Monsanto

CONFIDENTIAL

FROM (NAME & LOCATION) W. R. Richard - Research Center

DATE : May 20, 1970 cc

SUBJECT : MONTHLY SUMMARY DETAILS - APRIL, 1970
FUNCTIONAL FLUIDS - RESEARCH

REFERENCE

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Attached is our April, 1970 Monthly Summary with Details for Functional Fluids-Research.

W. R. Richard

ms

Att.

PLAINTIFF'S EXHIBIT
MBR 4/15/82

11261

MONTHLY SUMMARY DETAILS - APRIL 1970

FUNCTIONAL FLUIDS - RESEARCH

AVIATION FLUIDS

Skydrol 500B: Marketing Technical Service (JFH)

Boeing is installing an AIT apparatus per ASTM D-2155. They asked us to run four fluid samples to check their apparatus. Results are:

Phosphate Ester "A"	735°F
Phosphate Ester "B"	930°F
Engine Oil "A"	765°F
Engine Oil "B"	755°F

The following fluid samples were analyzed:

Fluid:	PIA Utility Reservoir	Texas International A/C 307 Right Systems	Left Systems
Visc. 100°F	7.88 cs	9.49 cs	9.32 cs
210°F	2.60 cs	3.10 cs	3.04 cs
Acidity	0.29 TAN	0.25 TAN	0.34 TAN
Moisture	0.45%	0.30%	0.34%
Density	1.0647	1.0591	1.0593

Skydrol 500C: Flight Test (1425243-JFH)

The properties of fluid samples from PAA received to date were analyzed by Jack Hinchen. The following conclusion can be drawn based on the assumption that aircraft top-up was the same for both the 500B and 500 C airplanes.

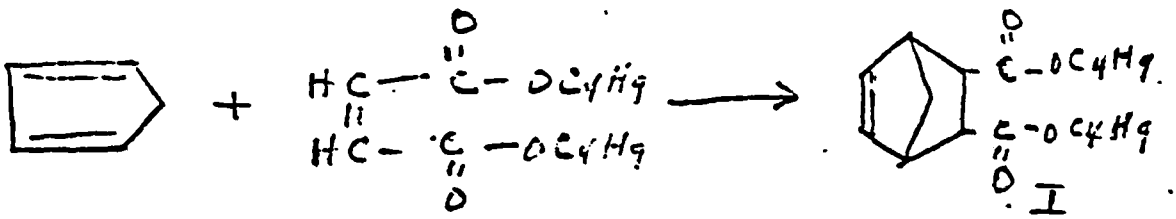
The overall conclusion is that topping-up a system with 500B does not affect fluid properties. That is no reduction in acidity is shown and the additional stabilizer is rapidly used up. In the 500C aircraft 1 1/2 top-ups stabilizes acidity at less than 0.40 TAN and stabilizer levels are high and remain that way. The 500B top-up shows no effect on viscosity but 500C viscosity approaches new fluid values. The 500B/500C mixtures in aircrafts after two 500C top-ups the moisture level is 0.30% and remains constant thereafter.

The 500C flight test at BOAC in Boeing 707's is underway. An analytical method was developed based on the VI improver's differences to determine the amount of 500C in 500B used samples. The method's precision needs to be refined but

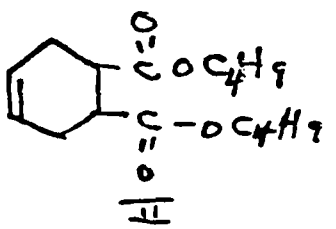
analysis of fluid from A/C ZG1 and A/C ZG2 showed 76 + 10% 500C. The ARB requires a minimum of 80/ 500C for a valid flight test.

Shaft Seals: (1425343-JFH)

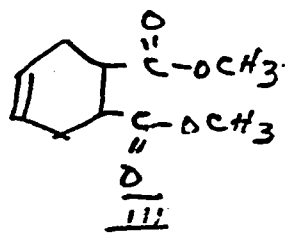
The following cyclohexenyl olefins were synthesized, will be epoxidized and evaluated.



I



II



III

It is interesting that the above reaction took place to yield I where the reaction between furan and dibutyl maleate yielded only starting material.

Skydrol Reformulation (RWS-RCR) 1425043

Thermal stability tests were run on THP formulations containing an AIT enhancer and certain amines. The results were encouraging. THP containing 0.50 percent 2,1,3-benzoselenodiazole servived 336 hours in the oxidative bomb test with a final acidity of 0.05. Metal attack was minimal and the fluid was clear and contained no precipitates. This formulation, see Table I, had an AIT of 825°F, up 125°F from THP neat. Those formulations containing 2,6-diaminopyridine and/or o-phenylenediamine in addition to the AIT enhancer were much less stable giving rise to metal attack and precipitates.

As an alternate to THP based fluids we formulated three potentially lower cost Skydrol LD fluids using TBP/TCP and less expensive VI Improvers. These formulations are given in the table below. Those containing Carbide's FH-25 acrylate VII, and R & H PL 10579 have better shear stability in the 10 hour probe test than Skydrol LD. Shaft seal, thermal stability, and valve tests are planned to evaluate these VI Improvers.

	OR 152611-1	152611-2	152611-3
	72.60% TBP 10.10% TCP 15.50% HF825 1.20% Uvex 711 0.60% FH132 0.27% H_2O.	71.40% TBP 9.91% TCP 17.00% PL10579 1.20% Uvex 711 0.10% FH132 0.27% H_2O	80.10% TBP 11.53% TCP 7.20% FH25 1.20% Uvex 2 0.50% FH13 0.27% H_2O.
Viscosity (cs)			
-40°F	360	391	—
100°F	11.26	11.29	11.19
210°F	3.83	3.86	3.87
Density			
23°C.	0.995	0.9946	0.995
Shear Visc after			
45 min	—	9.91	9.47
90 min	—	9.72	8.77
3 hr.	—	8.64	8.24
6 hr.	—	8.03	7.58
10 hr.	—	7.78	7.33

NOTES:

1. PL 10579 is a butylmethacrylate polymer in dioctyladipate. Priced about 85¢/lb in bulk.
2. FH25 is an acrylate polymer priced at 55¢/lb in bulk (>TCL) on 2-15-79
3. Viscosity of Stryclad LB after 6 hr probe Test was 7.0 cs.

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(1425243-RWS, RCR)

Two shaft seal tests were run on formulations containing 3,4-epoxycyclohexylmethyl acetate and 4-methylcyclohexene oxide. The results of both tests, No. 54 and 55, were good; no leakage and little to no deposit formation. The formulations were based on DBPP and contained the 500C additive package less FC-98 and Unox 221. The concentration of each epoxide was set so as to give the fluid an initial oxirane content of 0.35-0.37%.

The major effort was directed at scaling up the synthesis of these and other epoxides.

3,4-epoxycyclohexylmethylacetate. This material was initially prepared using m-chloroperbenzoic acid. A product of high purity (99.5% by G.C.) but mediocre yield, 51-52%, was obtained. A better procedure was needed owing to the relatively high cost of m-chloroperbenzoic acid ($23.50/100g).

A procedure using peracetic acid was developed. (Peracetic acid is inexpensive: $0.48/lb in bulk; $1.25/lb in gallons). This procedure is similar to that described in USP 2,838,524 and USP 2,716,123 wherein sodium carbonate and sodium acetate are used to buffer the reaction and retard formation of autoxy compounds. A 84% yield was obtained. The product distribution was 92% epoxide, 8% olefin. This is similar to the epoxide/olefin distribution in Unox 221 (89/11). The procedure is adaptable to the other epoxidations we have planned.

Methyl-3,4-epoxycyclohexylcarboxylate. This epoxide was prepared using the peracetic acid method described above. The product consisted of 83.6% epoxide, 16.4% olefin. The synthesis is being scaled up to provide sufficient material for several shaft seal tests.

Two routes were used to prepare the starting material methyl-3-cyclohexenylcarboxylate. The esterification of the acid in methylene chloride was easy and direct; however, the acid is expensive ($19/100g). The Diels-Alder synthesis was superior. The product was obtained in 88% yield from the $AlCl_3$ catalyzed reaction of butadiene and methylacrylate. This procedure follows that described in J.O.C. 30,3567(1965).

Future

1. Shaft seal tests will be run on 500C type formulations containing the following epoxides (prepared via peracetic acid
   a. Methyl-3,4-epoxycyclohexylcarboxylate
   b. Dimethyl-3,4-epoxycyclohexyl-1,2-dicarboxylate
   c. 4-methylcyclohexene oxide
2. Bomb tests at 225°F will be run on fluids containing each epoxide at 0.35% oxirane in DBPP at 0.50 H_2O.

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TABLE I

275° Oxidative Bomb Testing on Trihexylphosphate Formulations.

	Composition	Test Length	Final TAN	Hrs. to 0.5 TAN	Metal Attack Al	Cd	Cu	Mg	Fe
140396-1	97.4% THP (lot 129814-77) 1.6% Unox 221 0.51% H_2O 0.5% 2,1,3-Benzoselenadiazole	336	0.05	—	NIL	-0.16	-0.02	+4.82	-0.02
140396-2	97.3% THP (129814-77) 1.6% Unox 221; 0.5% 2,1,3-Benzoselenadiazole 0.1% 2,6-diaminopyridine 0.52% H_2O.	336	3.28	~250	-0.03	-0.31	-1.05	-4.14	-0.04
140396-3	97.1% THP; 1.6% Unox 221 0.50% 2,1,3-Benzoselenadiazole 0.30% o-phenylene diamine 0.52% H_2O.	168	0.78	~150	-0.02	-0.69	-1.01	-0.30	-0.03
140396-4	97.0% THP; 1.6% Unox 221 0.50% 2,1,3-Benzoselenadiazole; 0.1% 2,6-diamino-pyridine; 0.30% o-phenylene diamine; 0.50% H_2O.	168	6.32	~90	+0.02	-0.86	-8.66	-2.22	0.03

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275°F Oxidative Bomb Testing.
Epoxide Evaluation in 500C Type Fluid.

NRP.	Composition	Test Length	Final TAN	Hrs to 0.50 TAN	Metal Attack Al	Cd	Cu	Mg	Fe
131681-1 (140321-1)	76.3% DBPP; 7.0% HF412 (30% solids); 10.0% PL4857 (30% solids); 6.2% Union Carbide C-14,16 α-olefin epoxide; 0.50% FH132; 0.24% H_2O.	264	1.37	~240	NIL	$\overline{0.77}$	-0.33	+0.41	NIL
131681-2 (140381-2)	80.10% DBPP; 7.0% HF412; 10.0% PL4857; 2.4% cyclohexene oxide; 0.5% FH132; 0.24% H_2O	360	0.12	~375	-0.02	-1.19	-0.40	+0.59	+0.06
131681-3	78.20% DBPP; 7.0% HF412; 10.0% PL4857; 3.80% Epoxy-cyclohexylmethyl acetate, (0.25% H_2O)	360	1.23	~320	+0.09	-3.34	-0.71	-3.41	+0.03

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Shaft Seal Evaluation (1425235-RLG)

Eight DBPP formulations were evaluated for shaft seal leakage and deposit. The two formulations which used diepoxide, Unox 289, caused high leakage and deposit. The six formulations which employed other acid scavengers did not leak although one had some light deposit. A formulation MCS 986, using Epi Res 5014 had light copper transfer from a thrust bearing cage to the races. The Seal Reports 48 to 55 are attached. (Pages 8-15)

SST Hydraulics (1409035-RLG)

Due to the Hydraunycoil B pump test failure by high wear and copper transfer in the AP10V/280°F (318°F hot spot) pump, a lubrication test was devised to screen candidates before pump test. The test employs the LFW machine under the following conditions

Test time = 24 hours
Test Temp = 280°F (or pump test temperature)
Ring Speed = 200 rpm
Load = 8 lbs (53 psi)

with conforming Dynalloy 603HT bronze conforming blocks on hardened steel rings. The bronze alloy is the same as that used on the steel on bronze wearing surface in the AP10V pump. The forging conditions are the same since the test specimens were machined from pump cylinder barrel forgings.

The weight loss results are:

Test No.	Fluid	Wt. Loss mgs. 603HT block	E9310 ring
1	Used Hydraunycoil B from pump test	36.0	5.1
2	New Hydraunycoil B	32.7	5.6
3	Skylube 450	0.8	0.2

The results for Skylube 450 were so encouraging that a 280°F/AP10V pump test was started and at 210 hrs there are no operational problems. The wear correlation between the LFW wear and pump test seems to be correct when comparing the 156 hr failure of Hydraunycoil B to the Skylube 450 pump test.

If the Skylube 450 pump test is successful, the next test should at low temperature AP10V pump test with a Bendix valve test in parrallel with the load valve. The low temperature pump test would determine if K-salt micell formation blinds filters at the low temperature and the Bendix valve test will give a measure of the Skylube 450 erosion resistance.

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SHAFT SEAL PERFORMANCE EVALUATION

Fluid Formulation OR 14035-1-5

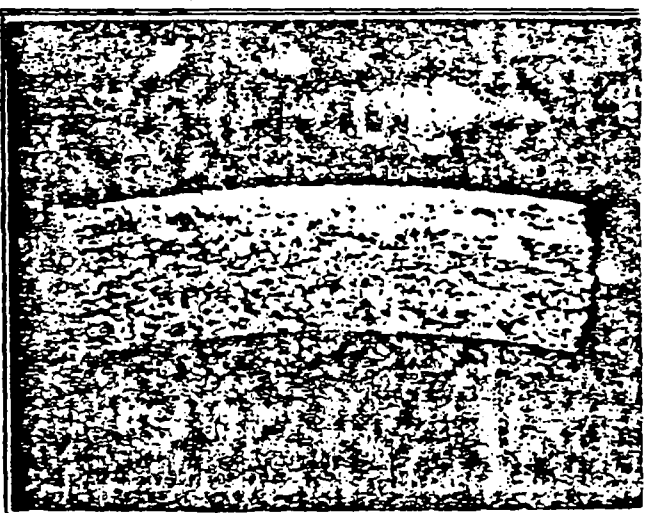
91.73% DBPP (A-D)	0.02% Benzotriazole
3.4% HF-412	0.25% H_2O
4.85% Unox 289	

Summary of Results

The total leakage in 214 hrs is 478 grams. A heavy resinous deposit covers the stator seal face.

Visual Condition

APlOV Shaft Seal "Bearium" Bronze Stator. Leakage path from outer to inner diameter. A heavy amber resinous deposit covers the entire seal face with a heavy concentration outside the sealing area on the atmospheric edge. Light wear is under the deposit.



Test No. 48

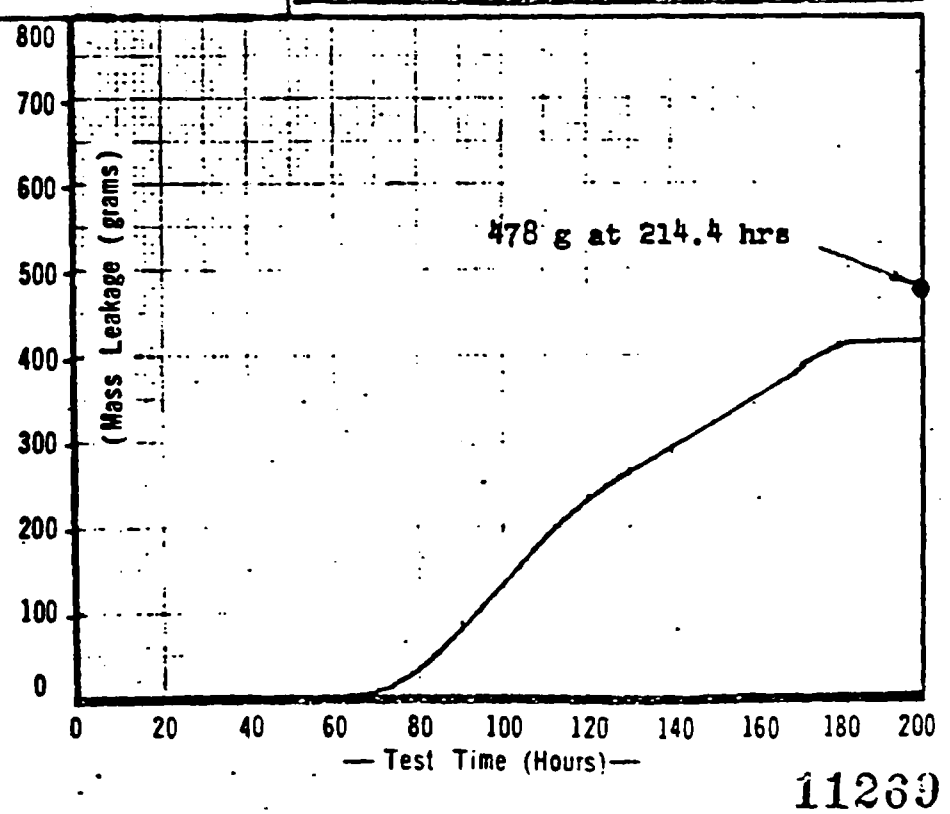
Tester No. C-1

Temp. 225°F

Time to 5 grams/hr. 85 hrs

Other

Stator thickness variation is 0.0002 in.



11239

SHAFT SEAL PERFORMANCE EVALUATION

Fluid Formulation OR.140381-4

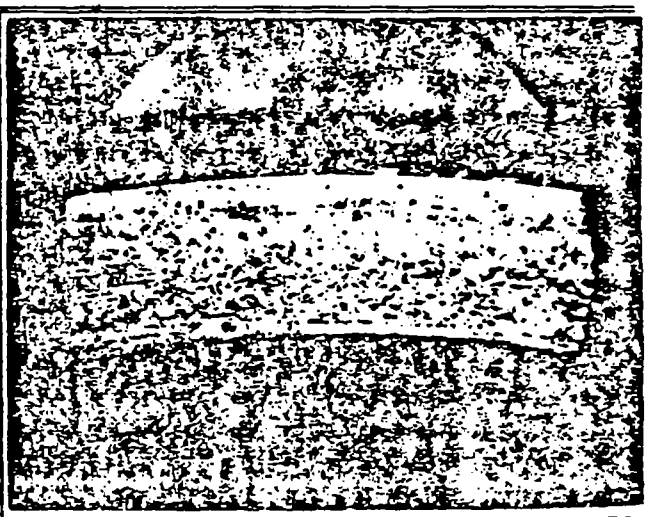
91.25%	DEPP (A-D)	0.27% H_2O
3.40%	HF-412	
4.85%	Unox 289	
0.50%	FH-132	

Summary of Results

The shaft seal had a total leakage of 644 grams in 212.0 hrs. The seal face has a white flaky deposit and no wear.

Visual Condition

AP10V Shaft Seal "Bearium" Bronze Stator. Leakage path from outer to inner diameter. A moderate amount of white flaky deposit covers the center of the stator seal face. The atmospheric edge has a black sludge concentration while the fluid side edge has no deposit. No wear is seen under the deposit.



