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ORGANIC CHEMICALS DIVISION RESEARCH

FINAL REPORT
ON
SALES SERVICE ON PYDRAUL FLUIDS-(1958-59)

Job No. 2-02-750.01-3551

July 30, 1960

Written by: J. D. Sullivan

Monsanto

ST. LOUIS

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Monsanto Chemical Company
Organic Chemicals Division
St. Louis Research Department

St. Louis Research Report No. 2785

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Written by: J. D. Sullivan

Work done by: R. E. Hatton
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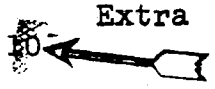

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INTRODUCTION

This report details the sales service problems involving Pydraul F-9, Pydraul AC, Pydraul 150, Pydraul 60 and Pydraul 625 referred to the Research Department during the years 1958 through 1959. It also includes evaluation and analyses of competitive fluids brought under observation during this period.

SUMMARY

This report does not encompass all of the sales service work done on functional fluids during 1958 through 1959, since it does not include those samples sent directly to the J.F.Q. Analytical Laboratories for regular analyses and relayed to the St. Louis Functional Fluid Application Laboratories for testing. These results were sent directly to the Sales Department for transmission to the individual customers. The work summarized in this report was concerned with fluid deposits, contamination problems, recovery techniques, compatibility studies and determing the component parts of new competitive functional fluids.

REFERENCES

1) Final Report "Sales Service Work on Functional Fluids, 1954-1955" Job No. 2633 by R. E. Hatton, April 2, 1956.

2) Final Report "Sales Service Work on Functional Fluids-1956" Job No. 2-02-750.01-3133 by J. D. Sullivan, May 14, 1957.

3) Final Report "Sales Service on Functional Fluids-1957" Job No. 2-02-750.01-3335 May 28, 1958.

EXPERIMENTAL WORK AND DISCUSSION

A. Competitive Fluids

1. Bayer Fluids (CA-9031 and CA-9032)

Bayer Leverkusen's CA-9032 is a clear, brown fluid containing 51.1% chlorine, no phosphorus and having the following physical properties:

Acidity	0.01 NN
Moisture	0.04%
Flash Point	270°F.
Fire Point	600°F.
Viscosity at 100°F.	11.49 cs.
at 210°F.	3.09 cs.
Viscosity Index	+148
Crystallizing Point	-13.6°C.
Sp. Gr. at 25°/25°	1.4298

Fractionating this fluid through an efficient laboratory column gave a first fraction (40°C. to 50°C. at 0.4 mm) equivalent to 46.8%. This fraction was identified by infra-red as being highly pure 1,2,4-trichlorobenzene. A second 45.8% fraction (50° to 160° at 0.35 mm) was separated through a punched column and identified by infra-red as being virtually identical to Aroclor 1248. By this method, Aroclor 1248 is readily distinguishable from Aroclor 1242 and Aroclor 1254. Calculating from an average chlorine content of Aroclor 1248, the base stock of Bayer's CA-9032 can be considered essentially a 50% mixture of Aroclor 1248 in 1,2,4-trichlorobenzene. The high viscosity index of this fluid despite the poor viscosity properties of the two main components necessitates the incorporation of a viscosity index improver into the formulation. An infra-red analysis of the 7.4% residue shows the presence of a polyether compound containing approximately five-carbon aliphatic chains and unterminated hydroxyl groups. The above infra-red results coupled with the fact that the fluid is essentially shear stable indicates that the viscosity index improver in CA-9032 is similar to the high molecular weight Ucon fluids.

On the other hand, Bayer CA-9031 is a brown fluid containing 23.42% chlorine, 4.24% phosphorus and having the following physical properties:

Acidity	0.02 NN
Moisture	0.06%
Flash Point	450°F.
Fire Point	600°F.
Pour Point	-10°F.
Viscosity at 100°F.	60.59 cs.
at 210°F.	7.44 cs.
Viscosity Index	+ 89
Sp. Gr. at 25°/25°C.	1.3019

These data indicate that CA-9031 is a formulation similar to Pydraul F-9 containing a phosphate ester (similar to TCP) and a chlorinated biphenyl (probably Aroclor 1248). The high

viscosity index of this fluid coupled with the fact that it is essentially shear stable is evidence that the formulation contains a very effective viscosity index improver.

