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Reactor Technology

REVIEW OF HYDRAULIC PROBLEMS ON THE
BONUS REACTOR PRESSURE CONTROL SYSTEM

MASTER

I. G. Speas
J. S. Cobb
S. E. Groothuis

Y-12 PLANT

Oak Ridge, Tennessee

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Oak Ridge, Tennessee
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Nuclear Division
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Oak Ridge, Tennessee

Attention: Dr. C. E. Larson, President

Subject: REVIEW OF HYDRAULIC PROBLEMS ON THE BONUS REACTOR PRESSURE
CONTROL SYSTEM

Gentlemen:

In accordance with the request in my letter to you of May 21, 1965, six copies of revised report (dated June 14, 1965), subject as above, have been delivered to us by Mr. Speas of Y-12.

We believe that the report as revised is now ready for distribution. Please prepare sufficient copies and distribute in accordance with UC-80, "Reactor Technology", AEC Standard Distribution Lists, TID-4500, 43rd edition, July 1, 1965. In addition to the standard distribution, please mail directly one copy each to the following individuals:

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Oilgear Company
160 Bear Hill Road
Waltham, Massachusetts

Mr. Norman Gibbs, Hydraulics Engineer
Oilgear Company
1560 West Pierce Street
Milwaukee 4, Wisconsin

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In the event you are aware of any manufacturer of hydraulic equipment or supplier of phosphate-ester hydraulic fluids that would be interested in the report, it would be appropriate to mail copies directly to them.

Very truly yours,

CC: R. L. Shannon
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Manager
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Reactor Technology
TID-4500 (43 Edition)

UNION CARBIDE CORPORATION
Nuclear Division

Y-12 PLANT

Contract W-7405-eng-26
With the US Atomic Energy Commission

REVIEW OF HYDRAULIC PROBLEMS ON THE BONUS
REACTOR PRESSURE CONTROL SYSTEM

I. G. Speas
J. S. Cobb
S. E. Groothuis

Oak Ridge, Tennessee
June 14, 1965

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Ackerson, R. D.	(AFSC)
Bailey, E. W.	
Ballenger, H. F.	(2)
Barnett, H. L.	
Bell, B. B.	
Burkhart, L. E.	
Center, C. E.	(ORGDP)
Cobb, J. S.	(5)
Cowen, D. D.	(ORNL)
Ebert, J. W.	
Evans, G. W.	
Fortenbery, M. J.	
Gibbs, Norman	(Oilgear-Milwaukee)
Gritzner, V. B.	
Groothuis, S. E.	(5)
Harding, J. E.	
Harwell, W. L.	(ORGDP)(5)
Hemphill, L. F.	
Hill, D. G.	
Huber, A. P.	(ORGDP)
Jennings, D. A.	
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Lewis, F. O.	(ORGDP)
Little, J. C.	
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Patton, F. S.	
Perry, A. E.	
Porter, Tom	(Oilgear-Waltham)
Rader, D. H.	
Speas, I. G.	(10)
Stroud, D. D.	
Trotter, T. C.	
Waters, J. L.	
Whitson, W. K.	
Winkel, R. A.	(Paducah)
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ABSTRACT

Y-12 Engineering personnel made a study of the BONUS reactor pressure control system to determine the cause of the malfunction of the hydraulic controls on the major steam valves. It was concluded that the principal contributor to the valve trouble was the high moisture content of the phosphate-ester hydraulic fluid. This excessive moisture reacted with the hydraulic fluid to produce an acid which, in turn, corroded the steel components of the system and produced fine abrasive particles that eroded the servo metering surfaces.

Modifications were recommended that should alleviate the current problems.

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INTRODUCTION

In March 1965, the services of Y-12 Engineering personnel were requested by the Atomic Energy Commission for a review of the BONUS Reactor pressure control system. Problems arising from the malfunction of hydraulic controls on major steam valves had been experienced for several months. A trip was made to the BONUS Site to discuss, inspect, and observe the system in service.