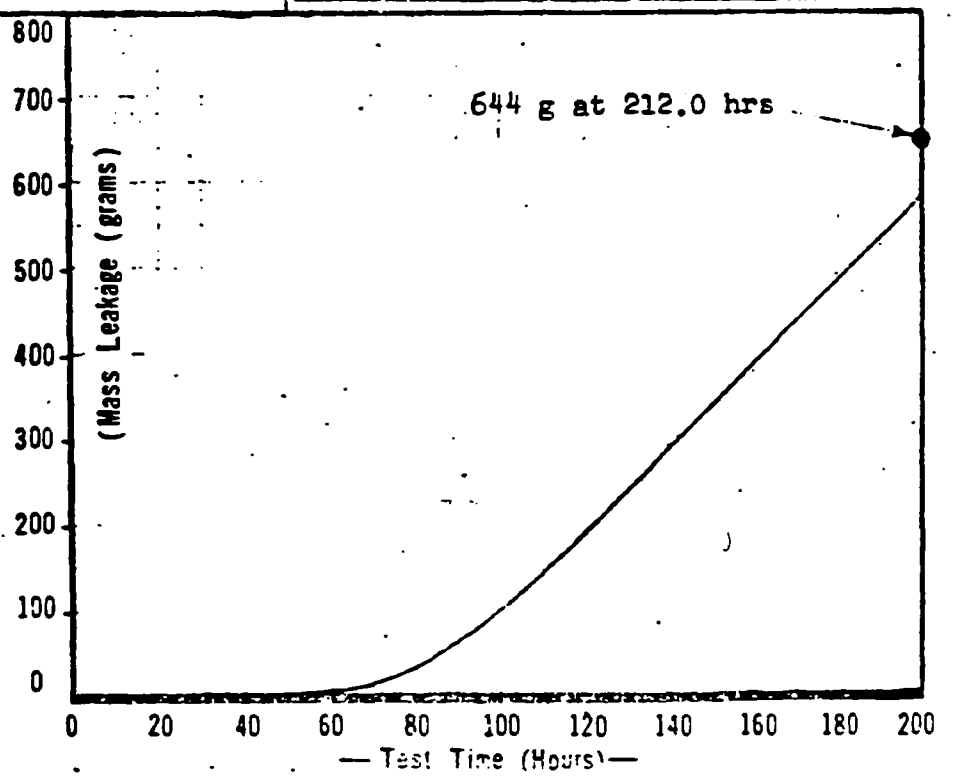
Test No. 49

Tester No. C-2

Temp. 225°F

Time to 5 grams/hr. 120 hrs

Other
Stator thickness variation is 0.0002 in.



11270

SHAFT SEAL PERFORMANCE EVALUATION

Fluid Formulation MCS 986

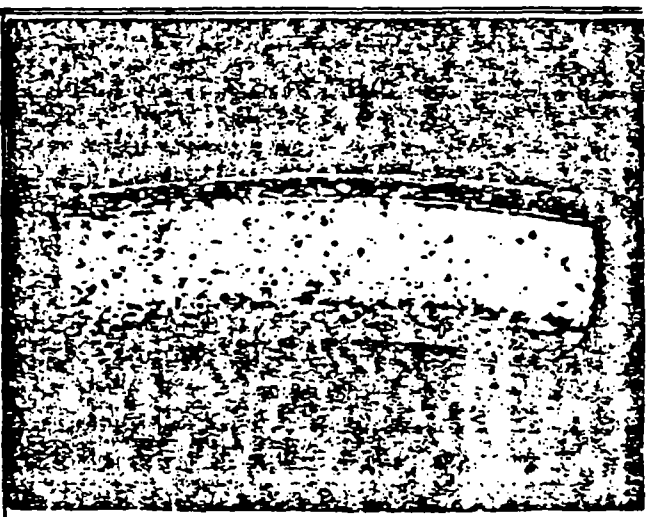
77.9% DBPP
7.2% HF 412 in DBPP (30% Sol.)
10.0% PL 4857 in DBPP (30% Sol.)
4.5% Epi Rez 5014
0.05% FH-132
0.22% H_2O

Summary of Results

In 213.7 hrs no leakage occurred. The edges of seal face have dark staining but no deposit is found.

Visual Condition

AP10V Shaft Seal "Bearium" Bronze Stator. Leakage path from outer to inner diameter. The edges of the seal face have a dark staining while the center of the face is clean. The seal face has a uniform smearing wear.



Test No. 50

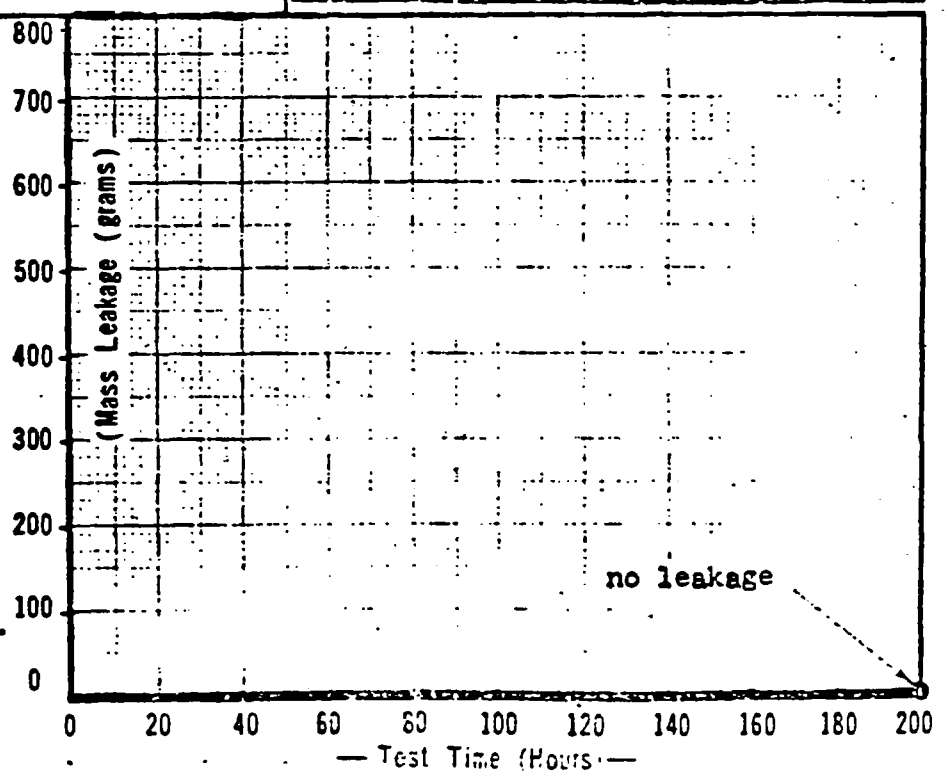
Tester No. C-1

Temp. 225°F

Time to 5 grams/hr. --

Other

Stator thickness variation is 0.0003 in. Bearing cage had light wear and copper transfer to balls and races.



11271

SHAFT SEAL PERFORMANCE EVALUATION

Fluid Formulation OR 140381-1

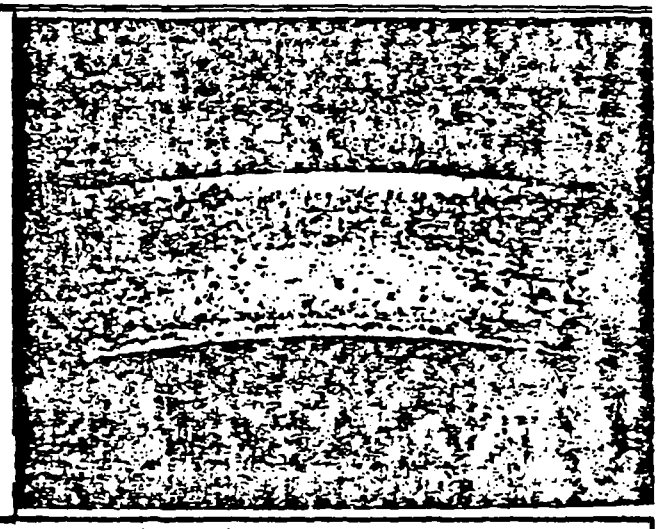
76.3%	DBPP (A-D)	0.5%	FH-132
7.0%	HF-412 in DBPP (30% Sol)	0.24%	H_2O
10.0%	PL-4857 in DBPP (30% Sol)		
6.2%	Olefin Epoxide		

Summary of Results

The seal stator has light staining and no leakage was observed during test duration of 212.3 hrs.

Visual Condition

AP10V Shaft Seal "Bearium" Bronze Stator. Leakage path from outer to inner diameter. A light rust colored stain covers the seal face. The stator has light wear.



Test No. 51

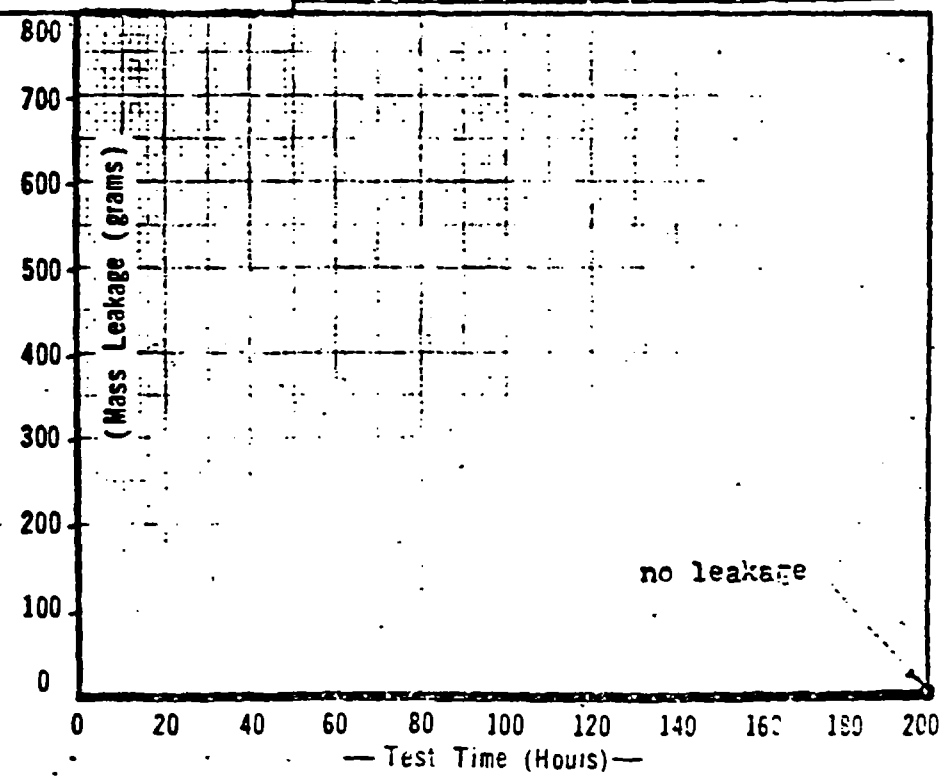
Tester No. C-2

Temp. 225°F

Time to 5 grams/hr. --

Other

Stator thickness variation is 0.0003 in.



11272

SHAFT SEAL PERFORMANCE EVALUATION

Fluid Formulation OR 140381-3

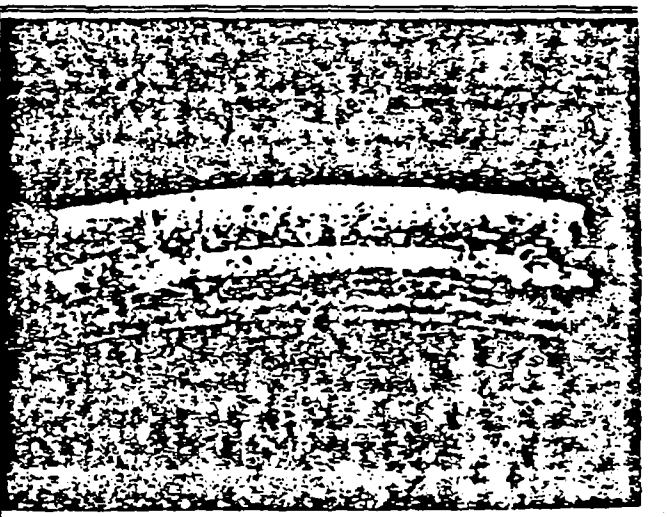
80.10%	DBPP	2.40%	Cyclohexene Oxide
7.0%	HF-412 (30% Sol)	0.24%	H_2O
10.0%	PL-4857 (30% Sol)		
0.5%	FH-132		

Summary of Results

The leakage was 1.6 cc in 262.4 hrs. The seal face has staining and light wear.

Visual Condition

AP10V Shaft Seal "Bearium" Bronze Stator. Leakage path from outer to inner diameter. The seal face has a bright copper color surface in the center with rust colored stains on each side. The center of the seal is lightly worn.



Test No. 53

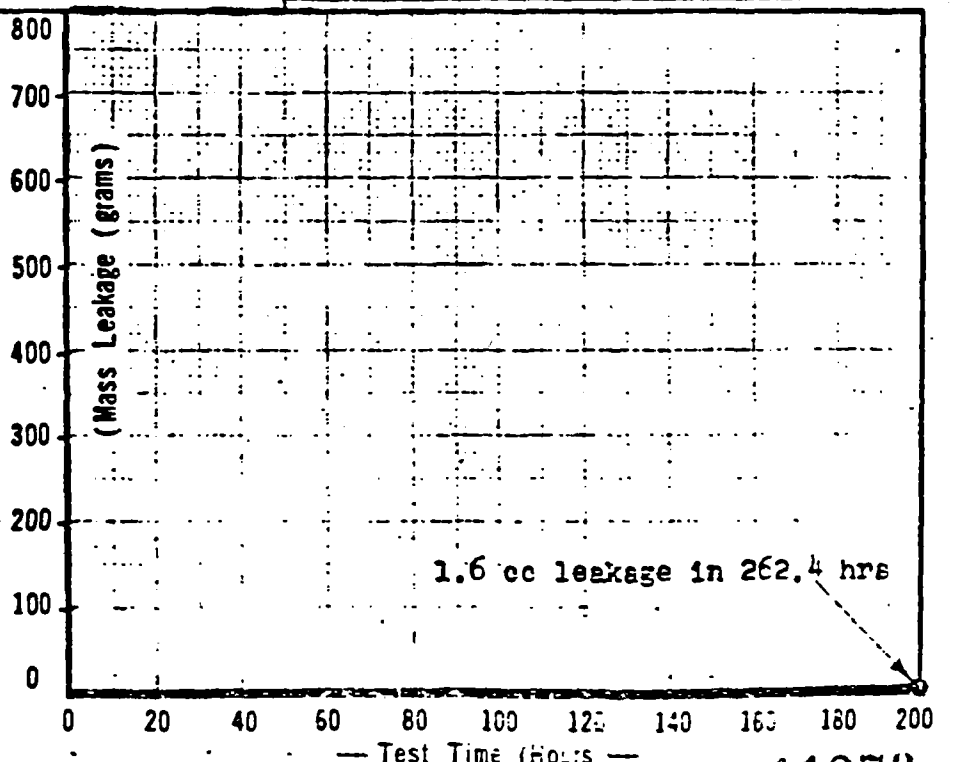
Tester No. C-2

Temp. 225°F

Time to 5 grams/hr. --

Other

Stator thickness variation is 0.0002 in.



11273

SHAFT SEAL PERFORMANCE EVALUATION

Fluid Formulation OR 152601-1

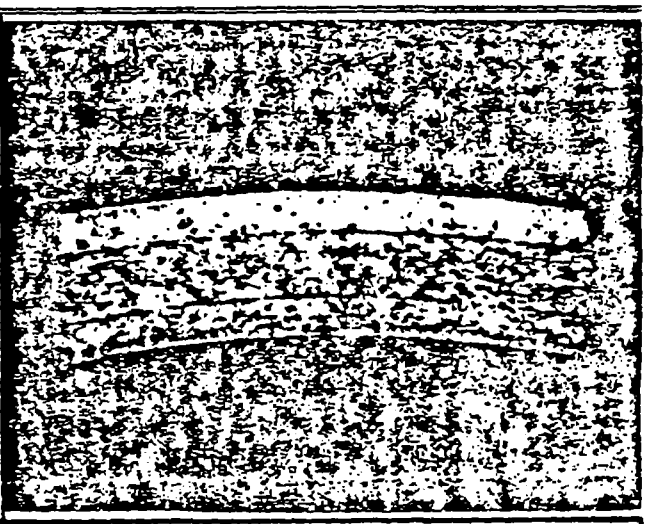
78.7%	DBPP (A-D)	3.8%	3,4-Epoxycyclohexyl methylacetate
7.0%	HF-412 (30% Sol)	0.5%	FH-132
10.0%	PL-4857	0.22%	H_2O

Summary of Results

The total leakage was 1.8 cc in 234.8 hrs. No deposits were present but the seal face has pitting and light wear.

Visual Condition

APlOV Shaft Seal "Bearium" Bronze Stator. Leakage path from outer to inner diameter. The center area of the seal face is pitted and lightly worn while the edges have light rust colored stain and little wear.



Test No. 54

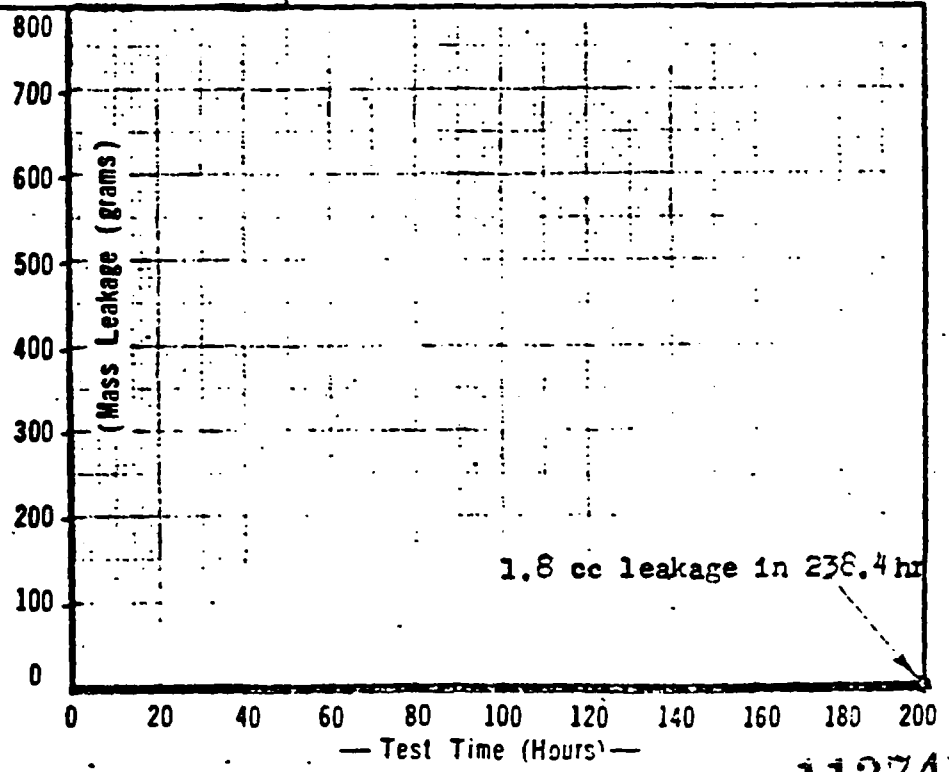
Tester No. C-1

Temp. 225°F

Time to 5 grams/hr. --

Other

Stator thickness variation is 0.0002 in.