2. Fyr-Trol 250

A gallon of Fyr-Trol 250, produced by Ergatomic Products Corporation was submitted for analysis and compatibility with Pydraul F-9. This was a sample of unused fluid received through Brown-Lipe-Chapin-Division of General Motors at Syracuse, New York. The following table lists the regular analysis of this unused Fyr-Trol 250 along with that for a used sample of Fyr-Trol received a year ago through the Newark Stove Company at Newark, Ohio.

	Used Fyr-Trol (Newark Stove Co.)	Unused Fyr-Trol (B-L-C Division of GM)
Acidity	0.22NN	0.06NN
Moisture	0.16%	0.33%
Pour Point	-15°F.	-25°F.
Sp. Gr. At 25°/25°C.	1.0976	1.0701
Viscosity at 100°F.	87.56 cs. 406 (SUS)	100.82 cs. 467 (SUS)
Viscosity at 210°F.	10.98 cs.	13.41 cs.
Flash Point	455°F.	450°F.
Fire Point	585°F.	565°F.
Viscosity Index	+117	+127

Comparison of these physical properties, especially viscosities, specific gravities and pour points, indicates that the formulation of this fluid has been substantially changed during this intervening period.

The used sample of Fyr-Trol was immiscible in Pydraul F-9, as 50% mixtures of the two precipitated out a tacky gel. The current Fyr-Trol 250 is compatible with Pydraul F-9 up to concentrations of 10%. Above 10 to 15%, a deposit of tacky solids is observed just below the surface of the liquid after the mixture stands for several days. The amount of these deposits is quite evident at 50% concentrations of Fyr-Trol 250 in Pydraul F-9 but not as copious as the tacky gel encountered in similar mixtures with the used Fyr-Trol fluid. Whether this improved compatibility with Pydraul F-9 is the result of changes in the Fyr-Trol formulation or the gel formation results from degradation during use was not determined.

3. Houghton's Gum Solvent B

Houghton's Gum Solvent B is a light yellow, neutral liquid containing 26.58% chlorine, no nitrogen, no phosphorus, and 0.06% sulfur. The fluid has a characteristic odor of o-dichlorobenzene. The presence of this compound was confirmed by comparative ultra-violet spectra. Calculating from the chlorine content (26.58%) of the Gum Solvent B gave 55.1% o-dichlorobenzene. Fractionating the fluid through an efficient laboratory column gave a first cut (up to 55°C. at 0.8 mm.) equivalent to 55.5% o-dichlorobenzene. This temperature corresponds to the atmospheric boiling point of o-dichlorobenzene, 180.4°C. The subsequent fractions contained less than 0.15% chlorine.

A second 32.5% fraction (70° to 140° at 0.8 mm.) had a specific gravity of 0.8290 at 25°/25°C. and contained 83% carbon and 13.5% hydrogen. A subsequent 9% cut was separated by a straight take-over distillation at 140° to 152°C. and 0.5 mm., having a specific gravity of 0.8389 at 25°/25°C. and containing 83% carbon and 13.3% hydrogen. The 3% residue contained a small amount of polymeric material possibly present as an additive or resulting from decomposition occurring by heating the fluid to 200°C. at 0.5 mm.

The wide distillation range (70°-152°) of the fractions following the sharp o-dichlorobenzene cut, coupled with their relatively low specific gravities (0.8290-0.8359) and high carbon-hydrogen content indicates that mineral oil is the second major component of this cleaning agent. The low percentages of nitrogen, phosphorus, chlorine and sulfur in the mineral oil portion indicates that definite purpose additives are probably not present. Therefore, Houghton's Gum Solvent B can be considered essentially a 50:50 mixture of o-dichlorobenzene and mineral oil.