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SUMMARY

From samples taken at the BONUS Site and analyzed at Y-12, the principal contributor to the valve control trouble was concluded to be high moisture content in the phosphate-ester hydraulic fluid (~ 2000 ppm compared to a recommended maximum of 100 ppm). Water hydrolyzed the ester yielding phosphoric acid. The acid, in turn, corroded the steel components of the system which produced abrasive corrosion products in the form of fine particles that eroded the servo valve metering surfaces (Figure 1, Page 12).

Excessive leakage at the high pressure drop ($\Delta P = 3000$ psi) through the scored valves resulted in overheating the hydraulic fluid which led to its partial thermal decomposition and a buildup of sludge in the valves. The situation was further aggravated by the piping arrangement which does not allow the capacity of the heat exchangers to be fully effective in cooling the hydraulic fluid.

A Y-12 designed portable filter-dryer unit was shipped to the BONUS Site and installed. In four days it reduced the water content to 80 ppm.

It was recommended that:

1. The Y-12 filter-dryer unit be retained in service on a permanent basis. (Cost - \$4000.)
2. The return lines be rerouted from the servo valves so that the full flow passes through the existing heat exchangers (Figure 4, Page 16). (Cost - \$400.)
3. Supplemental valving be installed as shown in Figure 5, Page 17, to enable the hydraulic pumps and accumulators to be removed from service for maintenance without interrupting the reactor operation. (Cost - \$1000.)

It is concluded that the system is basically sound and well designed with the exceptions noted. These troubles arose from the phosphate-ester fluid which is employed because of its fire-resistant properties. The use of this substance for hydraulic applications is a relatively recent innovation and information on the very special precautions which must be exercised is not generally available.

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DISCUSSION OF THE STUDY

BACKGROUND

Initial tests of the turbine bypass valve and the minimum-flow bypass valve in October 1964 appeared satisfactory. However, sluggish operation of the minimum-flow bypass valve soon developed. The trouble was attributed to foreign particles in the hydraulic fluid. The two-stage spool-type servo valves were replaced with single-stage swing-plate valves. Inspection of the units removed showed excessive wear at the metering edges of the moving pilots, and a brown varnish-like coating was observed at the leakage points between the servo moving pilot and the fixed port parts of the valves.

The phosphate-ester hydraulic fluid in the sump (Houghto-Safe 1120) was circulated through filters, dumped, and replaced with new fluid. Appendix A lists the physical properties of this liquid.

In December, the turbine bypass valve failed to open promptly on demand. Examination revealed a brown varnish buildup in the valve.

A one-micron Millipore filter unit and pump were installed in a bypass loop at the hydraulic fluid sump.

The leakage through the servo valves became excessive and they were replaced with valves of the same design. Again, a brown varnish buildup and erosion of valve parts were observed.

FIELD OBSERVATIONS AT THE REACTOR SITE

Inspection of the reactor control system by Y-12 personnel revealed several irregularities.

Eroded Servo Valve

Excessive erosion was evident on the body lands of a single-stage swing-plate valve which had been removed from service (Figure 1). This erosion was apparently caused by a large amount of abrasive contaminants in the hydraulic fluid circulating system. The contaminants are the result of corrosion of the steel sump by phosphoric acid. Phosphoric acid arises from excessive moisture in the phosphate-ester fluid.

Foreign Deposits in the Servo Valve

A brown deposit buildup was observed at the metering port ends of the sliding plate. A flow test while the valve was in service had indicated a leakage rate of five

Figure 1. CROSS SECTION OF A SERVO VALVE.

gallons per minute. Acceptable normal leakage is about 0.5 gpm. The high flow with a pressure drop ($\Delta P = 3000$ psi) generated excessive temperatures ($> 200^{\circ}$ F) resulting in decomposition of the phosphate-ester fluid. Since there was no heat exchanger in the return line to the sump, the system temperature progressively increased to a high level.