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SHAFT SEAL PERFORMANCE EVALUATION

Fluid Formulation OR 152601-2

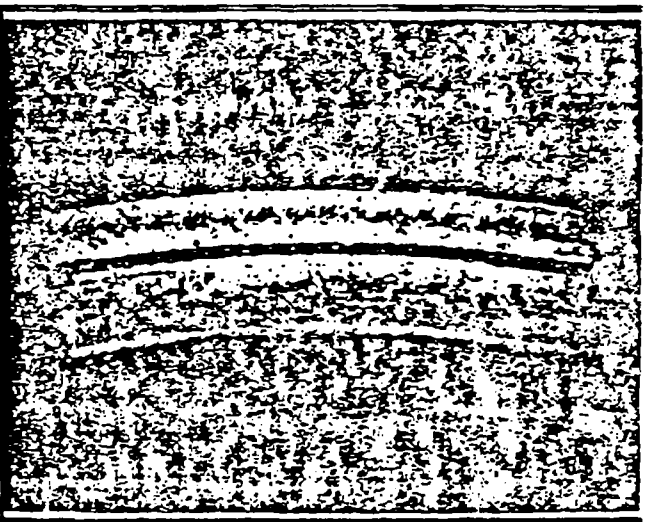
80.0% DBPP
7.0% FH-412
10.0% PL-4857
0.5% FH-132
2.5% 4-methylcyclohexene oxide
0.22% H_2O

Summary of Results

The leakage was 1 drop in 200.4 hrs. Light deposits and stains are on the moderately worn seal face.

Visual Condition

AP10V Shaft Seal "Bearium" Bronze Stator. Leakage path from outer to inner diameter. The stator face has a light resinous deposit on the atmospheric half of the dynamic seal area. A thin black stain-deposit is on the center area while rust and copper color stains are on the fluid half of the seal face. Moderate wear is present.



Test No. 55

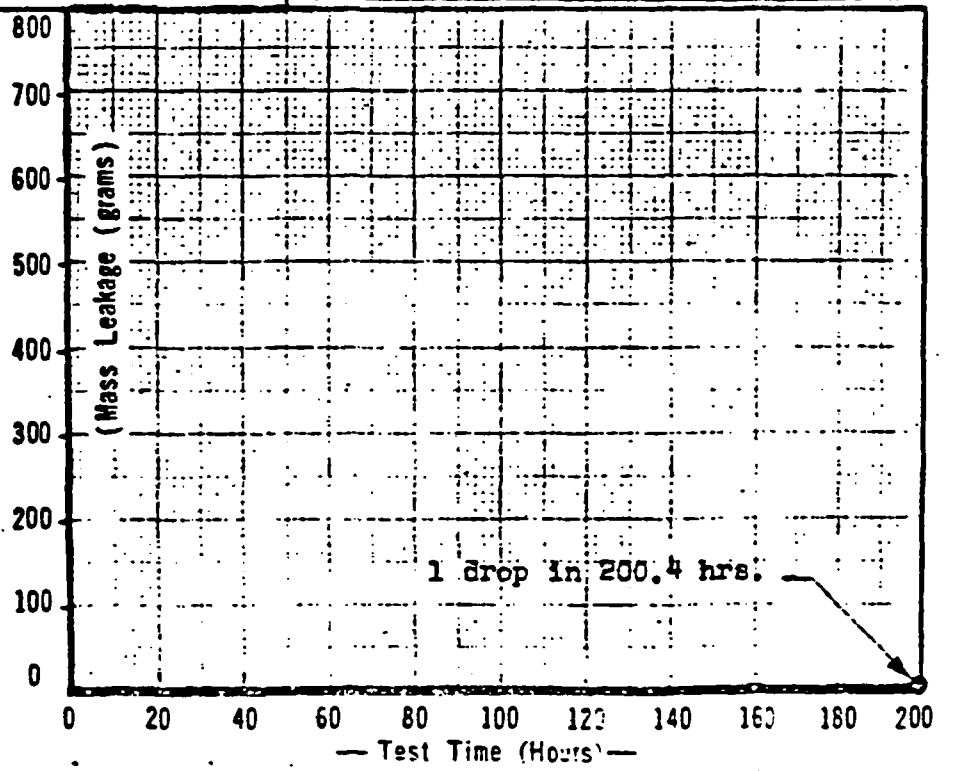
Tester No. C-2

Temp. 225°F

Time to 5 grams/hr. --

Other

Stator thickness variation is 0.0002 in.



11275

SHAFT SEAL PERFORMANCE EVALUATION

Fluid Formulation OR 152601-2

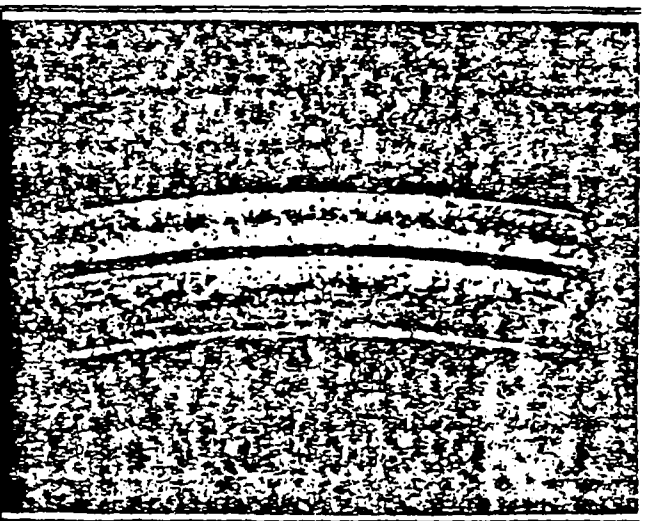
80.0% DBPP
7.0% FH-412
10.0% PL-4857
0.5% FH-132
2.5% 4-methylcyclohexene oxide
0.22% H_2O

Summary of Results

The leakage was 1 drop in 200.4 hrs. Light deposits and stains are on the moderately worn seal face.

Visual Condition

APIOV Shaft Seal "Bearium" Bronze Stator. Leakage path from outer to inner diameter. The stator face has a light resinous deposit on the atmospheric half of the dynamic seal area. A thin black stain-deposit is on the center area while rust and copper color stains are on the fluid half of the seal face. Moderate wear is present.



Test No. 55

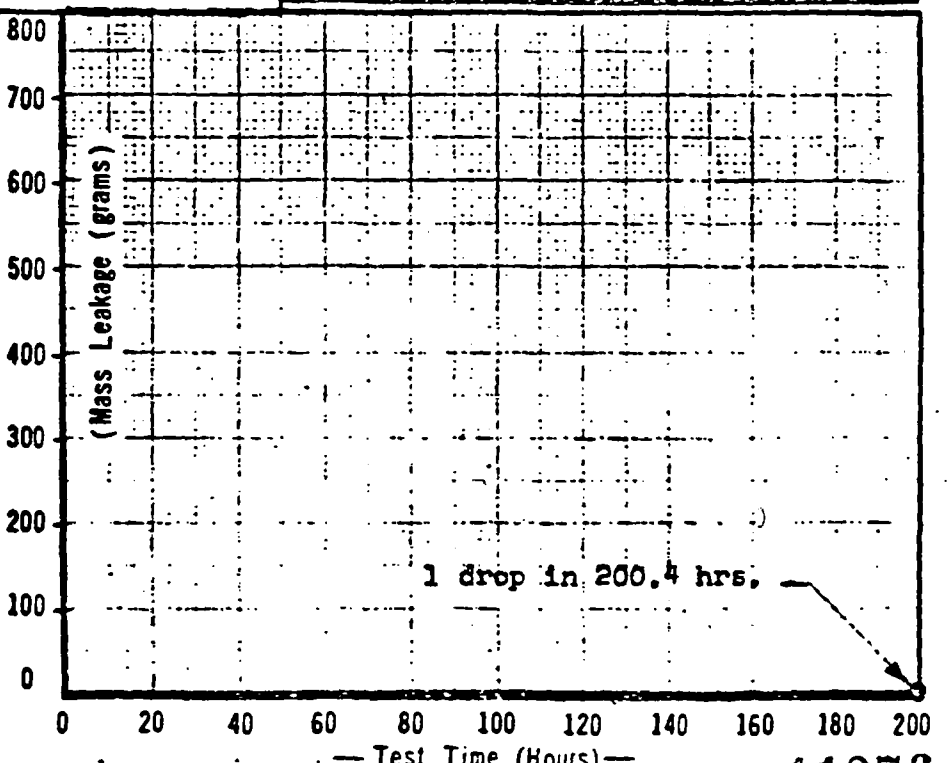
Tester No. C-2

Temp. 225°F

Time to 5 grams/hr. --

Other

Stator thickness variation is 0.0002 in.



11276

Metals in Solution (1425235-RLG)

A 24-hr. LFW/280°F/200 rpm/8 lbs load test using Mueller Bronze Alloy 603HT on E9310 steel was performed on Skydrol LD, QL 30. The metal combination is the same used on most sliding bronze on steel surfaces on the AP10V pump. The bronze test block was conforming to the steel ring which allows wear with little change in geometry which is more simulative of pump wear than the 4-ball test. The weight loss results are:

0.3 mgs on the 603HT Block
0.4 mgs on the E9310 Ring

A 72 hr test under the above conditions will be run too, if wear is accelerating and to determine if copper solubility is accelerating between 24 and 72 hrs.

Skylube 450 (1432159-SLR)

A Skylube 450 production run was made in the W-Bldg. facilities, totalling 2000 gal in 5 batches. Some plugging of the final cartridge filter was experienced, but it was determined that this was not due to the fluid, but rather to rust and scale in the system. All five batches were satisfactory on foam, Pb corrosion, Ryder and O & C. Two additional batches, representing reworked QM-5, an earlier batch that had been double-charged with amines, gave high Mg corrosion in the O & C test. A lab reworked sample gave a good O & C test. The W-Bldg reworked batches will be further checked.

Analyses (by RAL) on the latest samples of used Skylube 450 from the Northeast Utilities industrial jets indicate continued satisfactory performance. One engine has over 620 hrs since oil drain.

Stauffer Jet II is seeing considerable service in industrial engines. The lower viscosity of a sample of Stauffer's used oil compared to our sample of fresh Stauffer Jet II suggested that a VI improver might be present in their oil, but sonic shear and other tests do not indicate the presence of a VI improver. Analysis of the base stock shows it to be a PE ester of butyric (9.5%), hexanoic (0.5%) and heptanoic (90%) acids. Analysis of the additive package is incomplete.

We have prepared complex ester salts in which chloromethyl-phosphonic and methylphosphonic acids replace the phosphorous acid. These salts will be evaluated in Type II formulations. We will be looking especially for possible improvement in load-carrying properties compared to the phosphorous acid complex ester salts.

Tentative Processes (1432259-SLR)

We have duplicated the literature preparation of 5-nitro-indazole, a 5-AI precursor. Modifications are being studied to adapt the method to pilot plant size preparations. A problem which may complicate self-manufacture of 5-AI is availability of an intermediate, 2-methyl-4-nitroaniline. American Cyanamid has stopped production of this material. Central Purchasing is seeking other sources.

Nyco Fluids (1432359-SLR)

We have confirmed that addition of our complex ester salt to Turbonycoil 93, while showing some improvement in O & C tests, does not bring it up to Skylube 450 performance. Comparisons are shown in the accompanying table.

CRD acids (1432459-SLR)

Enough C_7 and C_9 acids are purified and sufficient C_8 acids are expected shortly from CRD to prepare a base stock sample for Ryder scuff test. Inadequate agitation in the 3-gal glass-lined autoclave will necessitate further modifications or purchase of another reactor for synthesis of large enough samples of acids to make fluids for bearing rig and fatigue tests.

COMPLEX ESTER SALTS IN TURBONYCOIL 93
OVCT TESTS

FLUID	% VIS. INC.	METAL CHANGE, MG/CM²					
		Mg	Al	Ti	Fe	Cu	Ag
TURBONYCOIL 93	505	-13.4	+0.04	+0.31	+0.03	-0.72	-0.02
TURBONYCOIL 93 + K-SALT	135	-4.1	+0.02	+0.35	+0.02	-0.46	-0.02
SKYLUBE 450, NO K-SALT	>500	-7.8	+0.16	+0.04	+0.02	-0.35	+0.01
SKYLUBE 450 (WITH SAME K-SALT AS ADDED ABOVE)	64	-0.09	+0.02	+0.02	+0.02	-0.21	+0.01

11278

Santomelt (1405343-JFH)

Technical data is being collected to present to the Air Force for Santomelt's use at their Tactical Air Bases. The required performance properties required are:

	Our Results
Immersion corrosion	pass
Stress corrosion	pass
Magnesium corrosion	report

The magnesium corrosion data collected is below.

	mg/in^2
Distilled water (control)	0.45; 0.39
10% Santomelt in distilled H_2O (pH=7)	38.4
10% " " " " (pH=10.5)	34.1
25% " " " " (pH=7)	341
25% " " " " (pH=10.5)	320; 343

Since the Air Force is interested in the 10% solution values our data is well within their desired values.

Santomelt for Commercial Use - The three problems facing Santomelt before complete acceptance are:

a) Aluminum Sandwich Corrosion
b) Hydrogen Embrittlement/Stress Corrosion
c) Magnesium Corrosion

The seriousness of the effect of magnesium corrosion is not completely resolved.

Progress was made in controlling the aluminum sandwich corrosion by increasing the concentration of the sodium meta silicate used. At present it is a toss-up as to include the formamide in the Santomelt formulation or not from a technical point of view. From a raw material cost picture formamide should be excluded.

A test was developed to evaluate the hydrogen embrittlement area and to ascertain the difference between stress corrosion and hydrogen embrittlement. Work in this area continues in search of a solution.

MCS 990: 75% formamide/20% urea/5% H_2O - a deicer developed by Canadian Dept. of Transport, ran into hydrogen embrittlement problems in Air Canada's testing. Since we developed a process and agreed to custom manufacture MCS 990 for Canadian DOT, we had the hydrogen embrittlement test checked by an approved independent laboratory. Test results show the product as manufactured passes but a 1:3 dilution with distilled water fails.

INDUSTRIAL FLUIDS (WHC, PDF, PFH, LFS, LRS)

Pydraul Reformulation -

Pydraul 625A

An additive package has been developed to improve the pump performance of 625A. The V105C pump had a total weight loss of 20.2 mg which is equivalent to the previous performance record (Fyrquel 220-18.4 mg total) and significantly better than Pydraul 625 or Fyrquel 550.

A process amendment to incorporate the additives is being issued.

Samples of the additive version of 625A and Fyrquel 550 have been sent to Alcor for deposition testing.

The oil gear test is at the 450 hour mark with no apparent wear.

The raw materials cost of the latest version of 625A is $1.77/gal versus $1.79 for 625.

Thirty-five gals of Pydraul 625A identified as Batch OR 139046 was blended for laboratory pump testing (V105C).

	49.10% wt.	Aroclor 5442
	49.50% "	Santicizer 140
	0.30% "	Santolube 70A
	1.00% "	Unox 221
	0.10% "	Benzotriazole
+	0.02%	FC-98
	20 ppm	DCF 200, 350 cs

The above formulation was tested in the V105-C pump stand with excellent results, total weight loss amounted to 20 mgs.

Pydraul 540A

The V105E results on an additive version of 540A (TBP and Cu inhibitor) look excellent. A V105C test is underway.

The tentative process for 540A is being written.

The raw materials cost of 540A is $.87/lb vs. $.83/lb for 540.

Five gals identified as Batch OR 139054 was blended for laboratory pump testing (V105E).

39.00% wt. Aroclor 1242
15.10% wt " 5442
32.00% wt Bright Stock 200
11.00% wt TCP
2.50% wt TBP
0.30% wt Santolube 70A
0.10% wt Benzotriazole

+ 20 ppm DCF 200, 350 cs

The above formulation was pump tested in the V105E screening pump with excellent results, total weight loss less than 10 mgs.

A second batch of Pydraul 540A (40 gals) was prepared (OR 139055) and is now being pump tested in the V105C pump stand, a portion of this fluid is reserved for an Oil Gear Pump Test (scheduled for late May). Oil Gear Pump, Pydraul 625A (OR 60697) has now accumulated 500 hrs.; indications point to a passing test.

Pydraul F-9A

A screening pump test of the Santicizer 711 version of F-9A is underway. If these results look encouraging, further properties will be gathered.

Pydraul A-200A

Testing will be completed as soon as possible on the 1242 version of A-200A.

Two non PCB versions of A-200A will be evaluated. Highest priority will be given to the TCB/5442 blend. The other will be a Arobromoclor 1232/5442 combination.

Pydraul 312A

After four runs on the computer we have evolved component viscosities which blend in a linear manner. The latest printout had a measured viscosity at 100°F within 2 cs of the predicted value. The next round should be within 1/2 cs. Once the linearization of viscosity is accomplished, the computer program can be used for the calculation of Pydraul 135, 230, 90 and 540 blends.

The calculated FR level was verified on a limited scale by full scale fire testing. A blend containing Arobromoclor 1231 was equivalent to Pydraul 135 on the FM spray and hot manifold. It was better than Pydraul 312 as predicted.

The composition of TCB/5442 and Arobromoclor 1232/5442 blend at various levels of FR follows.

Pydraul 135A

It appears possible to remove PCB's by utilizing TCB/5435. However, the sediment formation in 5435 blends will have to be stopped. Anniston is working on the problem. Analysis of the filtrate from a 5435/TCB blend showed a mixture of mono, di, tri, and tetra chloro terphenyl with about 70% being dichloro. Anniston doubts these results and is running their own analysis.

They believe the material to be slightly chlorinated quaterphenyl. The sample of 5035 sent for distillation by QET analyzed as 34.2% Cl. Anniston is working on 35-37% Cl materials. Gene Arnett feels that the specific gravity changes rapidly enough below 37% Cl to enable process control to ± .5% Cl.