4. Pennzoil-200

Pennzoil-200 is a light red fluid with a specific gravity at 25°/25° of 1.0685 and a viscosity of 42.8 cs. at 100°F. This viscosity corresponds to 199 SUS. It contains 0.003% phosphorus and less than 0.03% chlorine, removing it from a phosphate ester base or a Pydraul-type fluid.

Since it contained a large percentage of water, Pennzoil-200 was analyzed by the empirical method outlined in Final Report, Job No. 2-02-750.01-3335, in order to compare it to the other currently popular water-based fluids. Pennzoil-200 is essentially soluble in alcohol and other polar solvents. A toluene extrusion showed Pennzoil-200 contains 51.3% water and that 18.8% of the fluid is volatilized along with the water, as water-soluble materials, under the conditions of the extrusion (110°C.).

The red water-soluble base was separated from the toluene by filtration, and submitted for infra-red analysis. Spectrum of this material shows that it is a polyether carrying a considerable number of hydroxyl groups and is very similar to Ucon 75-H-90,000 and Niax Diol 10-HEG-112, except that the hydroxyl content is higher. There is a strong, unexplainable band at 6.42 microns which may or may not be due to an additive. Identification of this component will require further work.

5. Socony's RL 315-3 Fluid

A sample of RL 315-3 from Socony Mobil Oil Company was submitted for analysis. The following table lists the characteristic properties of this fluid.

Acidity	0.05NN
Moisture	0.01%
Chlorine	0.27%
Flash Point	515°F.
Fire Point	600°F.
Sp. Gr. at 25°/25°	1.1802
Pour Point	+5°F.
Viscosity at 100°F.	126.8 cs.
at 210°F.	7.54 cs.
Phosphorus	7.06%

RL 315-3 is a phosphate ester type fluid similar to Cellulube 550. A typical sample of Cellulube 550 contained 7.22% phosphorus and had a viscosity of 7.85 cs. at 210°F.

6. Socony's PD-309C Fluid

The following table lists the analytical data determined on a sample of PD-309C obtained from the Spang Chalfant Plant of the National Supply Division of U. S. Steel.

% Phosphorus	1.06%
% Chlorine	44.45%
Pour Point	20°F.
Sp. Gr. at 25°/25°C.	1.4324
Viscosity at 100°F.	79.99 cs.
at 210°F.	4.17 cs.
Viscosity Index	-734

The low viscosity index eliminates the incorporation of a viscosity index improver into the Socony formulation so that an estimation of its composition can be made by relating the percentages of phosphorus and chlorine to the viscosity of the fluid. Calculating from the percent phosphorus gave 13.3%

tricresyl phosphate and the high specific gravity (1.432) would indicate that Aroclor is the chlorine containing component of the formulation. By plotting the viscosity of Socony's PD-309C (79.99 cs. at 100°F) and the average TCP viscosity (34 cs. at 100°F.) against percentages by weight on an ASTM Standard Viscosity-Temperature Chart showed that an Aroclor base stock equivalent to 90 cs. at 100°F. was required to maintain the TCP at a 13.3% level. Aroclor 1248 is not thick enough and too much TCP (64%) would have to be added to Aroclor 1254 to reduce the viscosity to 79.99 cs. at 100°F. A 90 centistoke Aroclor base stock could be formulated by mixing 32.5% Aroclor 1254 with 67.5% Aroclor 1248. Diluting this Aroclor base stock with 13.3% TCP would result in a formulation (approx. 80 cs. at 100°F.) containing 13.3% TCP, 28.2% Aroclor 1254 and 58.5% Aroclor 1248 having a chlorine content of 43.3%, calculated on the average chlorine percentages of the Aroclors. Socony's PD-309C has a viscosity of 79.99 cs. at 100°F. and a chlorine content of 44.45%. A similar formulation may be prepared containing 13.3% TCP, 36.9% Aroclor 1242 and 49.9% Aroclor 1254 having a chlorine content of 42.4%.