Rust in the Hydraulic Fluid Sump

Inspection of the inside of the 100-gallon steel tank revealed rust deposits on the top and sides above the liquid level. There was also evidence of contaminants on the surface of the fluid.

Inadequate Cooling

The existing cooling system consists of a heat exchanger in the sump line from each unloading valve, and a self-actuated temperature control valve in the cooling water line with its temperature sensing bulb in the hydraulic fluid inlet. The hydraulic fluid return lines from the turbine bypass valve and the minimum flow bypass valve are routed directly to the fluid sump with no provision for cooling.

Inadequate Valving for Maintaining Hydraulic Pumps and Accumulators

The existing fluid system does not have adequate valving to take pumps and accumulators out of service and keep the system active.

Common Time Delay Relay for Two Circuits

The time delay relay in the minimum-flow bypass valve circuit is common to both the servo system and the solenoid valve sequencing circuits so that a failure of the delay relay could prevent the trip operation.

ACTION TAKEN TO DATE

Three steps have been initiated by the field party to improve the purity of the hydraulic fluid system:

1. Samples of new phosphate-ester fluid and used fluid from the system were taken at the BONUS Site and shipped to the Y-12 Plant Analytical Laboratory for analysis (see Appendix B). The analyses revealed an excessive moisture content (2010 ppm) and high iron contamination of the used fluid.
2. The fluid sump was drained, cleaned, and filled with new phosphate-ester fluid containing a moisture content of 1180 ppm.
3. A portable filter-dryer unit was shipped from the Y-12 Plant and temporarily installed with hose in the hydraulic fluid system (see Figures 2 and 3). Analysis of the hydraulic fluid after 87 hours of circulation through the unit showed that the moisture content had been reduced to an acceptable level (80 ppm). Experience at Y-12 indicates that the Molecular-Sieve dryer can further reduce the moisture content to as low as 10 ppm. Appendix C gives the correct drying procedure for new filter tubes and a Molecular Sieve bead refill for the filter unit.

GENERAL CONCLUSIONS

After overall evaluation of the pressure control system, the following conclusions were reached:

1. The turbine trip valve (TTV), turbine admission valve (TAV), and their associated hydraulic systems are an integral part of the General Electric turbine. The governor motor drive has been modified to make a positioner for the TAV which is compatible with the steam pressure controller. The TAV positioner was found to be satisfactory under conditions of no load and can be expected to work in the steam pressure control loop.

Figure 2. EXISTING HYDRAULIC PUMPING SYSTEM.

Figure 3. TEMPORARY INSTALLATION OF THE FILTER-DRYER UNIT.

2. The turbine bypass valve (TBV) and minimum flow bypass valve (MFBV) are hydraulic cylinder actuated valves with servo positioners. They have a common hydraulic power supply.

The existing hydraulic components are satisfactory for the type of service required. Corrosion of the steel components in the system should be controlled by maintaining a low moisture content (< 100 ppm) with the Molecular-Sievedryer. The single-stage servo valves which were substituted for the original two-stage type are the best known valves for this application. The existing Denison fixed-displacement, 1800-rpm, 9.5-gpm, hydraulic pumps and automatic pump startup system are adequate. The Aircraft Porous Media 10-micron filters in the pump discharge header and at each servo valve are adequate in combination with the filter-dryer unit.

3. The steam pressure control loop consists of Foxboro electronic pressure transmitters and three-mode controllers which drive the TAV, TBV, and MFBV positioners described in Points 1 and 2. The TAV loop will be set to the desired steam pressure, the MFBV will be set approximately 10 psi higher, and the TBV about 20 psi higher so that each will take over control in sequence if the preceding system cannot take the required steam flow. The controllers are equipped with circuits to prevent "reset wind up" when they are not controlling. This control system is adequate.