Composition & Properties of 312A Blends

	ΣA-ΣB=0.0 Equivalent to Hydrasal 320	ΣA-ΣB=-0.06	ΣA-ΣB=-0 Equivalent Hydrasal 31
Kerr McGee Br.S.	30.27	35.7	41.1
TBP	2.5	2.5	2.5
CDP	4.5	4.5	4.5
TCB	24.6	24.7	24.6
5442	38.2	32.6	27.1
Calc Visc @ 100°F	67.0cs	67.0cs	67.0cs
Actual Visc @ 100°F	64.97	65.05	—
Calc Density 25°C	1.161	1.130	1.099
Cost/lb	$.091	$.087	$.083
Actual Density	—	1.188	
Cost/gal		$.863	

Kerr McGee Br.S.	29.3	34.7	40.1
TBP	2.5	2.5	2.5
CDP	4.5	4.5	4.5
AB1232	36.7	36.9	37.0
5442	27.0	21.4	15.9
Calc Visc @ 100°F	67.0cs	67.0cs	67.0cs
Actual Visc @ 100°F	—	66.55cs	—
Calc Density 25°C	1.223	1.193	1.162
Cost/lb	$.097	$.088	$.084
Actual Density	—	1.150	—
Cost/gal		$.844	

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Pydraul 625A, AC-A, AC-A W.G. 28

Viscosity-temperature curves, Timken loads, relative drop sizes, and Falex Wear and Load were obtained on the revised air compressor lubricants for a revised sales brochure. These values follow:

	Timken O.K.Load	Film Strength
Pydraul 625A	30	11,700
Pydraul AC-A	45	15,100
Pydraul AC-A, Winter Grade 28	24	8,600

Listed below are results of Falex Load and Wear Tests on Pydraul AC-A, Pydraul AC-A Winter Grade 28 and Pydraul 625A. Tests were run by the Chrysler Procedure.

Falex Load Test

	Passing Load
Pydraul AC-A	4025 lbs
Pydraul AC-A, W.G. 28	4175 lbs
Pydraul 625A	4250 lbs

Passing load was determined by the average of two tests.

Falex Wear Test

	Wt. Loss, Pin	Wt. Loss, Blocks
Pydraul AC-A	0	0.5 mg
Pydraul AC-A, W.G. 28	0.6 mg	0.9 mg
Pydraul 625A	1.1 mg	5.8 mg

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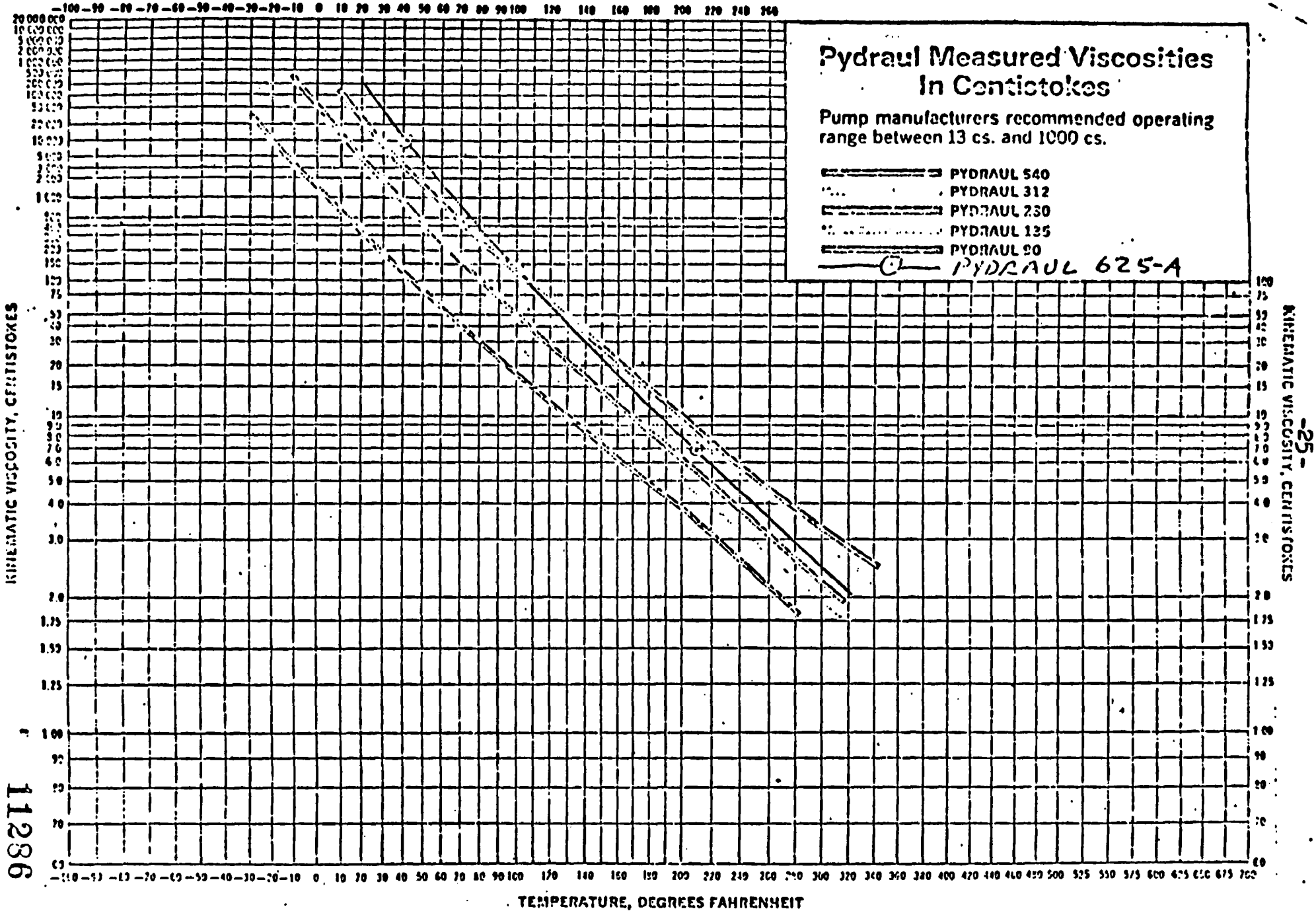
	Drops/ml at 25C
Mobil DTE 24	37
Mobil DTE 26	36
Pydraul AC	41
Pydraul AC-A	39
Pydraul AC, W.G. 28	39
Pydraul AC-A, W.G. 28	40
Pydraul 625	43
Pydraul 625A	39
Fyrquel 550	37
Fyrquel 220	37
Fyrquel 150	37
Fyrquel 300	37

The new formulations are completely miscible in all portions with Fyrquel 150, 220, 300, and 550; Houghto-safe 1010, 1120, and 1130; and our new phosphate esters, MCS 030E, MCS 050E, and MCS 0115E.



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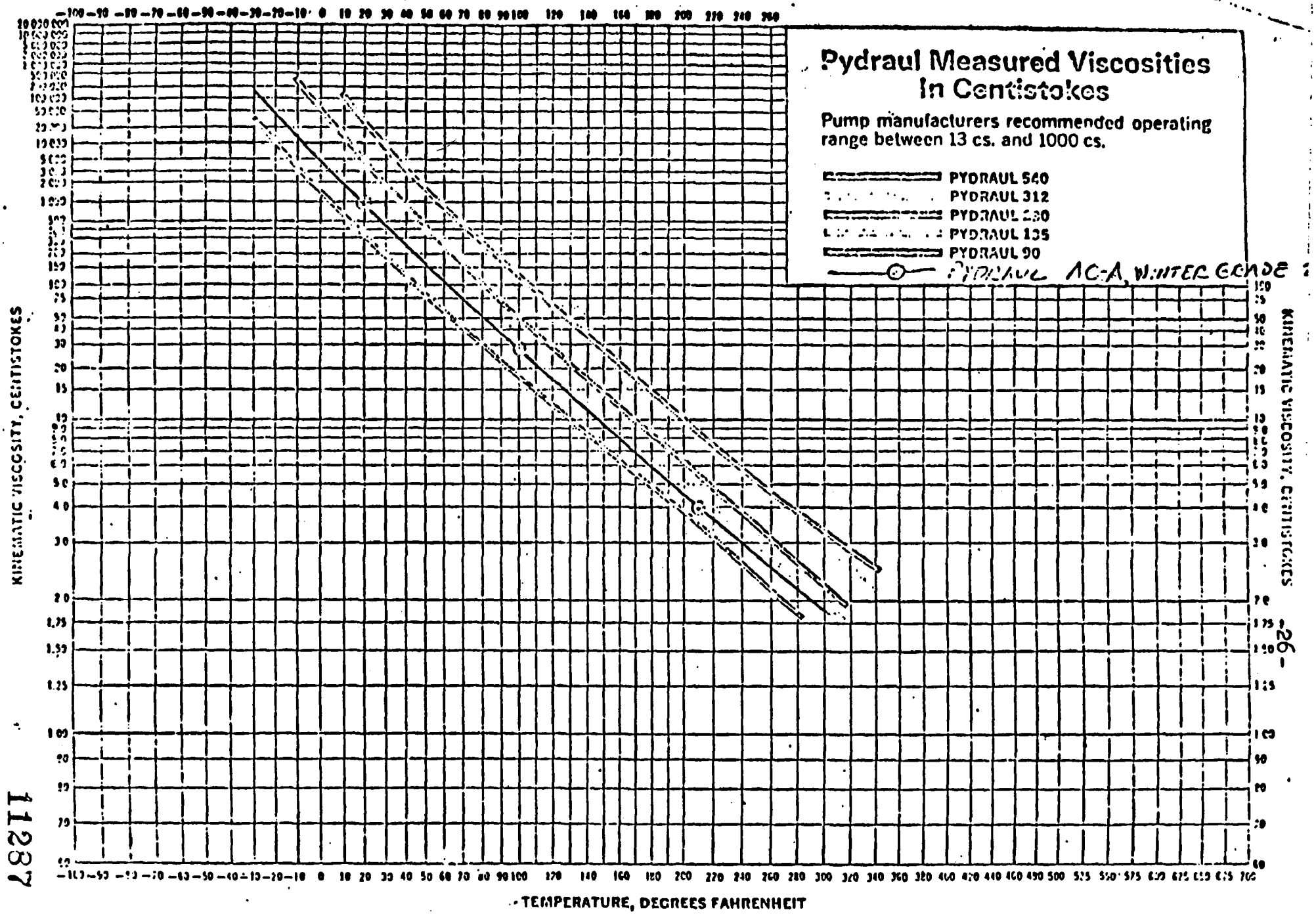

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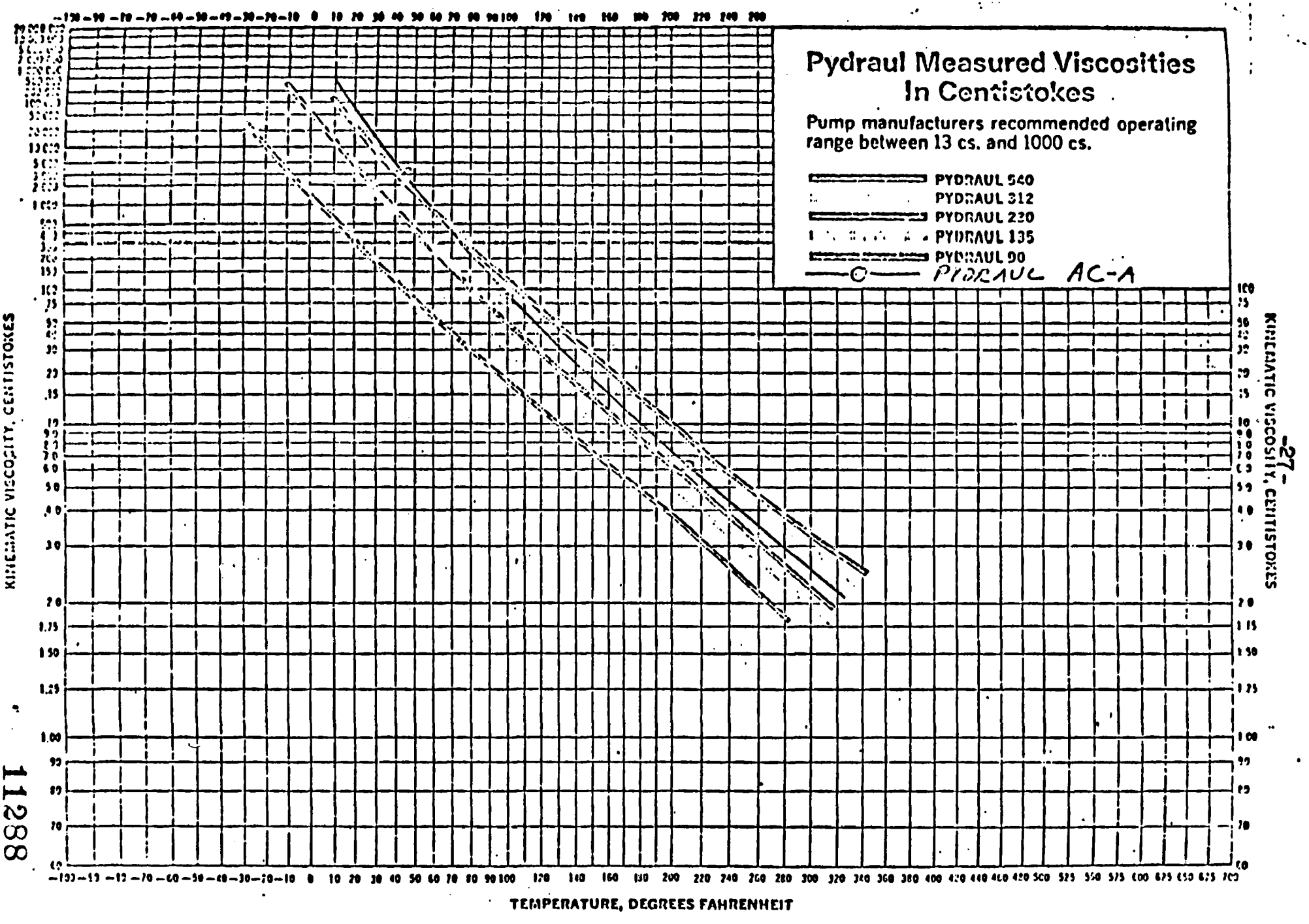

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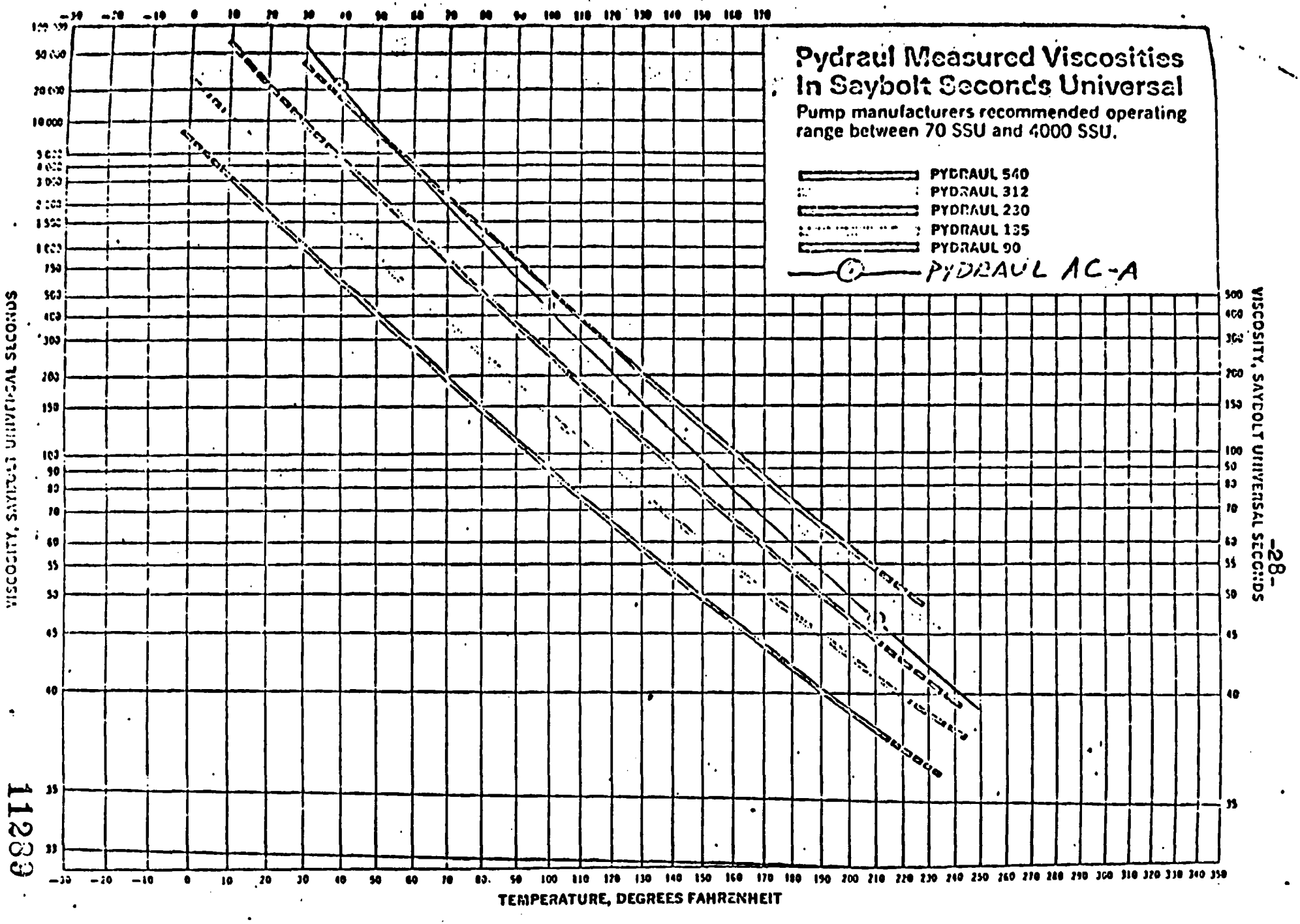

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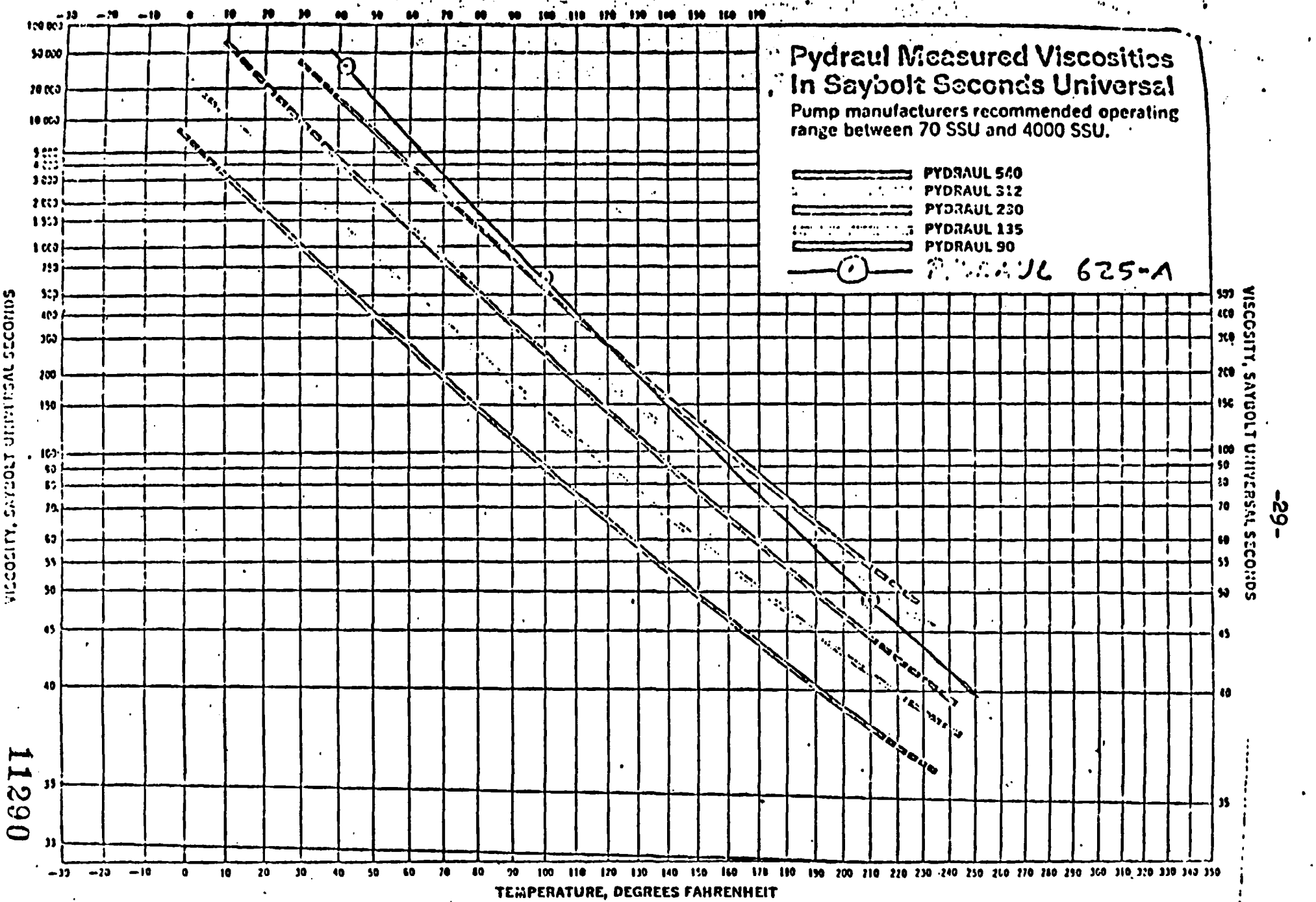

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Pydraul Measured Viscosities In Saybolt Seconds Universal

Pump manufacturers recommended operating range between 70 SSU and 4000 SSU.