Excluding the possibility of various additives, the physical properties listed in the above table would indicate that Socony's PD-309C fluid contains Aroclor 1248, Aroclor 1254 and tricresyl phosphate in approximately 59%, 28% and 13% concentrations, respectively.

7. Socony's DTE-103 and Cellulube 300

A sample of Socony's DTE-130, contaminated by Cellulube 300 as a black immiscible lower layer, was submitted by the Air Reduction Corporation, Alton, Illinois. The Cellulube (approx. 20%) formed an emulsion when mixed with the hydrocarbon oil. This muddy appearing fluid was drained from a crankcase which was lubricating the cylinder of one of their Chicago Pneumatic air compressors when a shaft twisted and a bearing failure occurred.

After standing for several days, the emulsion separated into two layers. The lower Cellulube layer was drawn off as a black muddy fluid leaving the clear light yellow hydrocarbon oil. The following table lists the analyses of these two layers:

Layers	Top	Bottom
Lot Number	C-1033	C-1032
Appearance	Lt. yellow oil	Black oil
Acidity	0.33NN	0.43NN
Moisture	0.02%	0.05%
Sp. Gr. at 25°/25°C.	0.9003	1.1369
Viscosity at 100°F.	------	72.9 cs.
Major Component	DTE-103	Cellulube 300

Shell 4-Ball Tests under 10 kg. and 40 kg. loads showed the DTE-103 layer to be a satisfactory lubricant, so the mechanical failures reported could not be attributed to the extraction of additives from the hydrocarbon oil by the Cellulube. However, a two-phase lubricant seems to have much poorer lubricity than either of the individual components as evidenced by numerous mechanical failures in the field where such a system is present or develops upon contamination.

This situation could be remedied by taking advantage of the fact that Pydraul AC is soluble in hydrocarbon oil and in this particular application, 20% Pydraul AC is readily miscible in DTE-130. On the other hand, a similar mixture of DTE-130 and Cellulube 300 forms a stable emulsion which separates slowly on standing.

B. Miscellaneous Monsanto Fluids

1. Tricresyl Phosphate (N.Y. Naval Shipyard)

A drum of cresyl diphenyl phosphate was sampled, analyzed and sent to the Material Laboratory (Code 942) at the New York Naval Shipyard. This lot was identified as B-2046 and the analytical data are as follows:

Acidity	0.06NN
Moisture	0.14%
Color	20 APHA
Refractive Index at 25°C.	1.5601
Sp. Gr. at 25°/25°C.	1.2047
Pour Point	-45°F.
Viscosity at 100°F.	17.53 cs.
at 210°F.	3.23 cs.
Flash Point	460°F.
Fire Point	over 600°F.

2. Cresyl Diphenyl Phosphate (N.Y. Naval Shipyard)

The sample of tricresyl phosphate representing a 55 gallon drum of Lot 2573 received from the Materials Laboratory of the New York Naval Shipyard, Brooklyn, N. Y. was submitted for analysis. The results of the analysis showed that the material was satisfactory for further testing.

3. F-9 Defoamer Concentrate (Dayton, Ohio)

Three pounds of defoamer concentrate, containing Dow-Corning 200 fluid, was prepared for the Sales Department to be used in a Pydraul F-9 installation at Dayton, Ohio.

C. Compatibility Tests on Monsanto Fluids

The Sales Department is interested in the possibility of using the dome type utility cans for packing five-gallon quantities of all the functional fluids. National Lead Company suggested an overcoat varnish which is alcohol resistant and sumitted samples of coated lithographed sheets for immersion tests in Monsanto's functional fluids. Both sides of these sheets were tested by pouring a spot of each functional fluid on a fresh surface and wiping the area dry with Kleenex. The amount of colored material which adhered to the Kleenex evidenced the extent of the attack on the treated surface. All of the fluids except OS-45 and OS-45-1 removed the bronze-colored coating from the "inside surface". The red paint on the "outside" lithographed surface, consisting of red emblems on a white background, was readily removed by Skydrol 7000, Skydrol 500, Pydraul 150 and Pydraul 60 but was essentially unaffected by Pydraul F-9, Pydraul AC, Pydraul 625, OS-81, OS-45-1 and OS-45.