RECOMMENDATIONS

In consideration of the foregoing factors, the following recommendations appear to be in order:

1. The filter-dryer unit should be permanently installed in the hydraulic fluid system. Appendix D gives the proper steps for operating this unit.
2. The return fluid from the servo valves should be routed through the existing heat exchangers to be cooled prior to mixing with the sump fluid (see Figure 4). This action will increase the effectiveness of the heat exchangers under conditions of high servo valve leakage. The proposed new hand-operated three-way valve and check valves will put both heat exchangers in service normally and will allow either of them to be valved out of service without a shutdown.
3. The ranges of the two heat-exchanger temperature controllers should be changed from the present 125 - 165° F and 115 - 180° F to 90 - 130° F, and each set at 110° F. The temperature sensing bulb should be relocated to the fluid tank. A new high-temperature switch which can be set at 120° F should be installed. The existing sump fluid level switch, pump output pressure switch, and new high-temperature switch should all be annunciated in the control room.

- ① Three-Way Valve - One inch, tee drilling (Republic 8031B-1HS2 or similar), for phosphate-ester hydraulic fluid
- ② Temperature Control Valve - Range 90 - 130° F
- ③ Check Valve - 1 1/2-Inches
- ④ Check Valve - 3/4-Inch
- ⑤ Temperature Switch (TS) - Range 50 - 150° F

Figure 4. PROPOSED ALTERATIONS TO THE HYDRAULIC FLUID COOLING SYSTEM.

4. The valving shown in Figure 5 should be installed to enable both accumulators to be in service simultaneously and allow any pump or accumulator to be valved out for maintenance without a shutdown.
5. A second time-delay relay should be installed in the minimum-flow bypass valve trip circuit.
6. A hydraulic fluid laboratory analysis program should be set up at the BONUS Plant to periodically check for water content and neutralization number. Until

Figure 5. PROPOSED ALTERATIONS TO THE HYDRAULIC FLUID PRESSURE SYSTEM.

this program is active it is recommended that fluid samples be sent every two weeks to the Y-12 Plant for analysis. Appendix E lists the recommended equipment to determine moisture content. Appendix F covers the procedure for determining the neutralization number.

7. The existing Millipore 0.45-micron membrane filter should remain in the hydraulic fluid system to be used periodically to sample the fluid system for evidence of contamination. However, if moisture and temperature are kept within the recommended limits (< 100 ppm moisture and < 115° F), there should be no contamination.

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PHYSICAL PROPERTIES OF HOUGHTO-SAFE 1120
PHOSPHATE-ESTER FLUID

Appearance	Light Green
Viscosity (SUS at 100° F)	230
Viscosity (SUS at 210° F)	43
Odor	None
Flash Point (° F)	485
Fire Point (° F)	690
Auto-Ignition Point (° F)	Over 1200
Boiling Point (° F)	Over 750
Pour Point (° F)	0
Density (ppg at 60° F)	9.5
Specific Gravity at 20°/20° C	1.15
Hot Manifold Test (° F)	Over 1200
Neutralization Number (KOH/gm,max)	0.1
Moisture Content (wt %,max)	0.1

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APPENDIX B

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LABORATORY ANALYSES OF THE HYDRAULIC FLUID

Type of Sample	Moisture Content (ppm) (1)	Neutralization Number (mgs KOH/gm)(2)	Interfacial Tension (dynes/cm) (3)
New Fluid	1180	0.043	23.0
Fluid from Sump	2010	0.043	22.7
Fluid from Sump on April 6 after 87 Hours Side Stream Flow through Filter-Dryer Unit at Rate of 3 gpm	80	0.032	23.3
Fluid from Sump on May 4 after Flow through Original Filter-Dryer Charge	10	0.021	23.6
Fluid from Sump on July 15 after Flow through Original Filter-Dryer Charge	10	0.022	Not Analyzed
Recommended Limits for Phosphate-Ester Fluid	< 100(4)	< 0.05	23.0 (min)