- PYDRAUL 540
- PYDRAUL 312
- PYDRAUL 230
- PYDRAUL 135
- PYDRAUL 90
- PYDRAUL AC-A, WINTER GRADE

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-30-

VISCOSITY, SAYBOLT UNIVERSAL SECONDS

TEMPERATURE, DEGREES FAHRENHEIT

German Mine Fluid

A Tentative Process was written for Pydraul 281, a revision of Pydraul 280 which provides improved low temperature performance and eliminates the use of Aroclor 1260.

Fluid Recovery and Reclamation

Initial work to assess recovery of Pydrauls from customer effluent streams has been started on samples from Johnson Motors, Central Diecasting, Lester Castings, and Timken Bearing Co. Qualitative results indicate large amounts of solid material present with the oily layer. Filtrations have been difficult and relatively large amounts of fluid remain with the solids. This work will continue to provide more data on quantity and quality of fluid recovered as well as suggesting methods for recovery.

Turbine and Air Compressor Lubricants
European Steam Turbine Control Fluid

Samples of Geigy Reolube TF 90, Reolube HYD 110, and trixylyl phosphate were screened for possible use as steam turbine control fluids in Europe. Of these the TF 90 material was considered the best candidate at this time.

Tests run to date included 350°F O & C, beverage bottle hydrolytic stability, and a screening pump test and wick fire tests on the TF 90.

MCS 979

The field trial at the AGTL Cremona station has passed 800 hours with no problems. The inner bearing on the DeLaval booster has reached 192°F on several occasions and AGTL is keeping a close watch on the situation. Tom Oneson has requested us to formulate a lower viscosity fluid in anticipation of warmer weather.

We would then be going back to essentially the same fluid as MCS 974. Perhaps an AIT enhancer could be utilized to upgrade the FR of MCS 974 but it is roughly equivalent to Pydraul 135 now.

Water/Glycol Fluids

A Tentative Process for Santosafe W/G fluids blending using Carbide concentrate and thickener has issued. A capital project for equipment to blend these fluids at JFQ plant has been approved.

A possible relationship between lubricating properties of W/G fluids and the cloud point at higher temperatures has been checked. (polyglycol thickeners have an inverse solubility and come out of solution at higher temperatures.)

Vickers 4-Vane Pump Tests
350F - 0 & C

Fluid	Viscosity 100F Initial	168 hr	% Change	Acidity Initial	72 hr SAN	72 hr TAN	168 hr SAN	168 hr TAN
Reolube TF90	41.1cs	194cs	+372	0.09	0	2.5	11.9	34.
Reolube HYD 110	45.5	382	+741	0.05	0.71	6.3	6.6	27.
Tricresyl phosphate (Oronite) #1	45.8	11,960	+39000	0.07	2.14	15.2	12.5	49.
Tricresyl phosphate (Stauffer) #2	44.8	157	+250	0.06	14.2	81.1	7.5	26.
Tricresyl phosphate #1 with Bantolube 70 and Chevron 2-21	38.5	278	+621	0.07	1.71	10.3	11.9	39.
Houghto-safe 1120	47.6	59.2	+24.3	0.05	0.18	1.74	1.07	4.6

-32-

11293

Oxidized Propylene Esters

Beverage Bottle Hydrolytic Stability

	INSOLUBLES % wt.	VISCOSITY % CHANGE @ 100F	ACIDITY CHANGE, FLUID mg KOH/gm	ACIDITY, WATER, TOTAL mg KOH	CU STRIP WT. CHANGE mg/CM2	CU STRIP APPEARANCE (2 OX max)
#1 OR-142898-7 REOLUBE TF-90	0.024	−2.2	+0.31	1.78	0.12	med to light copper color - no etch or pitting
#2 OR-142898-8 REOLUBE HYD-110	0.018	−5.1	+0.06	1.56	0.56	brownish-magenta no etch or pitting
#3 OR-142898-9 CIBA TRIXYLYL PHOSPHATE (CAN 1)	0.048	−3.7	−0.07	2.62	0.20	med. copper color - no etch or pitting
#4 OR-142898-10 CIBA TRI-XYLYL PHOS. (CAN 2)	0.021	−16.7	+0.01	2.00	0.75	dull-reddish gray or brown coating but etch or pitting
MIL-L-9457	≤0.5	—	≤0.2	≤5	0.3 max	no pitting or corrosion color

11294

European Turbine Control Fluids With Flammability Tests

Fluid	U.S. Bu. Mines Pipe Cleaner Wick	Brown Boveri Asbestos Wick
Pydraul 150	11 cycles	ignites second cycle
Pydraul 135	32 cycles	ignites 9th cycle
Reolube TF90	20 cycles	>10 cycles - flash but no sustained burning.
Houghtosafe 1120	20 cycles	>15 cycles no ignition.

Results of this work presented in the following table indicate factors other than cloud point temperature affect lubrication.

Water/Glycol Cloud Point

Fluid	Cloud Temperature	Screening Pump Rating	Lubricity
Ucon Hydrolube 150CP	191-200F	1	excellent
Ucon Hydrolube 200NM Special	214	2	"
Ucon Hydrolube 150LT	205-212	3	"
Houghto-safe 520	215-220	4	"
Park W/G 100	193	5	good
Wyandotte FR, 150SUS	196-198	6	"
Mobil NYVAC FR 200	223	7	fair to poor
Wallaces Water glycol	90-95	8	"
Houghto-safe 620	>240	9	"
Jefferson FF-313	200-210	10	Very poor
Santo-safe W/G 15	214-218	—	—
Santo-safe W/G 20	214-218	—	—
Santo-safe W/G 30	214	—	—
Dow W/G Candidate	>230	—	—

11296

Phosphate Ester Hydraulic Fluids

Pump test results on the pilot plant prepared cumylphenyl nonylphenyl phosphate ester are listed below.

MCS-050E (OR 142228)(NCPDPP Ba. #1)- 220 SSU which was prepared by D. L. Kocher was pump tested in the V105C pump stand with excellent results. Total weight loss was less than 20 mgs which is comparable to Fyrquel 220.

MCS 050E (OR 139050) 220 SSU which is identical to the above formulation, but using Pilot Plant Batch #2 (NCPDPP) was prepared -

65% NCPDPP Ba. 2
35% TPP

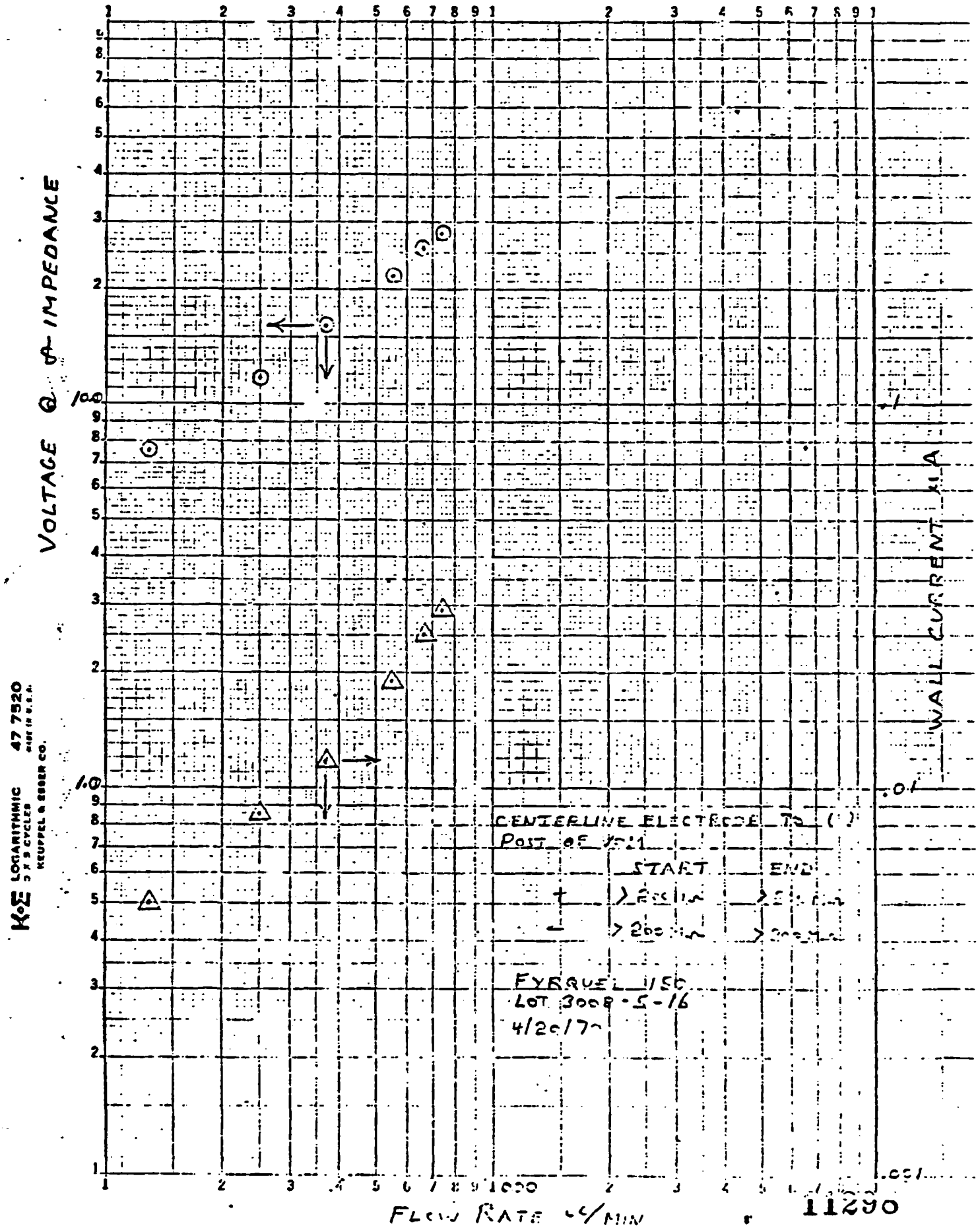
This formulation is presently being pump tested in the V105C pump. Preliminary inspection indicates a reproducible test.

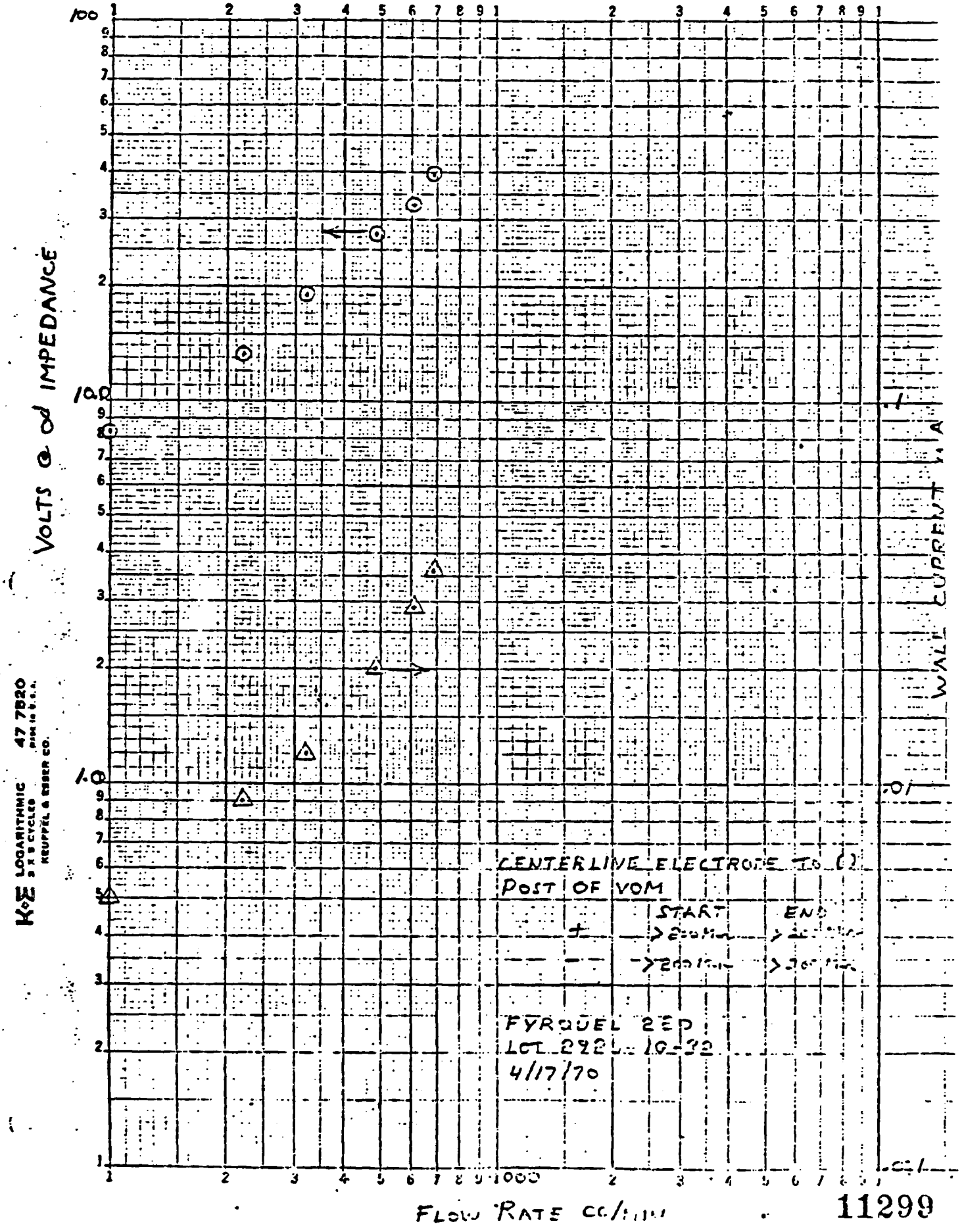
V105C Pump Stand "J" is presently being modified for testing phosphate ester fluids. MCS 030E is scheduled to be pump tested in this stand; start-up is scheduled week of 4-26-70.

Phosphate Ester Hydraulic Concepts (1315635-RLG)

Two base fluids NCPDPP and OXDPP, one fluid with a surfactant additive NCPDPP + 0.04% FC 98, and two competitive fluid formulations Fyrquel 220 and Fyrquel 150 were investigated on the Boeing streaming potential apparatus.

The results indicate that Fyrquel 150 and Fyrquel 220 are similar in streaming potential and wall current while NCPDPP has lower streaming potential and higher wall current. The addition of FC 98 reduces the streaming currents and potential. OXDPP has stream currents similar to the Fyrquels. The attached graphs have streaming potential and wall current data vs. volume flow rate through the apparatus.





