessment of this response on the basis of the SRC-II gas oil mutagenic response data, in view of the fact that the nitrogen content of the naphtha fraction of the product should be essentially depleted under the relatively severe hydrotreating conditions shown in Table 2.4.1, used for the whole liquid product.

2.4.3 Effect of hydrotreating on SRC-II whole liquid product storage stability

Again, data were not found which would indicate the extent to which this liquid is stabilized in time as a function of hydrogen

added during hydrotreatment. There is apparently no quantitative way to establish the level of hydrogen addition required to produce a given extent of stabilization.

2.4.4 Physical and thermal property data

The situation here appears to be the same as for the other coal-derived liquid fractions. Based on the Econergy Associates study,¹² we conclude that the hydrotreating heat balances can be off as much as 7%, based upon the use of thermal property correlations from the petroleum industry.

2.4.5 SRC-II whole liquid hydrotreating assessment

The SRC-II whole liquid product can be hydrotreated by commercial technology up to chemical hydrogen consumptions of about 3,100 SCF/bbl. This level of hydrotreatment can remove as much as 99.999% of the nitrogen and 99.89% of the sulfur, from the Pilot Plant SRC-II samples. As these samples were somewhat lighter than the liquid expected from the Demo Plant, exact prediction of the yields, particularly the gas yield, from these data cannot be expected. The heteroatom removal data provide reasonably good correlation with hydrogen consumption, as well as the change in API gravity. The lack of thermal property data may introduce errors in the process heat balances by as much as about 7%, according to a previous study.¹² Mutagenic response test data for the whole liquid were not found, but it should be possible to make at least approximate estimates based on the PNL data on the SRC-II gas oil.

Stability data as a function of hydrogen added during hydrotreatment are not available. There appear to be no quantitative criteria which a designer can use to establish the extent to which this material should be hydrotreated, if at all, on site. The most troublesome point is the fact that the Demo Plant product should be heavier than the Pilot Plant product, the material upon which all previous studies have been made, due to the presence of an appreciable fraction of 800°F+ material. In spite of these shortcomings, however, a hydro-

treating process for the whole liquid product could probably be designed for the Demo Plant, recognizing that the gas yields and thermal balances may contain a moderate amount of error. The hydro-treating experience on the heavier gas oils, such as the EDS and the H-Coal products can supplement the SRC-II data through appropriate correlations.

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APPENDIX A

Inspections of the SRC-II Pilot Plant Coal and Product
and Projected Data for the Demo Plant

Table A.1. Comparison of Powhatan No. 5 and the Demo Plant
coals, weight percent MF basis

Component	Demo Plant Design Basis Panhandle Feed Coal Composition			Composite of Fort Lewis Pilot Plant Powhatan No. 5 Coal Shipment
	Low Reactivity	Design Reactivity	High Reactivity	
Carbon	73.93	70.49	67.50	72.25
Hydrogen	5.11	4.88	4.69	4.94
Nitrogen	1.22	1.12	1.32	0.83
Oxygen	7.54	7.87	8.27	6.68
Sulfur				
Pyrites	1.8	2.0	2.2	1.91
Organic	1.3	1.54	1.9	1.70
Sulfate	<u>0.07</u>	<u>0.05</u>	<u>0.05</u>	0.05
TOTAL	3.17	3.59	4.15	3.66
Chlorine	0.03	0.05	0.07	0.07
Ash	9.0	12.0	14.0	11.57
(Iron Content)	<u>(1.58)</u>	<u>(1.75)</u>	<u>(1.93)</u>	<u>(1.92)</u>
TOTAL	100.00	100.00	100.00	100.00

Source: ref. 6.