Since rubbing the surfaces in the presence of the functional fluids, would not be typical for this application, individual test sheets were partially immersed in each functional fluid in closed containers at room temperature. After several weeks, all of the fluids, except OS-45 and OS-45-1, changed the bronze "inside surface" to a gold color in comparison to the similar unexposed area. In Pydraul 60, Pydraul 150, Skydrol 7000 and Skydrol 500, substantial amounts of red paint could be removed from the "outside surface" with Kleenex after several days immersion and entirely removed by the same technique after one month exposure. The red paint had no tendency to run into the white background and the white paint was essentially unaffected by the exposure.

Substantial amounts of red paint were more difficulty removed from the sheets in contact with Pydraul 625, Pydraul AC and Pydraul F-9 after one month immersion periods. In addition, Pydraul 625 and Pydraul AC caused the white background of the outside surface to become somewhat tacky. The painted outside surface was entirely resistant to OS-81 after a one-month exposure. A permanent blue film was deposited on the exposed painted surface immersed in OS-45-1 but this surface was entirely resistant to OS-45 after the one-month test.

In summation, only OS-45 was entirely resistant to the bronze "inside coating" and the "painted outside surface". OS-45-1 did not attack the inside bronze coating but fouled the external lithographed surface. The outside painted surface was resistant to OS-81 but this fluid removed the bronze inside finish. All of the other fluids attacked both sides of the sample sheets under the test conditions.

D. Pydraul F-9 (Carter Carburetor)

A substantial amount of Pydraul F-9 was accidentally contaminated with a water-based hydraulic fluid during an operation at Carter Carburetor Company. Samples of this fluid, having an acid number of 0.21 NN and containing 0.73% water, were submitted for a recovery evaluation. Filtration through Dicalite 586-B lowered the water content to 0.07% and reduced the acidity to 0.12 NN. The viscosity and specific gravity of the treated fluid was within specifications and the Dicalite treatment was recommended as a satisfactory method of recovery.

E. Pydraul AC

1. Pydraul AC Deposits (Electric Energy Inc.)

A sample of black semi-solid material was submitted for analysis by Electric Energy Inc., Joppa, Illinois. This sample (#833) was representative of the deposits removed from the low pressure cylinder of their #5 compressor in which Pydraul AC was used.

This sample was continuously extracted with benzene for three 48-hour periods. After each extraction period, the sample was dried and reground to insure intimate contact of the solvent with the solids in order to facilitate the removal of the fluid from the residual deposits.

After the first extraction period, 39.3% of the sample was removed as a blackish brown oil having an acidity of 0.33NN. Successive extractions completely freed the solids of benzene-soluble material. The residue, representing 62.5% of the original sample, was dried to a fine charcoal-like powder containing 56.6% iron, 14.9% hydrogen, 0.81% carbon, less than 1% chlorine and 0.04% phosphorus.

These analyses indicate that the solids isolated from these deposits are essentially inorganic materials. The acidity (0.33NN) of the recovered Pydraul AC is not excessive for a used fluid and eliminates the possibility of sludge formation due to the degradation of the lubricant. The low chlorine and phosphorus content of the residue show that excessive amounts of Pydraul decomposition products are not present in these deposits.

2. Saturation of Pydraul AC with Methane

Before Pydraul AC could be considered for application in gas compressors, it was necessary to determine the change in the physical properties of Pydraul AC saturated with methane at 1000 psi.