- (1) Moisture Test (by Karl Fischer process) - Moisture may be in the hydraulic fluid as free or dissolved liquid. Most of the oxidation products formed in a lubricant are soluble in water. The water then becomes acidic and will also act as a catalyst in the presence of iron and copper to form metallic soaps, which in turn accelerates oxidation.
- (2) Neutralization Number (reported as mgs KOH/gm sample) - This test indicates acids which are formed in oxidation of the hydraulic fluid and other products of oxidation which are built up in the fluid. Neutralization tests do not determine the exact amount of acids present, but show the presence of materials in the fluid that react immediately with KOH at the particular temperature and the time involved in making the test.
- (3) Interfacial Tension - Force in dynes necessary to detach a planar ring of platinum wire from a water-oil interface under rigidly standardized nonequilibrium conditions. Interfacial tension values indicate a particular sensitivity to suspended particles in a hydraulic fluid. This test is an index of the molecular attractive forces between unlike molecules. When oxidation products form, polar molecules at the water-oil interface become oriented and reduce the interfacial tension.

- (4) New fluid as received from the manufacturer will contain approximately 1000 ppm moisture. This fluid should be added to the system through the filter-dryer unit to reduce the moisture.

Comments - Operational variations will affect the neutralization number and interfacial tension. With warmer fluid temperatures, the moisture in the fluid acts as a solvent and the neutralization number increases. With cooler fluid temperatures, the fluid is less active, acid precipitates into sludge, the neutralization number decreases, and the interfacial tension increases.

Spectrographic reports of impurities in the sump fluid prior to cleaning and from the millipore filter are shown in Figure 6.

SSL SPECTROGRAPHIC REPORT													
Material Type		Date		Requester		Batch Number		Requisition Number		Plate Sheet No.			
Non U		4-2-65		Dr. M. M. M.				432664					
REPORTING UNIT						REPORTING BASIS							
☒ % ☐ ppm ☐ Other						☒ Sample as Received ☐ Metal Basis ☐ Other							
☐ µg/ml ☐ Total µg						☐ Dry Basis ☐ Ash Basis							
17482													
Ag	2.005	Al	.03	Au	<.02	B	2.01	Ba	<.002	Be	<.001	Bi	2.01
Ca	.06	Cd	<.01	Co	<.005	Cr	.02	Cs	2.6	Cu	.012	Fe	720
Hf	2.02	K	<10	Li	<.002	Mg	.01	Mn	.20	Mo	.005	Na	.03
Nb	2.02	Ni	.20	P	710	Pb	.01	Pd	.01	Pt		Rb	<.15
Sb	2.04	Si	.15	Sn	.02	To	2.15	Th	2.04	Ti	.015	U	<.3
V	<.002	W	<.08	Zn	.08	Zr	.002						

(a) Residue From Fluid In Sump Prior To Cleaning

SSL SPECTROGRAPHIC REPORT													
Material Type		DATE		REQUESTER		BATCH NUMBER		REQUISITION NUMBER		PLATE SHEET NO.			
REPORTING UNIT				REPORTING BASIS									
☐ % ☒ ppm (Mg/g by wt.) ☐ Other				☐ µg/ml ☐ Total µg				☐ Sample as Received ☐ Metal Basis ☐ Other ☐ Dry Basis ☐ Ash Basis					
Ag	<1.2	Al	1155.	Au	<4.6	B	11.6	Ba	438.9	Be	<.2	Bi	<2.3
Ca	877.8	Cd	104.	Co	<1.2	Cr	104.	Cs	438.6	Cu	1155.	Fe	1155.
Hf	<4.6	K	207.9	Li	13.9	Mg	115.5	Mn	50.8	Mo	13.9	Na	808.5
Nb	<4.6	Ni	13.9	P	7230.	Pb	207.9	Pd	<.5	Pt		Rb	<34.6
Sb	<9.2	Si	3465.	Sn	104.	Ta	<34.6	Th	<9.2	Ti	50.8	U	<69.3
V	1.8	W	<18.5	Zn	877.8	Zr	2.8						

(b) Residue From Millipore 0.45 Micron Filter

Figure 6. SPECTROGRAPHIC REPORTS.