Table A.2. Properties of SRC-II process products received by Chevron

Description Chevron identification	Whole SRC-II WOW 3666	SRC-II naphtha WOW 3750	400°F+ SRC-II WOW 3751
Wt. % of whole SRC-II	100	29	71
Inspections			
Gravity, °API	18.6	36.2	10.7
Aniline point, °F	<30	56.4	
Sulfur, wt. %	0.29	0.26	0.25
Total nitrogen, wt. %	0.85	0.42	0.99
Basic nitrogen, wt. %	0.7		0.82
Oxygen, wt. %	3.79	3.51	3.63
Carbon, wt. %	84.61	83.66	85.86
Hydrogen, wt. %	10.46	12.15	9.27
Hydrogen/carbon atom ratio	1.47	1.73	1.29
Chloride, ppm	50	18/20	77/62/81
Pour point, °F	Below -80		-50
Group type, LV %		Low mass/ FIAM ^a	
Paraffins	b	23	b
Naphthenes	b	38	b
Olefins	b	5	b
Aromatics	b	34	
Ramsbottom carbon, wt. %	0.70		0.81
Hot heptane asphaltenes, ppm	468		2200
Benzene insolubles, wt. %	<0.03		<0.03
Refractive index (80°C)	1.5073		1.5340
Ash, wt. %	0.004		0.010
Molecular weight	132	118	160
Bromine number	70	49/52	69
Viscosity, cSt			
At 100°F	2.196	0.8933	4.213
At 130°F	1.1617		
At 210°F			1.238
ASTM distillation, °F	D 86/D 1160	D 86	D 1160
St/5	154/217	151/179	393/413
10/30	281/382	197/249	427/448
50	438	290	471
70/90	484/597	332/366	506/590
95/EP	699/850	380/393	662/847
% Overhead (excl. trap)	98	98	99
% In flask	0	1	1
% Trap	2	1	0
TBP distillation, °F (simulated by chromatography)			
St/5	56/189	53/153	233/366
10/30	241/379	173/235	382/430
50	424	291	477
70/90	473/562	345/395	527/639
95/99	642/820	408/442	707/849
Maleic diene value, cg I ₂ /g	30.7	3.28	37.3
Water by distillation, vol %	0.6		
Acid neutralization, mg KOH/g	0.2		

^aParaffins and naphthenes were determined by mass spectrometry; aromatics and olefins by FIAM (fluorescent indicator analytical method). Reliability of these analyses for this type of sample is uncertain.

^bSee Table 6.

Source: ref. 4.

Table A.3. Composition of whole SRC-II process product and the 400°F+ SRC-II process product by 22-component mass spectrometric analysis, as determined by Chevron^a

Type Chevron identification	Whole SRC-II WOW 3666	400°F+ SRC-II WOW 3751
Composition, LV %		
Saturates		
C(N)H(2N+2), Paraffins	7.2	4.6
C(N)H(2N), Monocycloparaffins	6.9	2.3
C(N)H(2N-2), Dicycloparaffins	2.2	1.8
C(N)H(2N-4), Tricycloparaffins	4.1	3.2
Total Saturates	20.3	12.0
Monoaromatics		
C(N)H(2N-6), Alkylbenzenes	17.9	13.3
C(N)H(2N-8), Benzocycloparaffins	13.9	15.3
C(N)H(2N-10), Bendodicycloparaffins	2.0	5.2
Diaromatics		
C(N)H(2N-12), Naphthalenes	27.8	33.2
C(N)H(2N-14)	5.1	6.8
C(N)H(2N-16)	7.0	8.1
Triaromatics		
C(N)H(2N-18)	3.0	4.0
C(N)H(2N-22)	0.9	0.6
Tetraaromatics		
C(N)H(2N-24)	0.2	0.1
Total Aromatics	77.7	86.6
Sulfur Compounds		
C(N)H(2N-4)S, Thiophenes	0.3	0.4
C(N)H(2N-10)S, Benzothiophenes	1.3	0.8
C(N)H(2N-16)S, Dibenzothiophenes	0.4	0.3
Total Sulfur Compounds	1.9	1.4
Calculated, Wt. % Composition		
Hydrogen (Corrected for N, S, O)	9.7	9.4
Carbon (Corrected for N, S, O)	85.4	85.7

^aImportant note: Because the large number of heteromolecules present are not included in this analysis (except for thiophenes), results should not be regarded as quantitative. However, this analysis does serve to indicate the types of hydrocarbons present with the following qualifications. Samples types correctly analyzed are: (1) C₁₂ to C₃₆, 250°F hydrocarbons; (2) olefin-free hydrocarbons; and (3) less than 5% oxygen, nitrogen, or sulfur compounds. Only listed types are considered; all others are ignored. Factors which contribute to an incorrect analysis are as follows: (1) large amounts of a single hydrocarbon; (2) unusual distribution of compounds; and (3) thermally unstable materials.

Source: ref. 4.