Pydraul AC was drawn into an autoclave under vacuum. The charged autoclave was then evacuated for 1/2 hour prior to successive sub-feed purgings of the stirred sample with methane at 300 to 500 psi, allowing no air to enter the system. Methane was then introduced into the stirred Pydraul AC until 1000 psi pressure was attained in the autoclave and the system was held at 950 to 1000 psi for one hour with constant agitation. Finally, after releasing the pressure the saturated Pydraul AC was poured into an open container and allowed to stand for one hour at room temperature before being analyzed. The fluid foamed more violently for a longer period of time, after the transfer, than similar treated OS-81 (2-02-750.01-3334, #10). The "active foam" subsided in approximately 1/2 hour but the sample kept releasing gas bubbles at a gradual reducing rate for several hours.

The following table lists some of the physical properties of Pydraul AC before and after contact with methane at 1000 psi.

Property	Before	After (saturated with methane)
Viscosity, 210°F.	88.02 cs.	85.4 cs.
, 100°F.	4.94 cs.	4.94 cs.
Sp. Gr. 25/25°C.	1.3477	1.3454
Pour Point	+20°F.	+20°F.
Flash Point	450°F.	460°F.
Fire Point	600°F.	600°F.

The data show that removal of air and saturation with methane does change the physical properties of Pydraul AC to a slight extent. However, it is believed that such changes are too small to have any effect on the functionality of the fluid.

3. Contamination of Pydraul AC (Mississippi River Fuel Co.)

A series of samples were submitted by the Mississippi River Chemicals Corp., Crystal City, Missouri, to determine the cause of fires in their ammonia plant, in which Pydraul AC was being used as an air compressor lubricant. Laboratory work conclusively showed that the Pydraul AC was grossly contaminated by hydrocarbon oil which could be ignited at the operating temperatures of the installation. Disproving the claim that "Pydraul will burn" under these conditions insures the continuation of this operation with Pydraul AC and offers increased sales potential for the fluid in this field.

In this compressor, Pydraul AC is used as a single pass lubricant and the fluid is discarded after it is removed from

the air stream. The air is compressed at 350°F. to 380°F. at 650 psi, cooled in a heat exchanger to 100°F., passed through an aglomerator, separator, scrubber and a drier prior to liquifaction. Fires were reported to have occurred in the heat exchanger and the drier. The following discussion summarizes the analytical data obtained on the materials isolated from samples of each stage of the operation.

a. Compressor

The sample was a single-phase black viscous oil removed from the second stage discharge of the air compressor. The entire sample was filtered through #2 Whatman paper prior to analyses. The following table lists the comparative analytical data for this recovered fluid (C-874) and New Pydraul AC.

	Recovered Fluid (C-874)	New Pydraul AC
% Chlorine	0.84%	30.78% (theory)
% Phosphorus	0.35%	3.53% (theory)
Sp. Gr. at 25°/25°C.	0.9044	1.346-1.350
Viscosity at 100°F.	229.6 cs.	82-92 cs.
at 210°F.	16.3 cs.	4.8-5.0 cs.
Acidity	1.3 NN	0.15 NN max.

Infrared analysis of this used fluid indicates that the sample is primarily an aliphatic hydrocarbon containing approximately 3% TCP. Calculating from the phosphorus content of the sample, the fluid contained 4.2% TCP. Decomposition of Pydraul AC into such an aliphatic hydrocarbon oil is eliminated by the low acidity, low chlorine content and low phosphorus percent of the sample. The only phosphorus absorption bands present in the infrared spectrum of the oil were associated with tricresyl phosphate.

b. Aglomerator

This sample of black oil represented the lubricant removed from the air stream in this operation and included a relatively small yellow water layer. The water layer had an acidity of 1.54 NN and a chloride content of 0.11%. The oil layer was dissolved in benzene, filtered through Dicalite, washed free of acid prior to the evaporation of the solvent and refiltration for analyses. Solutions of this lubricant in benzene were very difficult to filter as a black gum collected on and clogged the filtering medium. On the other hand, solutions in acetone filtered readily, even through paper and a black, carbon-like material was removed. The following table lists the analytical data on this recovered fluid (C-873).