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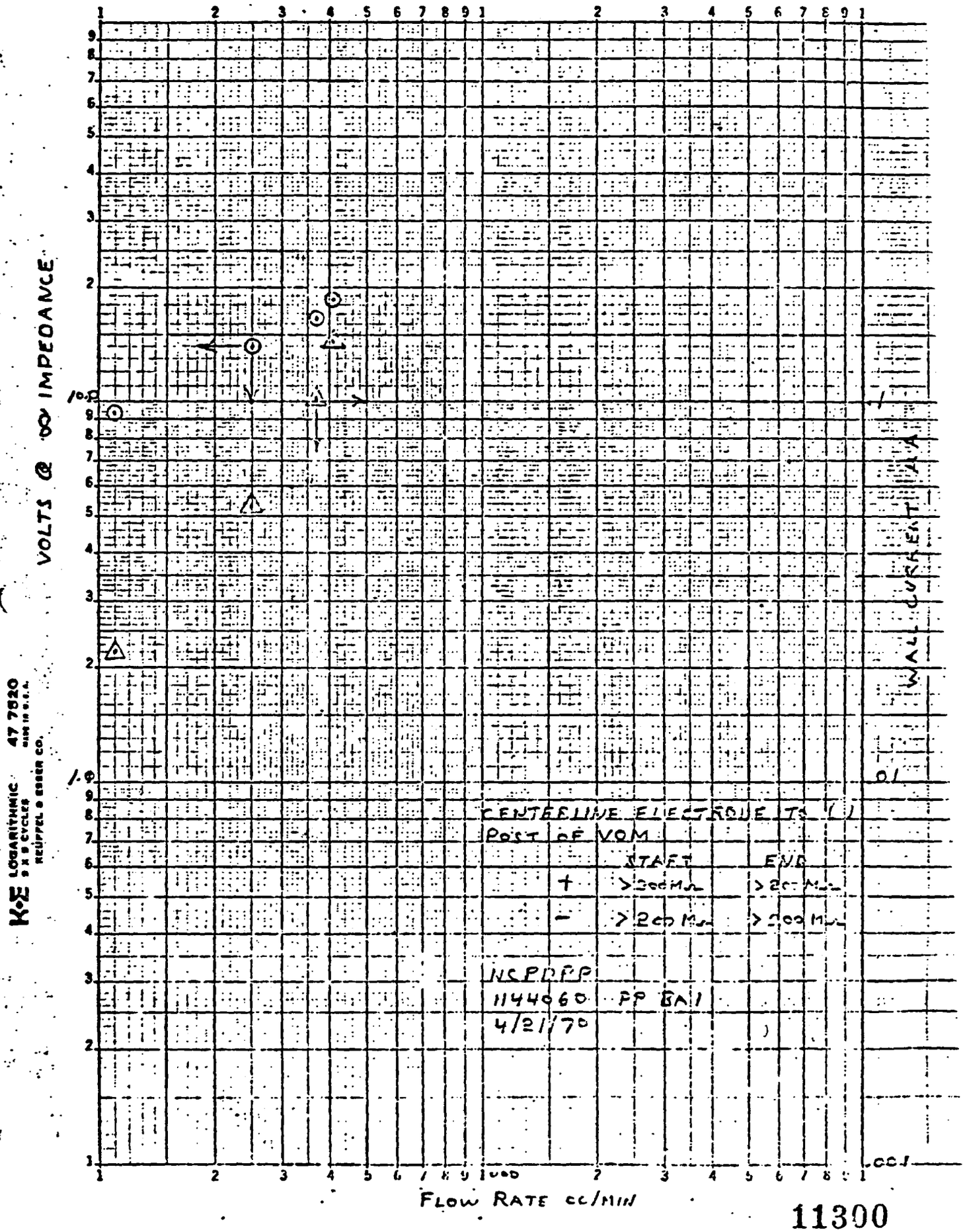

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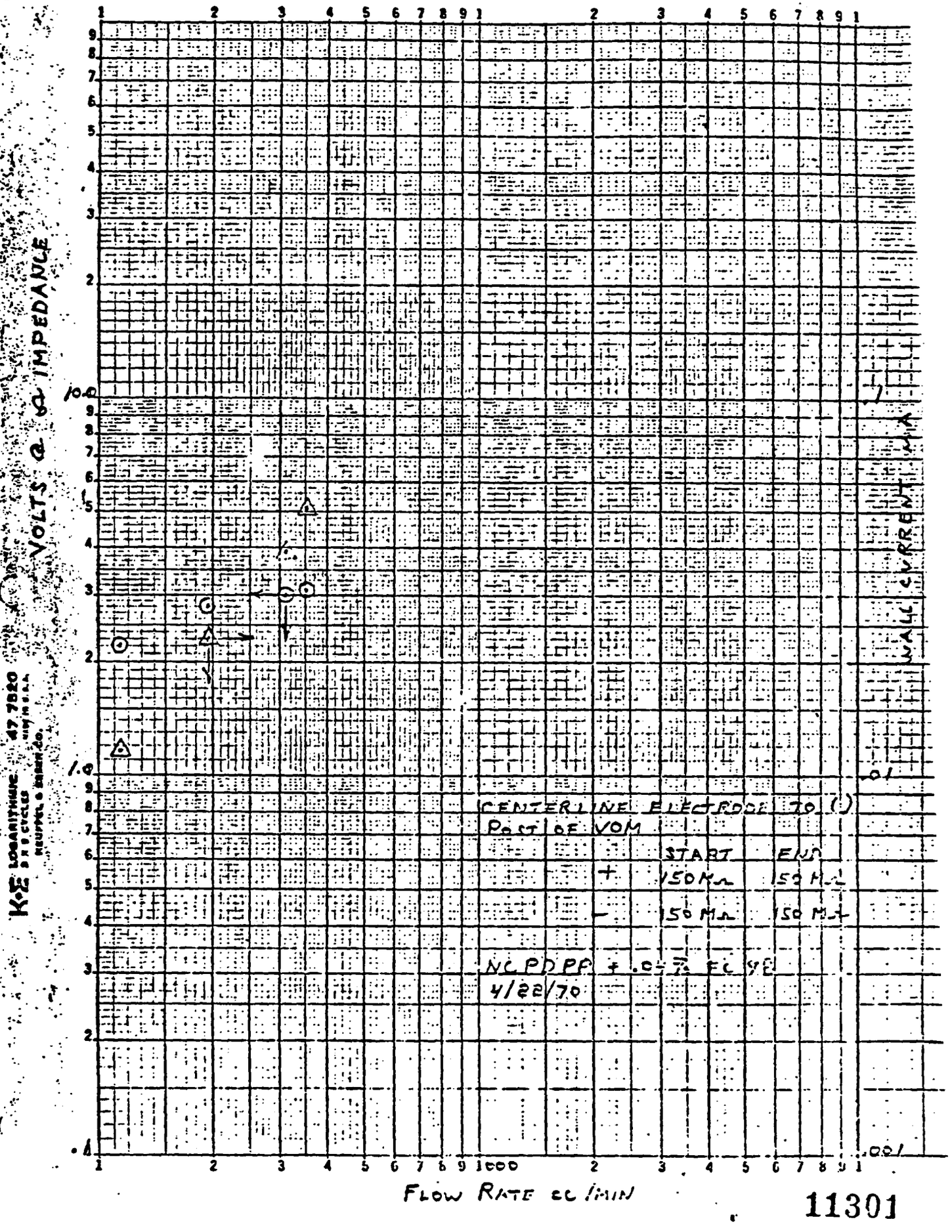

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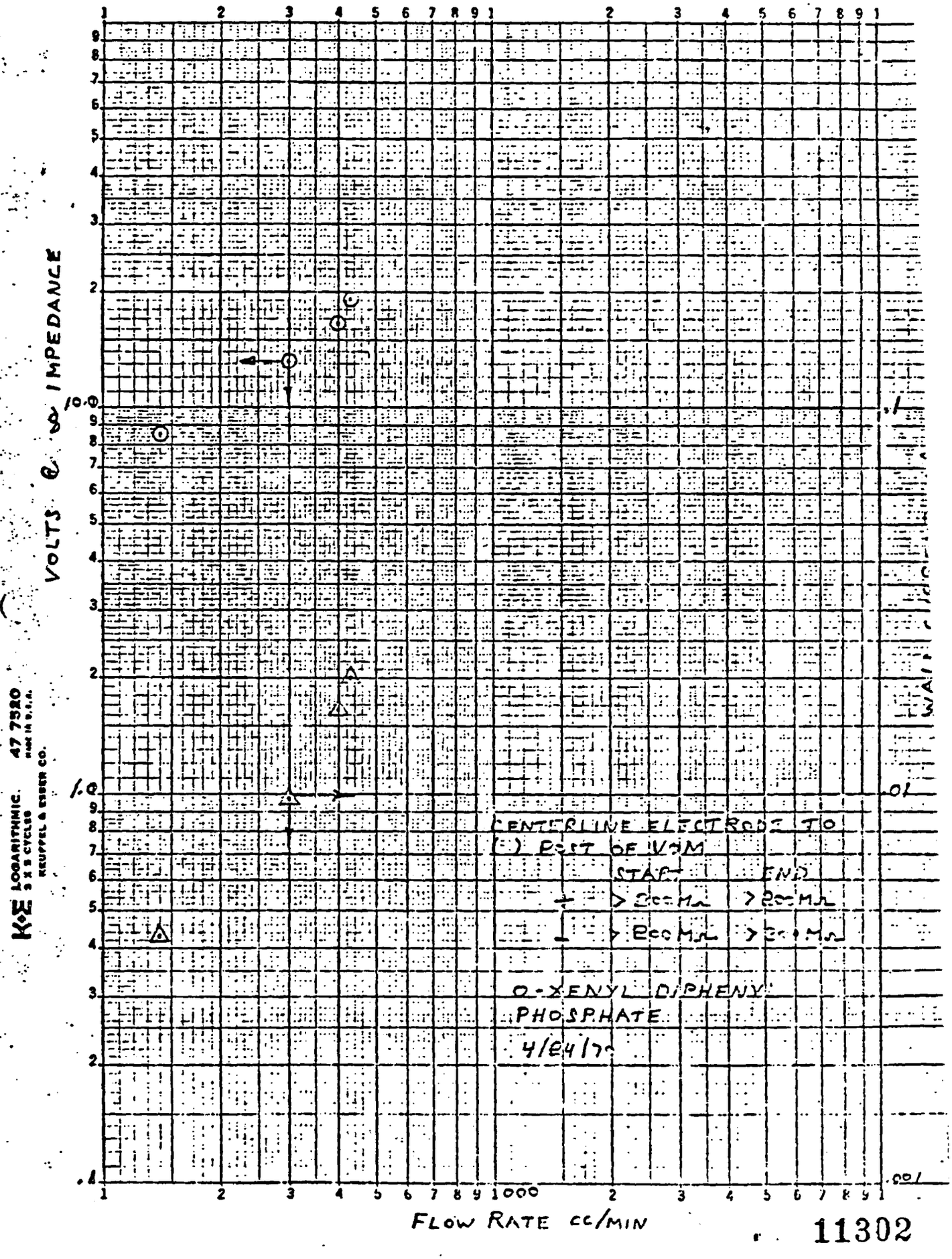


11302

Thermal Stability of Cumylphenol (P.P. SPL. 37) (H. Luebke & E.Emery)

INTRODUCTION

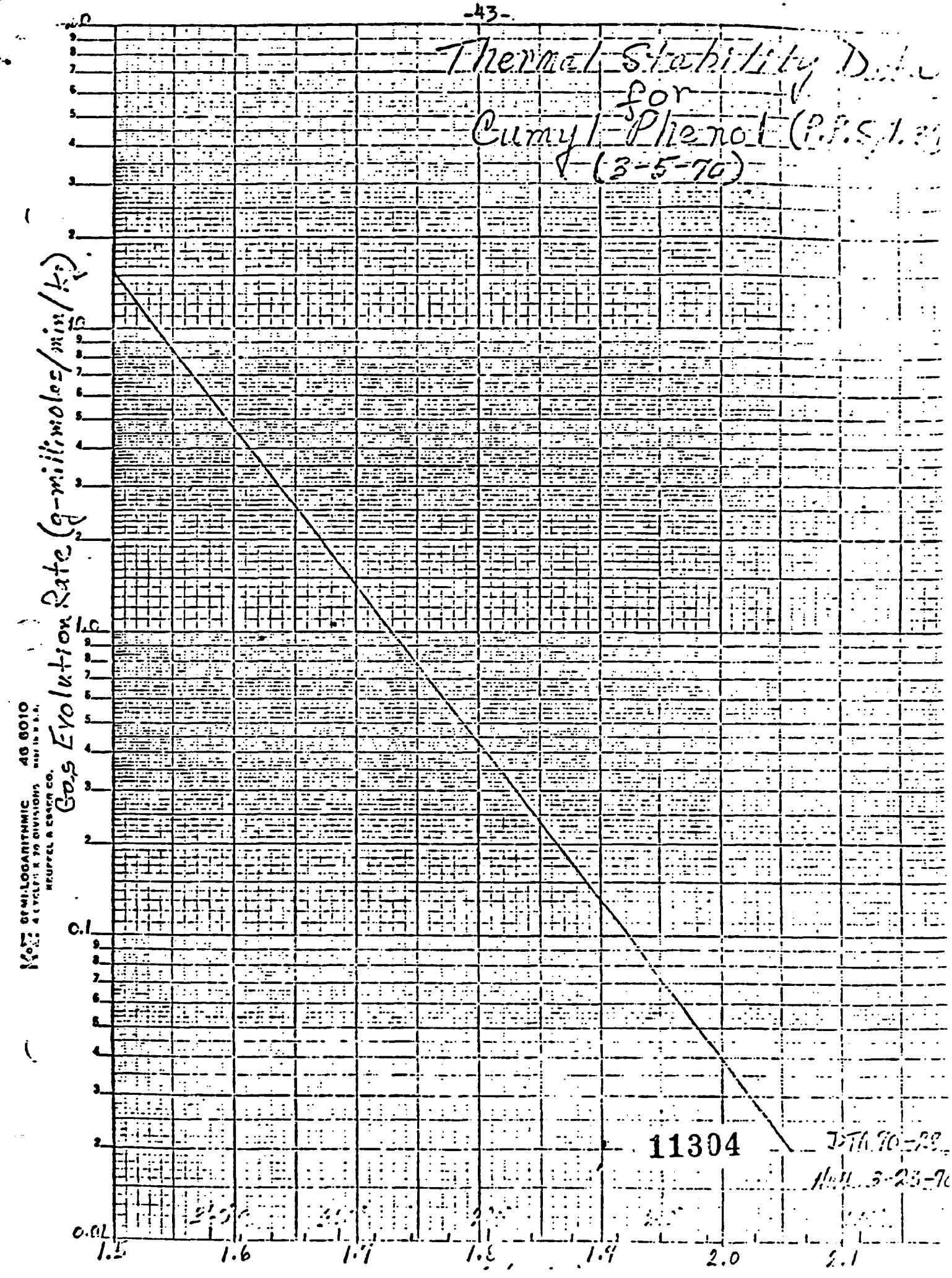
Thermal stability data for cumylphenol were requested to provide process safety information for operation in the Pilot Plant. A maximum pot temperature of about 280°C is used for the distillation.

SUMMARY

The thermal stability of a Pilot Plant sample of cumylphenol (Spl. #37, 3-5-70) has been determined with the Monsanto built pressure DTA apparatus. The experiment was run under a nitrogen atmosphere. Only a very minor exothermic reaction was observed above about 280°C. Gas evolution was detected at about 210°C and a rate of 0.4g - millimoles/min/Kg (9cc/min/Kg, STP) was measured at the maximum proposed pot temperature of 280°C. A plot of gas evolution rates vs temperature is attached. Gaseous decomposition products were identified by GC/MS as carbon dioxide, methane and 2 and 3 carbon hydrocarbon fragments.

We foresee no serious process hazards due to thermal decomposition of this compound. The rates of gas evolution are low and the slope of the curve is quite flat, indicating a small temperature coefficient.

Reference - DTA #70-28



Marketing Support

Citgo FR-23

A recent Citgo fluid similar to Pydraul 312 has been characterized as follows:

1) 65% Aroclor 1248
13% Phosphate ester; 4% triphenyl
9% cresyldiphenyl
10.5% Petroleum Oil
(Same MW & MW distribution as found in Citgo CHX-604)
1.5% VI improver, (same type as found in Citgo CHX-604)

2)

	Citgo CHX-604	Citgo FR-23
Vis at 100°F, cs:	70.93	71.6
Vis at 210°F, cs:	7.122	7.67
V.I.	48.4	71.5
Sp.G. at 25°C		1.28

3) FR of subject sample as defined by the 'Wick Test' (insufficient sample for normal FR determinations) indicates an improvement over Citgo CHX-604 which was in turn rated a definite pass by the F/M Hot Channel and H.P. Spray tests.

Basically we are dealing with the same components as found in their earlier fluid, CHX-604. The increased FR noted is undoubtedly due to less petroleum fraction as compared to the earlier fluid. (See 10/9/69 memo WRR from PFH)

Both of these fluids are in the 300 SUS range; however, the VI #s of FR-23, is roughly 20 points higher indicating a more potent VI improver and possibly more shear stable since it was reported that sheardown was a problem with the CHX-604 fluid. We do not plan to characterize the VI improver at this time.

It is our opinion that they have incorporated more potent and possibly a more shear stable VI improver and have reduced the oil content to achieve similar viscosities.

11305

Hamworthy Compressors/Pydraul 625

Three compressor parts from Hamworthy Compressors were tested for compatibility with Pydraul 625 and Pydraul 625A. The parts were of a perspex compound and the results were negative.

The parts were cut in half and then partially immersed in each fluid for 168 hrs at 158°F.

The parts did not change greatly in dimension but some dissolving did occur. This caused the fluid to become very slimy. Another part which was a gasket for a flow meter became very swollen and soft.

These parts would not be recommended with either fluid.

Recovered Pydraul 312/G.M. Bedford

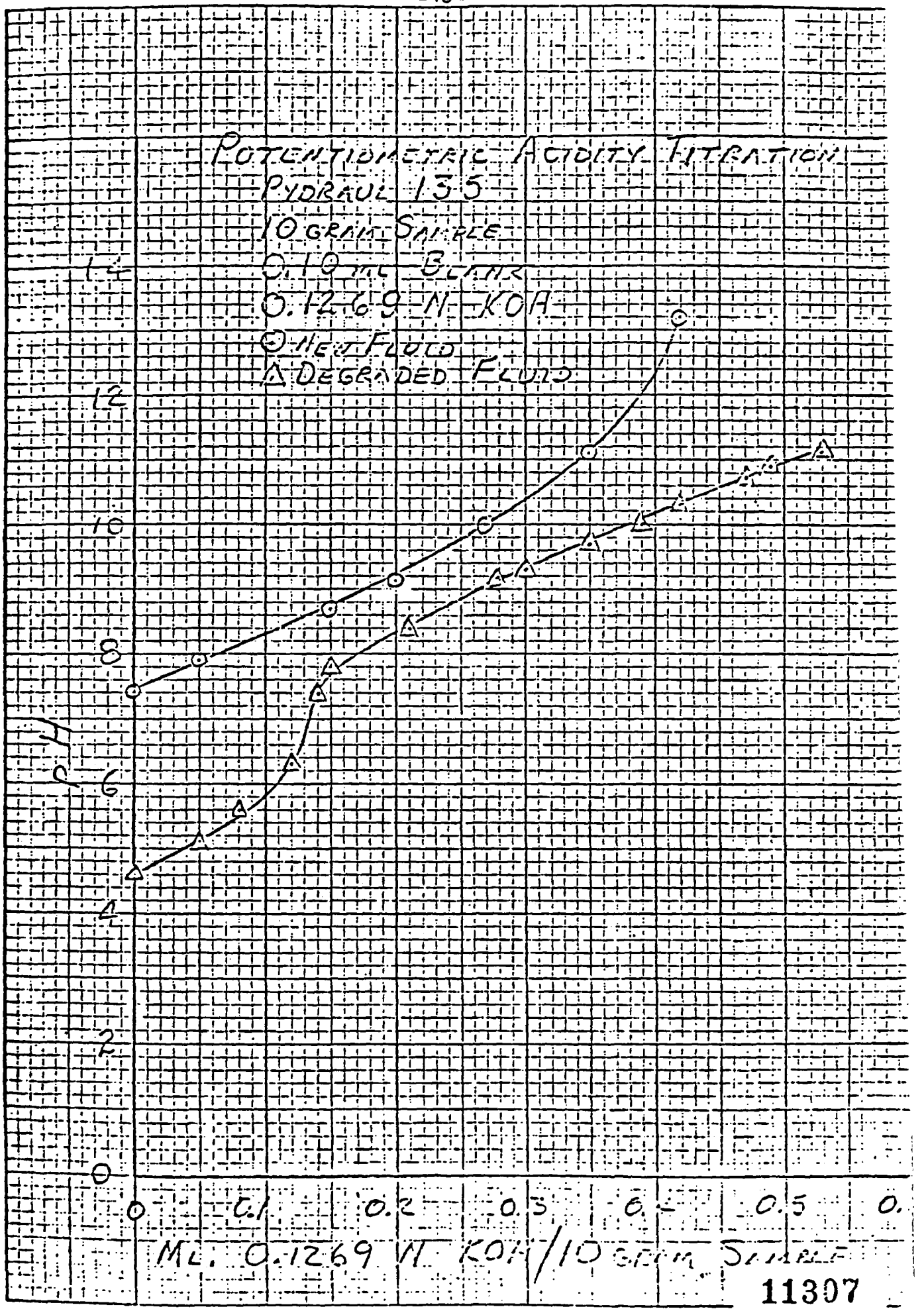
Recovered Pydraul 312, Findett lots CF 56 and CF 57 appear to be nearly equivalent to new Pydraul 312 as judged by the Factory Mutual Spray fire test.