Table A.4. Metals in SRC-II process product blend, in sample received by Chevron

	Analysis of blend ^a	Calculated from analyses of components
Inspections		
Metals by emission spec., ppm		
Aluminum	4	5.4
Boron	0.1	0.2
Calcium	0.4	1.2
Chromium	0.8	0.7
Iron	7.5	6.5
Magnesium	0.3	0.3
Manganese	0.2	
Sodium	0.2	2
Nickel	0.2	0.2
Silicon	2.7	3.2
Tin	0.3	
Titanium	0.4	5
Vanadium	0.1	0.2
Zinc	0.3	
Other metals analysis		
Arsenic, ppb	14	33

^aAverage of two analyses. Relatively poor reproducibility suggests metals are mainly due to entrained sediment or fines rather than soluble compounds.

Source: ref. 4.

Table A.5. UOP inspections of raw and hydrotreated SRC-II distillate blend

Parameter	Feed		Mildly ^a hydrotreated		Severely ^b hydrotreated	
	Drum 1	Drum 2	WLP ^c	400°F+	WLP ^c	400°F+
UOP Sample No. ^d	<u>3777-6</u>	<u>3777-7</u>	<u>3777-21</u>	<u>3777-34</u>	<u>3777-28</u>	<u>3777-41</u>
API @ 60°F	9.7	10.1	17.0	13.6	23.4	17.7
Specific gravity @ 60°F	1.0021	0.9993	0.9529	0.9752	0.9135	0.9484
Weight %						
Hydrogen	8.68	8.72	10.38	9.98	11.18	10.59
Carbon	85.83	86.13	86.91	86.84	87.17	84.86
Oxygen	2.02	1.73	1.08	0.98	0.67	0.83
Weight ppm						
Sulfur	3300	3815	129	106	98	109
Nitrogen	8100	9040	5510	6000	1900	1730
FIA ^e , vol %						
Color band ^f		36.1	19.7	19.4	6.3	
A	90.1	54.5	59.1	69.9	50.8	73.9
P&N	9.9	9.4	21.2	10.7	42.9	26.1
ASTM D 1160 distillation, LV %, °F						
IPB/5	386/407	335/395	280/350	418/428	234/302	408/440
10/30	417/445	410/444	374/422	436/462	331/400	448/463
50	486	472	460	487	446	490
70/90	535/645	512/656	505/611	539/641	490/575	536/625
95/EP	711/799	705/810	687/779	691/790	628/724	668/748
% over	99.0	99.0	99.0	99.0	99.0	99.0
TBP distillation, LV %, °F ^g						
IRP	347	292	241	388	195	368
10	404	397	359	424	315	438
30	438	438	415	456	392	457

^aFeed was Drum No. 1.^bFeed was Drum No. 2.^cWhole liquid product.^dUnderlined samples were characterized by PNL.^eFluorescent indicator analysis for aromatics (A), paraffins (P), and naphthenes (N).^fIndicates highly polar compounds.^gTrue boiling points based on standard correction of ASIM-D1160 distillation data. This correction method assumes that the D-1160 yields are the same as the TBP yields from the 50% point to the end point.

Source: refs. 6, 16.

Table A.6. UOP high resolution mass spectral characterization
of SRC-II gas oil drum no. 1 (3777-6)

Sample analysis summary				
	Wt. %	Avg. C No.	Avg. MW	Avg. z No.
Compound type C_nH_{2n-z}				
Saturated hydrocarbons (P + N)	8.4	15.5	216	0.7
Aromatic hydrocarbons	53.8	14.3	186	14.1
Aromatic nitrogens	7.0	11.7	167	10.8
Aromatic oxygens	27.6	10.4	153	8.9
Aromatic sulfurs	1.2	13.7	209	14.6
Aromatic dioxygenates	1.7	13.1	203	13.5
Aromatic trioxxygenates	0.3	12.5	212	10.8
Elemental analysis by MS				
	Wt. %			
Atomic carbon	87.72			
Atomic hydrogen	8.29			
Atomic nitrogen	0.59			
Atomic oxygen	3.22			
Atomic sulfur	0.18			
	100.00			

Source: ref. 16.

Table A.7. Phase 0 design SRC-II Demo Plant
vacuum flash overhead components

Component	Flow rate, <u>M</u> /hr	Mole % of total
C ₄	0.2	0.07
C ₅	0.1	0.03
150-350°F	1.8	0.61
350-600°F	111.8	38.01
600-900°F	148.6	50.53
900°F+	31.6	10.74
Total	294.1	99.99

Source: ref. 1.

ORNL DWG 81-23

Fig. A.1. Comparison of SRC-II process boiling curve of Pilot Plant sample received by Chevron with projected Demo Plant product boiling curve (from FE-2315-47).