% Chlorine	23.2%
% Phosphorus	2.42%
Sp. Gr. at 25°/25°C.	1.1821
Viscs. at 100°F.	97.9 cs.
at 210°F.	6.71 cs.
Acidity	2.4 NN

Although these results are closer to the properties of Pydraul AC than the compressor sample (C-874), the phosphorus and chlorine content of the sample indicates serious contamination and the viscosity and specific gravity of the sample indicates that this contaminant is probably a hydrocarbon oil.

c. Separator

This sample represented the water removed from the air stream and was a stable milky emulsion. Extractions with benzene removed a small amount of oily material having an acidity of 2.5 NN but failed to break the emulsion. A Dean-Stark extrusion showed that this emulsion contained less than 0.5% benzene soluble material in water.

d. Scrubbers

1. Caustic Scrubber

This sample was a light yellow, slight hazy solution containing 0.003% phosphorus.

2. Water Scrubber

This clear water-white sample contained 0.002% phosphorus.

These low percentages of phosphorus may be attributed to natural occurring contaminants and not to decomposition of tricresyl phosphate.

Work was continued on the problem of Pydraul AC contamination at the Mississippi River Chemical Corp., Crystal City, Missouri. Since the source of the contamination could not be found, samples of the lubricant were removed from each stage of every air compressor employed in the initial step of this operation. These samples were filtered through a cake of Dicalite in the laboratory before being submitted for infrared analyses and physical property determinations. The following table lists the results of this investigation.

Identification Compressor	Stage	Position	Percent Pyd: AC	Acidity (NN)	Sp. Gr. at 25°/25°	Viscosity at 100°F.
C-201-A	1st	Left	18%	2.46	0.939	174.6
C-201-A	2nd	Left	9%	2.36	0.913	241.8
C-201-A	1st	Right	18%	2.96	0.959	202.9
C-201-B	1st	Right	18%	5.22	1.003	224.6
C-201-B	2nd	Right	11%	2.98	0.915	204.4
C-201-B	2nd	Left	7%	2.35	0.906	226.1

Infrared spectra shows that the balance of the samples is an aliphatic hydrocarbon oil and these results are confirmed by the low specific gravities of the used fluid. New Pydraul AC has a specific gravity of 1.346-1.350 at 25°/25°C. This contamination has been attributed to the design of the installation and/or faulty operations and no further work is planned on this problem.

f. Pydraul 150 Deposits

Two samples of gray semi-solid material, removed from separate systems using Pydraul 150, were submitted for analyses. These Pydraul deposits were received from two different locations of the Republic Steel Company. Each of these samples were continuously extracted with benzene for three 24-hour periods. After each extraction period, the samples were dried and reground to insure intimate contact of the solvent with the solids in order to facilitate the removal of the fluid from the residual deposits. The analyses of these deposits are discussed below.

1. Republic Steel - Chicago, Illinois

At this location the deposits were scraped from a by-pass filter of an Askania Regulator. After the first extraction period, 55.5% of the sample was removed as a brown oil having an acidity of 3.7 NN. Successive extractions completely freed the solids of benzene-soluble material. The residue, representing 34.6% of the original sample, was dried to a chalky white powder had a 35.8% ash content as a measure of the inorganic material present.

2. Republic Steel - Cleveland, Ohio

At this location the sludge was removed from the filter of a hydraulic regulating system at the steam reducing station. After the first extraction period, 64% of the

sample was removed as a brown oil having an acidity of 2.4 NN. Successive extractions completely freed the solids of benzene soluble material. The residue, representing 10.5% of the original sample, was dried to a gray chalky powder containing 7.2% iron and 1.78% phosphorus. This gray powder had a 33.2% ash content as a measure of the inorganic material present.