APPENDIX C

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- ## DRYING PROCEDURE FOR THE NEW FILTER TUBES AND A NEW MOLECULAR SIEVE BEAD REFILL FOR THE FILTER-DRYER UNIT

3. Disconnect the two sets of flanges at the bottom of the Molecular-Sieve tank and remove the tee section for access to the perforated sleeve. Remove the sleeve to allow the beads to fall from the tank into the container. Clean the sleeve if required. (Notice that the existing bottom sleeve is too long to be removed without raising the tank to allow the sleeve to clear the platform. To prevent this inconvenience, the present sleeve can be bent or cut for removal and then replaced with a new, shorter sleeve which will be shipped to the BONUS Site.)
4. Reinstall the bottom sleeve and tee section.
5. Disconnect the two sets of flanges and union to remove the piping section at the top of the Molecular-Sieve tank. Remove the perforated sleeve from the top of the tank.
6. Pour a new charge of Type 4A, 8 x 12-mesh beads into the top of the tank. Approximately 60 pounds are required. Tap the side of the tank to pack the beads. The free space in the top of the tank should be six inches from the face of the flange on the tank.
7. Reinstall the top sleeve (clean if required) and piping section.

TO DRY THE NEW BEADS AND FILTER TUBES

1. Close Valves 8, 9, and 7. (Valves 2, 3, and 11 should still be closed from the previous draining operation.)
2. Open Valve 13. The vent plugs in the tops of all three filters should still be removed from the second step to remove the excess hydraulic fluid. Also, remove the 1/2-inch drain plugs located on the bottom side of the two upstream filter housings.
3. With the 12-kw heater off, slowly open Valve 14 to allow 5-psig, dry, oil-free air to go through the heater, sieve bed, and filters, then exhaust through the filter top and bottom ports previously opened. (Air flow must be maintained through the heater when the heat is on to prevent the heater element from burning out.)
4. Turn on the heater switch and adjust the thermostat to 250° F. Crack Valve 9 to establish a flow through the downstream filter. Observe the exit temperature at the downstream filter vent port using a hand thermometer as this filter will dry first. This temperature should eventually reach 160 - 180° F. (A higher temperature than 220° F will burn the filter material.) Maintain the effluent temperature of 160 - 180° F from the downstream filter port for 60 minutes.

5. During the drying time of the downstream filter, the filter ports on the two upstream filters should be checked for the effluent air temperature. When the 160 - 180° F range is reached, hold for 60 minutes. When the downstream filter becomes dry, close Valves 9 and 10 and replace the vent plug.
6. When the upstream filters become dry, crack Valve 7 to maintain an air flow through the heater, close Valves 4 and 5, and replace the vent and drain plugs in the two upstream filters.
7. Reset the heater thermostat to 400° F. Adjust Valve 7 to establish an adequate flow through the sieve bed so that TI-1 will reach a temperature of 300° F. Hold this temperature for 10 minutes.
8. Turn off the heater switch and adjust the thermostat to 0° F.
9. Immediately close Valves 14, 13, and 7.
10. Allow the sieve bed to cool to ambient temperature which can be observed on TI-1 and TI-2. During this period the filters will be cooling.
11. Proceed to refill the sieve tank and filters with hydraulic fluid and establish the flow as described in Appendix D - Operating Procedure for Filter-Dryer Unit.

Warning - Air temperatures entering any part of the system must not exceed 600° F.

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OPERATING PROCEDURE FOR THE FILTER-DRYER UNIT

(See Figure 7 for Valve Numbers)

NORMAL OIL FLOW (assume all valves are closed)

1. Open Valves 1 and 2 (or 3).
2. Remove the small vent plug from the top of the upstream filter which is to be used.
3. Start the pump and operate it until oil appears at the vent.
4. Stop the pump and replace the vent plug.
5. Open Valve 4 (or 5) and partially open Valve 8 to vent air from the sieve bed tank.
6. Start the pump and operate it until oil appears at Valve 8 (use a container to catch the oil).
7. Close Valve 8 and open Valves 9, 11, and 12. Remove the vent plug from the top of the downstream filter. Continue to operate the pump until oil appears at the vent.
8. Stop the pump and replace the vent plug.
9. Start the pump and keep it on stream. Rotameter should indicate a flow of three gallons per minute.