ORNL DWG 81-8901

(a) Wt. % yields based on nominal C₅ IBP of 90°F.

(b) Vol. % yields normalized to 400°F at demo plant 33% point.

Fig. A.2. Comparison of distillation curves for projected SRC-II Demo Plant product and UOP hydrotreating sample from the Pilot Plant.

APPENDIX B

Hydrotreating Data for SRC-II Hydrotreated Naphtha

ORNL DWG 81-12

Fig. B.1. Hydrotreating of hydrotreated SRC-II naphtha: nitrogen removal as a function of LHSV (from FE-2315-47).

APPENDIX C

Inspections of Raw EDS and H-Coal Process Gas Oils

Table C.1. Inspections of raw EDS liquid product received by UOP

Drum No.	2	3	1
Sample No.	3532-1	3532-3	3532-2
°API @ 60°F	7.6	7.6	8.0
Sp. Gr. @ 60°F	1.0173	1.0173	1.0143
<hr/>			
Distillation, ASTM D-1160			
IBP, °F	406	400	408
5%	428	424	436
10%	435	436	443
20%	452	460	460
30%	469	490	490
40%	511	521	531
50%	561	567	574
60%	619	631	630
70%	720	730	730
80%	825	822	842
90%	908	905	915
95%	955		
EP	978		
% Over	96.0	90.0	90.0
% Bottoms	4.0	10.0	10.0
Hydrogen, wt. %	8.73	8.99	8.87
Carbon, wt. %	86.00	87.61	86.45
Sulfur, wt. %	6500	6510	6710
Nitrogen, wt. %	4580	5050	4950
Oxygen, wt. %	9100	19800	28000
Con. carbon, wt. %	4.02	3.55	3.84
Heptane insolubles, wt. %	8.11	8.77	8.17
Stm. jet gum, mg/100 ml	11501	6356	7479
882°F - liquid, vol %	86.7		
Heptane insolubles, wt. %	1.75		
Benzene insolubles, wt. %	0.01		

Source: ref. 16.

Table C.2. Inspections of raw EDS liquid product received by UOP

Drum No.	1
Sample No.	3532-5
°API @ 60°F	3.5
Sp. Gr. @ 60°F	1.0481
<hr/>	
Distillation, ASTM D-1160	
IBP, °F	403
5%	447
10%	454
20%	471
30%	511
40%	563
50%	620
60%	690
70%	778
80%	878
90%	(85%) 930
95%	
EP	
% Over	85.0
% Bottoms	15.0
Hydrogen, wt. %	8.66
Carbon, wt. %	89.22
Sulfur, wt. ppm	4900
Nitrogen, wt. ppm	7130
Oxygen, wt. ppm	19300
Con. carbon, wt. %	6.66
Heptane insolubles, wt. %	6.45
Stm. jet gum, mg/100 ml	10063

Source: ref. 15.

Table C.3. Inspections of H-Coal atmospheric still bottoms received by UOP

HRI Sample No.	As Received		After Flash Distillation	
	LO-585	LO-586		
Sample No.	37-1118	37-1117	3531-10	3531-11
°API @ 60°F	7.4	12.1	12.9	8.9
Sp. Gr. @ 60°F	1.0187	0.9854	0.9799	1.0078
Distillation, ASTM D-1160				
IRP, °F	412	410	418	415
5%	463	467	467	462
10%	485	474	480	490
20%	509	495	499	518
30%	530	519	517	538
40%	558	545	536	553
50%	580	566	557	571
60%	603	591	578	590
70%	631	621	603	615
80%	661	666	639	645
90%	715	710	690	689
95%	768	772	727	715
EP	857	855	770	769
% Over	99.0	99.0	99.0	99.0
% Bottoms	1.0	1.0	1.0	1.0
Hydrogen, wt. %			9.57	9.14
Carbon, wt. %			89.28	88.98
Sulfur, wt. ppm	951	690	500	800
Nitrogen, wt. ppm	3534	2966	4180	4100
Oxygen, wt. ppm	5006	5480	5200	5200
Con. carbon, wt. %	0.41	0.49	0.01	<0.01
Heptane insoluble, wt. %	0.37	0.53	0.11	0.05
FIA, vol %				
A	90.9	83.7	84.0	90.2
O	0.0	0.0	0.0	0.0
F&N	9.1	16.3	16.0	9.8
Stm. jet gum, mg/100 ml	620	933	4.7	37

Source: ref. 14.

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