Although large portions of the Pydraul 150 sludges were soluble in benzene, when the solvent is evaporated, gray solids precipitate from the isolated fluids indicating the presence of substantial amounts of partial esters. Similar precipitates result from solutions of the isolated oils in 95% ethanol. Ash determinations on the residual chalky powders indicate the presence of large amounts of organic material which could be decomposition products of Pydraul 150 having sufficient salt-like character to render them insoluble in benzene. After such extensive extractions of the deposits, the phosphorus remaining in the chalky powder would be present as a metallic salt.

In an effort to determine if Pydraul 150 deposits were resulting from outside contamination, a sample of "kish" was obtained from Keokuk Electro-Metals Co. "Kish" is a fine metal-like mixture of iron oxide and silicon oxide used in steel foundries. Only 1.6% of the "kish" was removed by continuous benzene extraction and the remaining material contained 4.3% iron but only 0.03% phosphorus. Accumulation of "kish" in the Pydraul 150 reservoir could not account for the inorganic phosphorus salts in the deposits but the "kish" could catalyze the decomposition of Pydraul 150 under high temperatures and/or pressure.

These residues appear to be examples of decomposition of the alkyl diaryl phosphate component of Pydraul 150 due to severe over-heating.

Samples of waxy deposits removed from separate systems using Pydraul 150 were submitted by different companies. The analyses of these deposits are discussed below:

3. Keokuk Electro-Metals Company #1

A sample of residue, removed from a hydraulic line at the Keokuk Electro-Metals Co., was essentially neutral when slurried in a suitable solvent. The residue was leached three times with hot benzene and the solvent was successively decanted through a Whatman #2 filter paper. After the insoluble material was transferred to the filter paper and washed with ether, the combined solvent washes were practically colorless.

The dried insoluble component was a gray, chalky powder, representing 27.5% of the residue, intermingled with black specks of magnetic material (probably iron and/or iron oxide). By the evaporation of the solvent, 72.5% of a greenish mobile oil was recovered having the characteristic odor of Pydraul 150.

4. Keokuk Electro-Metals Co. #2

The gray semi-solid was continuously extracted with chloroform for 24 hours to give a residue of 23.6%. This residue was further extracted (continuously for 16 hours) with benzene during which the amount was reduced to 23%. The residue was dried to a chalky gray powder containing 1% phosphorus and 13% iron. Elementary analysis of this powder gave the following results:

Si	0.05-0.15%
Mn	0.02-0.10%
Mg	0.001-0.01%
Pb	0
Al	0.01-0.06%
Cu	0.01%
Ca	0.02-0.03%
Cr	0.001-0.002%

After such extensive extractions of the residue, the phosphorus remaining in the gray powder would be present as a salt and from the metallic analysis, the decomposition product is probably iron phosphate. Because of the many forms of iron phosphate, a calculation of the percentage present cannot be made.

The combined solvents were evaporated to give 75.7% of a dark brown oil having an acidity of 4.8NN calculated on the original sample and 6.3NN calculated on theisolated oil. Further decomposition of the fluid was evidenced by the gray solids which precipitated from a solution of this dark brown oil in 95% ethanol indicating a substantial amount of partial esters even in the isolated oil.

5. Columbia Geneva Steel

The dark brown waxy material submitted by the above company was removed from an open hearth ladle auto pour unit using Pydraul F-9 and Pydraul 150. The entire sample was continuously extracted with chloroform for 24 hours to give a residue of 24.7%. The residue was dried to a chalky

gray powder containing 1.4% phosphorus and 10.9% iron. Elementary analysis of this powder gave the following results:

Si	0.05-0.15%
Mn	0.02-0.10%
Mg	0.01-0.20%
Pb	0.1 -0.3%
Al	0.01-0.06%
Cu	0.01
Ca	0.06-0.10%
Cr	0.002-0.01%

Evaporation of the chloroform gave a black oil having an acidity of 52.2NN. As in the case of the Keokuk sample, this is another example of decomposition of the alkyl diaryl phosphate component of Pydraul 150 due to severe over-heating.

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