Pydraul 135 Acidity/Republic Steel

Laboratory degraded Pydraul 135 and new fluid were furnished to R. R. Klinge of Republic Steel to serve as standards for measuring fluid acidity.

Attached are typical titration curves for these two samples which indicate the differences expected as fluid is degraded. The ASTM D-664 procedure was followed with the end point taken at pH 11. Total acid numbers (TAN) in milligrams of KOH per gram of fluid were calculated to be 0.17 for new fluid and 0.29 for the degraded fluid.

Two characteristics are noted for degraded fluid. The initial pH drops lower and may eventually get into the strong acid region below pH 4. Also, more weak acids are being formed and the titration curve flattens and extends to pH 11.



11307

Assistance to Manufacturing

Recovered Aroclor 1242 for Pydraul Blends

Specifications for Aroclor 1242 returned by G.E. and Westinghouse for use in Pydraul blends were suggested (see following page). Steps are being taken to allow the plant analytical lab and manufacturing department to work directly with Findett on approval of recovered fluids for use in Pydraul blends.

Pydraul 320 Customer Complaint

Gross separation of Pydraul 320 was reported in Japan. This appears to be caused by exposure to low temperatures. About 45% is in the top layer and 55 below. These layers remix readily at room temperature (75°F). Fire resistance of the two layers as judged by a pipe cleaner wick test is similar to the complete fluid. Interim recommendations will be to remix fluid at room temperature before using. Further attention will be given to this problem as these fluids are reformulated to eliminate PCB's.

Pydraul Acidity Specification Limits

Currently produced Santolube 70A has a slightly lower acidity than the old Santolube 70. This results in problems meeting minimum acidity spec limits for some Pydraul blends. Each 0.1% of Santolube 70A contributes 0.05 to 0.06 TAN to acidity of finished fluid. Specification limits will be changed as needed.

RECOVERED AROCLOR 1242 TENTATIVE SPECS FOR USE IN PYDRAUL BLENDS

		Recovered Aroclor 1242 for use in any Pydraul or Santosafe	Recovered Aroclor 1242 restricted to use in Pydraul 312 and Santosafe 300 Series*	JFQ Meth No.
Color, ASTM D-1500		1.0 max	3.0 max	--
Appearance, visual		Clear, light yellow liquid free of sediment.	Clear, yellow liquid free of sediment.	16
Neutralization Number, mgKOH/gm		0.03 max	0.03 max	8
Moisture, KF in methanol		100 ppm,** max.	100 ppm,** max.	7
Sp. Gravity 25/25°C		1.380-1.395	1.350-1.395	11
Viscosity, cs at 100°F		16.1-18.6	16.1-18.6	12
Oxirane Oxygen		0.1% max.	0.1% max.	*
GLC Analysis	a)	≤ 0.2% max area low boiler peaks near new 1242 peak envelope.	same	
	b)	no low boiler peaks far removed from new 1242 peak envelope.		
	c)	other peak typical of new 1242.		

* Includes Pydraul 312, 135, 790, 230, and 320; Santosafe 300, 150, and 100.

** Water contents as high as 180 ppm should make Pydraul with acceptable moisture and appearance; however at this water level Aroclor 1242 alone is very cloudy.

*** Procedures to be supplied later. Currently being run by Research.

11309

DIELECTRICS

Biodegradable Capacitor Fluids (R. H. Munch)

A modified Aroclor 1242 designated Aroclor 1242B has been produced by fractionating Aroclor 1242 to reduce the penta and tetra chlorinated components to a minimum value. The new product has only .16% penta, 2.3% of the amount in standard Aroclor 1242 and 16.3% tetra, 67% of the amount in the regular material. Biodegradability data for the new product are tabulated below along with similar data for standard Aroclor 1242 for comparison.

Biodegradability

	Peak 1	Peak 2	Peak 3	Peak 4
Aroclor 1242 Amount Remaining (after 1 week)	Nil	66%	64%	42%
Aroclor 1242B Amount Remaining (after 1 week)	Nil	10%	33%	1%

These data seem to indicate that the Aroclor 1242B is significantly more biodegradable than Aroclor 1242. The attached sheet gives the specification properties for Aroclor 1242. Those which have been determined for Aroclor 1242B are given on the same sheet. These data indicate that the new fluid could be substituted for the old in capacitors with no modifications in capacitor design or processing methods required.

Arrangements have been made to produce 500 to 1,000 gal. of the Aroclor 1242B. The fractionation is to be carried out in the Dayton MRC pilot plant because no suitable still exists in our St. Louis pilot plant. The adsorbent treatment will be done in the St. Louis pilot plant if the truck strike does not interfere. The large sample of the new product is to permit use tests in capacitors by our customers.

Preliminary work indicates that a product having amounts of penta chlorinated biphenyl too low to detect and tetras reduced to about 1.3% can be produced by fractionating Aroclor 1132. The fractionation must be done so as to reduce the concentration of the 4,4' dichlorobiphenyl isomer to 2.0-2.3%. If this is not done crystallization takes place. Further work will be done to determine the best procedure for making a product of this type.

Monsanto
ORGANIC CHEMICALS DIVISION

ANNISTON AND KRUMMRICH
FINISHED PRODUCT SPECIFICATION

PRODUCT -50- Aroclor 1242, Electrical Grade | DEPT. 246 27

PLANT	PRODUCT CODE	USE
WGK, Ann.	81101 - 82612	Sales

DATE EFFECTIVE	SALES CODE
6/17/66	1040-240-04/11/16-03/09

APPROVALS

PRODUCTION	RESEARCH
P. E. Heisler	R. W. Bucknell
SALES	QUALITY CONTROL (Chief Chemist)
W. E. Ault	W. J. Gresham O. E. Dolin

GENERAL DESCRIPTION

Also known as Pyranol 1499

CHEMICAL FORMULA

MOL WT.

SUPERSEDES SPECS. OF	SAMPLE FOR ANALYSIS	ISSUED BY
4-13-62	3 x 5 pt. Amber Bottles	J. P. Prader

	CHARACTERISTIC	LIMITS		METHOD WGK	METHOD Anniston
	UNRESTRICTED INFORMATION	1242	1242B		
(R)	Color (APHA)	40, max.		10084	14-44-54
(R)	Condition	Clear		10084	G-1
(R)	Sp. Gr. @ 25/15.5°C.	1.381 - 1.392	1.367	10114	14-49-54
(R)	Acidity (mg. KOH/g.)	0.010, max.		10087	14-42-54
(R)	Moisture (H_2O)	35 ppm., max.		10620	14-53-54
(R)	Refractive Index @25°C.	1.6240 - 1.6260	1.6221	10125	14-34-54
(R)	Inorganic (Free) Chlorides	0.05 ppm., max.		12741	014
(R)	Pour Point	-14°C. or Lower	-14°C	10115	14-47-54
(R)	Dielectric Constant (1000 cycles @ 100°C.)	4.70 - 4.90	4.47-60Hz	11608	14-36-54
(R)	Resistivity (500 VDC @ 100°C. 0.1" Gap) As OHM-CM x 10^9	500, min.	$24,000 \times 10^9$ Dissipation Factor-60H=,100 .0009	11607	14-35-57
(R)	Hydrolysis Stability Test (As Chlorides)	0.5 ppm, max.		12672	010
(R)	Monsanto Stability Test (As Chlorides)	0.4 ppm., max.		11503	14-61-57
(R)	Distillation Range (ASTM D-20, Corrected)			10117	14-31-54
	10% Distilled by wt.	325°C., min.	324°C		
	90% Distilled by wt.	360°C., max.	339°C		
	Sulfates	None		12169	------
	Dielectric Strength @ 25°C.	35 KV, min.		11605	043
	Flash Point (°F)	338 - 392.		10123	14-28-54
	Fire Point	None		10123	14-28-54
	Corrosion Test (6 hrs. @ 210°C. with bright aluminum foil)			10126	14-55-57
	Change in wt. of AL %	0.0			
	A.C. Color (APHA)	60, max.		10084	14-44-54
	A.C. Condition	Clear		10084	G-1
	A.C. Acidity (mg. KOH/g.)	0.010, max.		10087	14-42-54
	A.C. Inorganic (Free) Chlorides	0.05 ppm., max.		12741	014
	Visc. @ 100°F. (SUS)	82 - 92	74.4 SUS	10086	14-44-52

(Over)

11311

GENERAL INFORMATION

Specific Heat @ 25°C.	0.30	
Coefficient of Expansion (25 - 65°C)	0.00068 cc/°C.	
Fixed Chlorine	42.5 - 53.5%	41.3%
Power Factor @ 100°C.	(Typical Values)	
60 cycles	5	.01%
1000 cycles	0.30	

SPECIAL CUSTOMER REQUIREMENTS

General Electric

Thermal Chemical Chlorides	0.65 ppm., max.	12722

Westinghouse

Hydrolizable Chlorine (Westinghouse Stability Test)	1.0 ppm., max.	T-2219

11312

Synthesis of Capacitor Fluids (1630459-QET)

n-Butyl pentachlorophenyl ether, synthesized last month, had an unexpectedly low power factor along with a reasonable dielectric constant. We are following up this lead by preparing butyl ethers of various lower chlorinated phenols and by modifying the alkyl group in pentachlorophenyl ethers. Compounds prepared to date include n-butyl-2,4-dichlorophenyl ether n-butyl-2,4,6-trichlorophenyl ether and n-heptyl pentachlorophenyl ether. The latter melts at 8°C while n-butyl pentachlorophenyl ether melts at 16°C. The relatively high melting points of the pentachlorophenyl ethers indicate a necessity for use of mixtures or a significant modification of the alkyl portion of the molecule to lower the melting points.

Capacitor Fluids (RWW)

A second batch of dichlorodiphenyl ether (3 Kg) was prepared. Yield is considerably improved (70%) but all too low. Air sparging after chlorination is suspected to cause polymer formation. This will be checked out on a smaller scale preparation.

Electrical properties for chlorinated α-methylstyrene dimers are attached. The dichloro derivative showed good resistivity and dielectric constant; however, the pour point is much too high. We will try blending to improve low temperature properties.

Aroclor Defense (RWW)

Only one experiment in the hot tube without catalyst was run. Very little isomerization of 4,4'-dichlorobiphenyl was noted.

A Suggested Procedure was written for bromine recovery from HBr off-gas in Bromo Aroclor production. The process comprises scrubbing demisted HBr with agitated calcium hydroxide slurry to pH 4-5, oxidizing with chlorine and steaming out bromine. Saturated bromine with 0.05% water is withdrawn from bottom of receiver and dried with activated calcined alumina to 20 ppm water level. The aqueous supernatant from the receiver is recycled. Overall bromine recovery will be better than 98%. This process is preferred over oxidation of the much more corrosive HBr.

Industrial Hydraulic Fluids and New Dielectric Fluids

A new batch of Bromo Aroclor 1232 is prepared by bromination of Aroclor 1223. This material exceeded desired Aroclor equivalence (~1237) and will be blended in new Pydraul 312. Properties are summarized in Table II. This material will be purified and tested for electrical properties.

Table I

Electrical Properties of Chlorinated Acid $AlCl_3$ Dimer at 100°C

	Dissipation Factor	Resistivity $\times 10^{12}$ at 500 V	Dk	Power Factor %
Monochloro-	0.00060	1630	3.66	+15
Dichloro-	.00126	674	4.48	+45
Target	$\leq$ .001	$\geq$ 10	5	-40

Table II

Physical Properties of Arochlor

	Viscosities in cs		Density	Arochlor
	100°	210°	d_{25}^{25}	"Equivalent"
1231	5.12	1.37	1.281	1236
1233	5.53	1.42	1.305	1240

Dielectric Liquids - H. Vodden, C. Coleman - RUABON

760.12.722.51
Electrical Fluids [illegible].

A visit to Micafil, Zurich elucidated the situation in respect of their licencees. Voltas, India are already in production and were said to be using Prodelec impregnants. Elektrosrbija, Beograd are not in production and are not expected to be for perhaps 2 years. [illegible]nussi, Italy and [illegible], [illegible] have no connection with Micafil.

[illegible], Bucharest was visited to find out the reason for their discontent with Monsanto. Their main reason was the allegedly high dissipation factors obtained on drum deliveries of Aroclor 1248. This was said to be higher than on competitive products from Bayer and Prodelec, and also to be higher than the value of 1.[illegible] quoted as a typical value in our [illegible]. A value of [illegible].8 at 90°C, 50Hz was said to have been obtained on 2[illegible].7[illegible]. A sample was brought back, and although the results on this are not conclusive, they tend to indicat that our dissipation factor is higher than is desirable or expected. It is recommended that the dissipation factor of future consignments to this customer should be checked before being dispatched so that we know the value at this sta

Two [illegible] dielectric test cells have been prepared for dispatch to Heavy Electricals, India. This exhausts our present stock and it is not proposed to have any more made because of the change over by [illegible] to the [illegible]nch method of measurement using the Balsbaugh cells.

760.12.022.44
Electrical Plastic Films.

A further meeting with [illegible] took place in London on 1[illegible].7[illegible]. It was again emphasised that [illegible] require Monsanto set specifications for poly[illegible] film because of the cost of their development work. It was agreed that this would [illegible] completed by the end of April.

In the meantime we have experienced a setback in the testing of the compatibility of the [illegible] experimental samples. Lack of reproducibility was obtained and the cause has been traced to the electrode cleaning sequence, but not positively identified. As a temporary expedient, with the pressing urgenc of this work, the test is being carried out using the [illegible] cell.

A visit to [illegible] Research, London was made for the purpose of seeing their capacitor winding, impregnating and testing equipment. As a result of their recommendations, Filmcap, near [illegible] was visited in order to see their design of winding machine in operation. These machines appear to fulfil all our forseeable requirements both for plastics film and [illegible] capacitors.

[illegible] [illegible] impregnation [illegible]
is not yet ready.

760.12.022.44
Electrical Plastics Films.

Some progress has been made on testing the samples submitted by [illegible] and 12 samples out of 44 have been tested to date. Poor repeatability is being experienced and present efforts are directed to find the cause of this. Dissipation factor is being measured at 500 volts because the sensitivity of the Unwood bridge is insufficient at 50 volts. Delivery of the new 701 bridge is awaited.

A meeting with BCL took place on 17.2.70. They wanted details of costs to equip themselves for carrying out a complete range of tests on their electrical films, including compatibility with Aroclor. They also intend to set up a capacitor testing operation. They require from Monsanto a complete specification for electrical film so that they can manufacture to this. They also require Monsanto to tell them the thickness and type of film where the market opportunities will be most favourable, bearing in mind that they are late in the field and their competitors have already established themselves in thicknesses of 10 microns and above. They incline to the view that the greatest opportunities lie with capacitors with a single layer of metallized film but impregnated rather than dry. They are particularly interested in Monsanto's developments in liquids that are suitable for use under self-healing conditions in such capacitors.

760.12.022.46
Miscellaneous Developments.

A visit to BRA disclosed that negligible progress has been made on our privately-sponsored project to test MCS 717 and an Aroclor 1242/[illegible] blend in polypropylene film capacitors. The reason given was delays in setting up their impregnation system. The results were promised for the end of April.

One aspect of their generally-sponsored project covers the testing of scavengers in askarels. In addition to the small sample of Pyranol 2 that we have supplied, they will require a quantity of 40 - 50 gallons for larger trials. At present we are unable to supply this because it is too much to supply from the laboratory and no pilot plant is available.

760.12.022.45.
Alternatives to Aroclors.

Three further modified samples have been received for electrical tests. We were told that attempts to refine the previous sample have not been successful.

760.12.722.50.
Electrical Fluids MTS Committee Work.

A meeting of BNA WG154 was attended in London. This meeting was mostly concerned with silicone fluids.

A 2 day meeting of BITA in Frankfurt was attended. This was solely concerned with toxicity problems and the matter of specifications, which concerned Pydelec, was not discussed. Jay intends to deal with this matter by correspondence.

11316

[illegible]
[illegible]

[illegible] have [illegible] these materials have been [illegible] be carried out over the next [illegible] within the next two weeks.

[illegible] his training and [illegible].

Job No. 760.12.722.51.
Electrical Fluids CTS.

The following customers were visited:-

1. ICAR, Monza, Italy: They have tested our impregnants A275 and A230 in their aluminium metallized polypropylene film capacitors and the results are unsatisfactory. It was found that corona discharges eroded the metal coatings resulting in a reduction in capacitance during ageing tests. The mechanism of this effect is not readily apparent and further investigation is required.

2. Zanussi, Pordenore, Italy: They requested further information to enable them to change from Aroclor 1254 to 1242. They also want a sample of purified Unox 206 sufficient to 'dope' 25 gallons of Aroclor 1242 for a trial. They do not use polypropylene film.

3. CGE, Milan, Italy: They prefer to continue to use Aroclor 1242 from the USA. They use P2 for motor capacitors using paper now but are looking at polypropylene film both metallized and non-metallized. They are expanding rapidly and may produce power factor correction capacitors in the future.

4. BICC, Helsby, England: Met Richardson during a visit for another purpose and he asked if I knew about a new Prodelec product called 'Metaline' with a DK of 4.2. He also mentioned that Newport dissipation factor readings were high compared with theirs, 2 to 3% for Newport corresponding to 1 to 1.5% at BICC.

5. LIFASA, Barcelona, Spain: Requested further information on design of a purification column and suppliers of components for a measuring system. A small manufacturer, but highly advanced technically, who uses Aroclor 1242 with Superad.

6. Bianchi, San Sebastian, Spain: Having problems with dust contamination so wish to defer change to 1242 until these are overcome. Requested details of automatic machine for injecting epoxy resin capacitor seals. Wanted further visit when they change to 1242 said to be 8 months hence. Advised them to run pilot trials to test sealing. Don't use polypropylene film.

11317

7. Fecha, Madrid, Spain: Having so much trouble with Aroclor 1242 that they are considering changing back to 1254. Use Superad with Aroclor also. Have made trials with polypropylene film from La Cellophane and Kalle but not using it at present. Personnel difficulties are hindering progress.

8. Cenemesa, Cordoba, Spain: Introduced Pyroclor 5 and after initial adverse reaction they appeared to accept what satisfactory experience of Westinghouse Canada indicated that it would be acceptable. Will place an initial order for trial quantity and make measurements on a transformer. Requested information on a source of supply of epoxy resin impregnated paper for transformers.