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APPENDIX E

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EQUIPMENT TO DETERMINE THE MOISTURE CONTENT OF THE HYDRAULIC FLUID

Instrument kits for electrometrically determining the moisture content (as low as 10 ppm) in hydraulic fluids are available from several laboratory supply companies. These titrators consist of the following components attached to rod stands:

1. Two burettes (one amber and one clear) complete with stopcocks, ball and socket clamps, and offset capillary tubing for insertion into a beaker cover.
2. Platinum electrodes and a milliammeter.
3. Magnetic stirrer and stirring bars.
4. Karl Fischer reagent.

Complete procedures for making a water content analysis are furnished with the titrating equipment.

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APPENDIX F

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PROCEDURE FOR DETERMINING THE NEUTRALIZATION NUMBER OF THE HYDRAULIC FLUID

PREPARATION OF THE REAGENTS FOR THE NEUTRALIZATION NUMBER ANALYSIS

Standard Sodium Butylate Reagent

Add 2.3 grams of sodium to approximately one liter of butyl alcohol. (The metallic sodium will react slowly with the alcohol.)

When dissolution is complete, make one liter of the solution in a one-liter volumetric flask and shake.

Standardize the sodium butylate solution by observing the following steps:

1. Weigh three aliquants of pure benzoic acid and transfer the acid to a 250-milliliter Erlenmeyer flask.
2. Add fifty milliliters of the titration solvent and swirl the liquid until the benzoic acid is dissolved.
3. When dissolution is complete, add one milliliter of bromothymol blue indicator solution.
4. Add sodium butylate solution from a ten-milliliter burette until a dark blue end point is reached.
5. Calculate the normality of each aliquant as follows:

$$\text{Normality} = \frac{\text{weight of benzoic acid}}{\text{milliliters of sodium butylate} \times 0.12212}$$

Normality of standard sodium butylate equals the average of the three aliquants.

Titration Solvent

Add 500 milliliters of butyl alcohol to 500 milliliters of toluene.

Indicator Solution

Weigh 0.1 gram of bromothymol blue indicator and dissolve it in 100 milliliters of ethanol.

PREPARATION OF THE SAMPLE

Fill two 1 by 2-inch plastic vials with the sample. Place a cap on each vial.

Obtain the gross weight of each vial containing the sample. Record the weight on the work card.

Empty the contents of the vial into a 250-milliliter Erlenmeyer flask. Allow the vial to drain until little oil remains in the vial.

Replace the cap and obtain the tare weight of the vial. Record the weight on the work card.

TITRATION OF THE SAMPLE AND BLANK

The titration is conducted in a carbon dioxide-free atmosphere since the gas will interfere with the end point color change. Into the 250-milliliter Erlenmeyer flask containing the oil, add 50 milliliters of the titration solvent (a 1:1 mix of butyl alcohol and toluene) and one milliliter of the bromothymol blue indicator solution. Swirl the contents until the sample is entirely dissolved by the solvent.

Titrate to a dark blue end point with 0.1 normal sodium butylate. As the end point is approached, the increments of base added should be no greater than 0.03 milliliter.

Make a blank titration on 50 milliliters of the titration solvent and one milliliter of indicator, adding the 0.1 normal sodium butylate in very small quantities. Record the quantity of the sodium butylate that was required to reach the end point.

CALCULATION

Calculate the total acid number as follows:

$$\text{Neutralization Number} = \frac{\text{mgs KOH}}{\text{gms of Oil}} = \frac{(A - B) \times N \text{ of Base} \times 56.1}{\text{Wt of Sample (gms)}}$$

A = milliliters of sodium butylate that were needed to titrate the sample.

B = milliliters of sodium butylate that were needed to titrate the blank.

N = normality of the KOH.