BEAMA Conference on insulating materials held at Earl's Court, London during the Electrical Engineer's Exhibition was visited and a paper entitled 'Transformer Askarels' was read. Only interest was from Castrol! Other papers on polypropylene film in capacitors aroused considerably more interest and many capacitor manufacturers from UK and overseas attended.

Laboratory work: The following TSLR reports have been issued:-

L7003-1611: Heavy Electricals, India - Assembly and calibration of 2 MCL MkIII dielectric test cells. This exhausts present stocks of components and it is not proposed to order further stocks in view of the change to the Munch Method.

L7001-1611: Insulating Sleeves and Tapes Ltd.
L7001-2711: Westinghouse Brakd and Signal Co. Ltd.
L7002-1711: Bruce Peebles Ltd.

RTW and ADM visited Newport plant for 1 day and 4 days respectively in order to carry out tests using their ESI 701 bridge and compare results obtained at Ruabon. This is part of the project to attempt to reduce excessive variability now being observed at Ruabon when following the Munch method of measuring dissipation factor.

Job No. 760.12.022.44.
Electrical plastic films.

The work of testing the BCL samples continues and about 30% have been completed. In view of the difficulties in obtaining reproducible results with the Balsbaugh cells and the low rate of testing, we have temporarily reverted to using the MCL cell and cleaning procedure. The solution method No.2 is still being followed.

We have been given a suggested limit for compatibility of the film with Aroclor using this method, but the other properties of the film which are required to draft a specification for BCL are still in doubt.

The visit to Italy was made in the company of Mr. J.L. Linsley-Hood of BCL who provided some samples of 8 micron film for the 3 manufacturers. A further sample of 8 micron film for ICAR is still awaited from BCL. ICAR did not give details of their requirements or prices paid.

There was no serious requirement for film at this time from the Spanish manufacturers visited.

BICC have offered us at scrap value 2 winding machines that they no longer require and this is being followed up.

CRL

ICAR Metallized Plastic Film Capacitor Fluid (RHM)

Icar has inquired about the availability of 2-butoxyethyl-2-chlorophenyl ether and 2-dichlorophenoxy methyl tetrahydrofuran, presumably for trial in their metallized plastic film capacitor project. These are fluids which Monsanto patented as dielectric fluids. Icar apparently discovered our patents while searching the patent literature looking for leads to help solve their problem.

I have secured copies of our patents and read them carefully. I find that the 2-butoxyethyl-2-chlorophenyl ether is listed as having a dielectric constant of about 5.8 and a power factor of 0.3% measured at 100°C and 10K Hz. It would therefore probably have a power factor of about 60% at 100°C 50 Hz. The 2(dichlorophenoxymethyl)tetrahydrofuran was found to have a dielectric constant of 6.3 and a power factor of 1.7% at 100°C and 10K Hz, or about 340% at 50 Hz. If my assumption that Icar wants to make capacitors for use at power line frequencies is correct, they would have no interest in such a material. Moreover, neither of these materials is available even in sample quantities.

Because it has been our feeling that minimum gassing under electrical stress was one of the important requirements of the Icar application, we have subjected a number of our materials to ASTM D2298-64T which is designed to determine stability of insulating oils under electrical stress. The data obtained are shown in Table I. Included in the table are data for Dialla Oil, a transformer oil, and Wemco C, a capacitor oil. Duplicate runs were made for each sample. The table gives the initial dissipation factor and resistivity for each sample, the dissipation factor and resistivity for each at the end of the experiment, and the average gassing coefficient in microliters per minute (AGC).

The data in Table I shed some light on the problem of selecting a fluid suitable for use in a metallized paper or film capacitor where the capacitor is expected to be self clearing by burning the thin evaporated film away from the area of a short circuit. There are two requirements.

First, minimum degradation of the dissipation factor and resistivity of the fluid. Second, minimum gas formation. If the degradation of dissipation factor and resistivity is too great the fluid in the vicinity of the fault will become conducting and the capacitor will fail completely. Data in the table show that the resistivity of Aroclor 1242 decreased by a factor of about 6×10^6 in this test. This is the reason a chlorinated impregnant is not suitable.

The other requirement is that the amount of permanent gas formed during the clearing process must not be enough to form a permanent gas bubble which would lead to breakdown. Thus, of the materials listed in Table I, Dialla Oil would be the least desirable. HB40, and mono isopropyl biphenyl would be relatively desirable, with HB40 being the more desirable because its resistivity is less degraded than that of the mono isopropyl biphenyl.

Of course other factors would influence the choice in practice. There may also be some question as to the validity of ASTM D2298-64T for this purpose. However, I don't know of a better test except capacitor manufacture.

I believe that we have done about all that we can do to help Icar in their search for an impregnant. If we want to try to do more we should have direct contact between their technical people and ours to make certain that we have correctly understood their problem.

TABLE I

STABILITY OF INSULATING OILS UNDER ELECTRICAL STRESS BY ASTM D2298-64T

	D.F. @ 100°C 60 Hz		Rs, 500V 100°C		Aroclor Gassing Test
	Initial	Final	Initial	Final	AGC
Dialla Oil (Transformer Oil)	.00054	.09936 .11136	519 x 10^{12}	.41 x 10^{12} .44 x 10^{12}	91.9 90.3
Wemco C (Typical Capacitor Oil)	.00072	.01374 .03918	52 x 10^{12}	4 x 10^{12} 1.4 x 10^{12}	58.6 60.2
A-215 (Alkyl Benzene)	.00012	.041* .50844	28,349 x 10^{12}	.09 x 10^{12} 1.27 x 10^{12}	22.1 22.1
Mono Isopropyl Biphenyl	.02910	.167* .161*	0.74 x 10^{12}	.01 x 10^{12} .01 x 10^{12}	1.6 2.4
HB-40	.00102	.60120 .50628	393 x 10^{12}	.25 x 10^{12} .31 x 10^{12}	1.6 1.6
Aroclor 1242	.00060	.58** .59**	170 x 10^{12}	.00003 x 10^{12} .00008 x 10^{12}	1.6 1.6

* Measured at 1K Hz
** Measured at 10K Hz

-90-

11321

AROCLOR DEFENSE (EST)

PCB, chloride, chlorine and total organic chlorine analysis of the gas samples collected during the thermal oxidation studies at the John Zink Co. established that Aroclor 1242, Inerteen PPO, and Aroclor 1260 can be successfully disposed of in this manner. The preliminary data has been sent to CRD and a report is being issued.

Sixteen sediment and water samples have been received from WGK for PCB analysis. Their analysis will be completed by May 15. The WGK analytical laboratory has ordered the necessary gear which will allow them to do their own PCB analysis and are scheduled to be on stream by July 1. Along these lines we will familiarize one of their personnel with the required techniques during the month of May.

Two 7-lb Mississippi River carp have also been supplied to us by WGK and are being prepared for PCB analysis. Samples of these fish will be returned to WGK for mercury analysis. It is also planned to examine the carp extracts by GC/Mass to see what others chemical residues can be observed.

Water and sediment samples from six accounts employing Pydraul fluids have been analyzed for PCB's. The results indicate that all plants are discharging low ppb to high ppm amounts of PCB's to associated sewers, streams, and lakes. The companies so far monitored all Johnson Motors (Waukegan, Ill.), Lester Castings (Bedford, Ohio), Central Diecasting (Chicago, Ill.), Wheeling-Pittsburgh Steel (Steubenville, Ohio,), Wisconsin Steel (Chicago,Ill), and Timken Steel (Canton, Ohio). This information is being generated to help guide a potential recovery and recycle program evaluation by Fluids' Marketing.

Muratic acid (HCl) from Monsanto Central Drumming has been analyzed by EC/GC for PCB's. Low levels of compounds having similar retention times to the lighter chlorinated biphenyls were observed. A larger sample has been extracted for GC/Mass confirmation of the suspicioned presence of PCB's. Two other products, food grade fumaric acid and saccharin are also presently being analyzed for PCB contamination. These results should be available early this month. If positive, no doubt, other products will have to be examined closely.

GC/Mass and EC/GC analysis of silo paint scrapings and raw milk extracts submitted to us by the State of Ohio, Dept. of Agriculture, Foods, Dairies and Drugs Laboratory confirmed the presence of PCB's in these materials. The silo scrapings contained 2.87% unaltered Aroclor 1254, the raw milk extracts contained an estimated PCB level of 0.6-0.7 ppm. Although the isomer distribution of the PCB's in the milk was altered with respect to Aroclor 1254, the isomers present were those that occur predominately in Aroclor 1254. Strikingly similar isomer alterations have been observed earlier in the electron capture chromatograms of an extract of a human fat biopsy sample, supplied to us by WARF Institute and in extracts of

raw milk samples supplied to us by the Maryland Cooperative Milk Producers. Both organizations had suspicioned PCB contamination; unfortunately, in both cases GC/Mass confirmation could not be obtained because of the limited amount of sample available (WARF) or the low level of contamination (MCMP).

The Bio-Test Laboratories has been contacted to arrange for the transfer of the tissues from various feeding studies they have conducted for PCB residue analysis. A rough estimate from them indicates that they have approximately 1000 cu. ft. of freezer space that is now filled. For this reason it was requested that a laundry list be prepared so that selected samples could be sent. We presently have only about 30 cu. ft. of storage space in St. Louis and see no reason to duplicate their facilities.

Air samples from the Appleton Coated Paper Co., Appleton, Wis. have been received and analyzed for PCB's. Low levels were detected, but the amounts present in terms of mg/m³ cannot be calculated until the actual number of liters of air passed through the scrubbers is obtained from them. If their sample sizes were on the order of 100 l. or more, the calculated amounts will probably be less than the recommended "safe" level.

An informational PCB problem review of the analytical methods and the data so far generated was held for representatives from Westinghouse.

A first quarter report has been written reviewing the results of the preliminary river die away and some continuous activitated sludge degradation of Aroclor 1221, 1242, 1248, 1254, and Arobrom 1228. In brief, the results were as follows, the only Aroclor which was degraded to any extent in the river die away experiments was Aroclor 1221.

Isomer alterations accompanied by the disappearance of 0.3-1.0 mg per 24 hr. periods were observed with all Aroclors (compared to 20 mg of LAS Dodecene-1 which is consumed in 24 hrs) feed to the semi-continuous activitated sludge units. The actual interpretation of this data awaits the evaluation of non-biodegradation routes which could account for the bulk of the Aroclor lost. This experiment is currently in progress.

In the past these experiments (adsorption, volatilization, etc.) have been hampered considerably by our inability to analyze the large number of samples required to obtain enough data points to compensate for the large variance generally encountered in biological experiments. In the future, this should be compensated for by the additional manpower which has been assigned to this portion of the project. The Physical Chemistry Group is currently setting up the necessary equipment to carry out biodegradation studies on Aroclors and Aroclor replacement products.

Electron capture chromatograms of recombined laboratory distillation cuts of Aroclor 1130 and 1142 (Aroclor 1242B) indicate that most of the "refractory" PCB isomers can be removed in this manner. Figure I compares the EC chromatograms of these potential substitutes with our current Aroclor 1242 production material. Figure II shows the isomer alteration that can occur via degradation of Aroclor 1242 in the semi-continuous activitated sludge units. Figure III shows the isomer alteration that occurs in a mammalian system (cow). It is important to note that the isomers present in the fractionated materials are those that appear least "refractory".

The following additional reports have been written and will be issued soon.

1. Relative Retention Times and Electron Capture Response Factors of Some Polychlorinated Biphenyl Isomers Relative to DDE.
2. A Study of the Completeness of Thermal Oxidation of Polychlorinated Biphenyls at John Zink Pollution Research.
3. The Partition of PCB Between Hexane and Various Organic Polar Solvents.
4. Extraction of Aroclor 1254 From Painted Surfaces by Water.
5. A Study of the Open Air Combustion of Paper Containing Aroclor 1242.
6. Determination of PCB Residues in Fish from Lake Michigan.
7. Solubility of Aroclor 1242, 1248, 1254 in water.
8. A Study of Aroclor Chemical Clean-up Procedures for Use with Electron Capture, Gas Chromatography Methods.

APPLIED SCIENCES

Environmental Aroclor Project (Keller-Tucker-Emery-Dietrich)

It appears that disposal of PCB's can be accomplished successfully by thermal oxidation. This conclusion was made after analysis of gas samples from John Zink Company. They were analyzed for PCB, chloride, chlorine, and total organic chlorine. Data were sent to CED and a report is being issued. Additionally, heats of combustio of the PCB's were estimated for CED evaluations.

Water and sediment samples from 6 Pydraul customers were analyzed for PCB's. It is concluded that PCB's are finding their way to sewers, streams and lakes inasmuch as amounts ranging from low ppb to high ppm were determined.

EC/GC determinations for PCB's showed low levels of materials in muriatic acid from Monsanto's central drumming facility. These contaminants appear to be lower chlorinated biphenyls. Confirmation studies are underway.

Paint scrapings from silos and raw milk extracts showed presence of PCB by GC/Mass and EC/GC analysis. The paint scrapings contained 2.87% Aroclor 1254. The raw milk contained an estimated 0.6-0.7 ppm PCB, probably originally Aroclor 1254 but with an altered isome distribution.

Analytical methods and data collected to date were discussed with Westinghouse in an informational meeting on the PCB problem.

A preliminary report on biodegradation of Aroclor was written. Tentatively, Aroclor 1221 degrades in river die-away experiments. Higher Aroclor products may not. More conclusive experiments are underway. A current study involves removal of certain fractions from Aroclor products which might be intractable to biodegradation. Electron-capture chromatograms suggest that most refractory materia can be separated by distillation.

We are continuing to assist the WGK Plant in monitoring bromine content of chlorine they use to make Aroclor products. Seven samples, collected in the past month, were shown by XRF to contain 250-400 ppm bromine.

We are preparing to determine the presence of dioxins in the presen of other chlorinated materials. GC studies show that octachloro-dibenzo-p-dioxin can be determined but that the tetrachloro isomer will have interference from the components in Aroclor 1260.

A better method to determine percentages of various components in undistilled Aroclor is necessary. A suitable GC method appears to have been developed as a consequence of studies made on 60 distilla-tion cuts from 4 Aroclor samples.

SKYDROL 500

Flight tests are currently being performed on Skydrol 500C by various airlines. Only tests involving at least 80% Skydrol 500C will be declared valid. The other component presumably would be Skydrol 500B, heretofore employed. A gravimetric/precipitation method depending upon interaction with the disparate VI improvers is satisfactory for new fluid blends but not for used fluids, because of degradation of the VI improvers. We are attempting to develop an NMR method whose success may hinge upon relatively comparable rates of the decomposition of the VI improver.

A hard deposit from a Northwest Orient Airlines shaft seal was shown by IR to be constituted of a heavy metal, degraded polymethacrylate VI improver and hydrolyzed phosphate ester. We have frequently observed such a material. It is apparently derived in part from Skydrol 500B in the aircraft hydraulic system.

OS-59

We determined by IR that a sample of our OS-59 returned by Maxwell Laboratories had no detectable contaminants. This tends to confirm our advice to Maxwell that OS-59, per se, is not suitable for making capacitors. Maxwell thought their poor experience might be ascribed to contaminated fluid.

MCS 524

By NMR, we showed that a commercial phenyloctadecanoic acid consisted of at least two isomers.

TYPE III, IV CANDIDATES

L-77, said to be a superior wetting agent for Teflon, aluminum and stainless steel was shown by IR and NMR to be a siloxane and that methyl groups and glycol ethers and/or methoxy groups are present.

MCS 571

We received a deposit from an MCS 571 system at Dalton. IR analysis showed that 96% consisted of inorganic salts, primarily bicarbonate as well as silicate. The balance of the sample was a carboxylate salt but not related to any components of MCS 571. Conclusion: the fluid did not contribute to deposit formation.

PYDRAUL 625

Canadian Liquid Air, Ltd. sent us two deposits collected from an exploded air compressor serviced with Pydraul 625. Extractions with hexane, chloroform, acetone and methanol left a highly magnetic residue. The extracts were shown by IR to contain carbonyl and carboxylate salt compounds containing phosphorus. Exact source of these products could not be determined.

SILICATE FLUIDS

The dielectric strength, power factor, resistivity and dielectric constant of Coolanol 25A and MCS 825A were measured to provide data for a product bulletin.

CUMYLPHENYL DIPHENYL PHOSPHATE

Timely analytical support via GC was provided to the pilot plant production of p-cumylphenol.

NASA TENSIMETER

We received a purchase order from NASA for a recording tensimeter. This instrument will be identical to that which we presently use for vapor-pressure and decomposition-temperature measurements on fluids. The components and materials for construction were ordered.

CAPACITOR-LIFE-TEST APPARATUS

Components were ordered for an apparatus for life-testing of capacitors incorporating experimental dielectric fluids.

HEAT TRANSFER FLUIDS (DRM)

Phenyl Ether (diphenyl oxide)

With the able cooperation of the CRD catalyst group, we this month fitted out a pilot unit for the synthesis of phenyl ether by phenol dehydration on a thoria catalyst. The reactor is a 5/8" stainless tube 18 inches long fitted with thermowell, catalyst support screen and steady flow access leads. It is heated to a reaction temperature (ca. 425°C) in a fluid bed thermostat. A metering pump supplies phenol to the reactor at preset flow rates. Reactor pressure is controlled by a pack pressure regulator on the reactor effluent line.

Three catalyst preparations were made using three different refractory supports, namely pumice, bonded silica, and Celite. The thoria was formed in situ by calcining the supports at 600°C after saturating with aqueous thorium nitrate. The presence of thorium oxide was confirmed by X-ray diffraction.

At this writing an initial run at 425°C with pumice-supported thoria has been completed. The first 14 ml of product was liquid at room temperature, verifying that reaction did occur. The next 30 ml of product crystallized, probably indicating low conversion of phenol. Analytical data are being collected.

DEVELOPMENT PROJECTS

Tractive Fluids (1331059-JCW)-

New Tractants

Substantial progress toward the development of new tractive fluids was made this month. A hydrogenated linear dimer of alpha-methylstyrene shows good traction, excellent oxidative stability and fair viscosity properties (Item 1, Table I). A branched octyldecalin shows good coefficient of traction, and fair viscosity and oxidation stability (Item 2).

Table I

Compound	Coeff. of Traction [a]	Viscosity, University 210	100	0	-20	100°F Viscosity	Acid No.
1. Hydrogenated Linear Dimer	.094	3.7	22.7	3,515	31,600	2.5	.17
2. Branched Octyldecalin	.088	3.6	21.9	3,413	36,000	36.5	2.9
3. "Linear" Octyldecalin	.071	2.5	11.4	395	1,447	11.4	2.0
4. Dodecylcyclohexane	.056	1.7	5.41		152		
5. MCS 460	.093	4.1	31.6	26,274		36	2.0
6. MCS 757	.095	3.7	22.3	2,580	18,000	24	1.4
7. MCS 614	.096	4.1	32.0	(3,150)	(31,000)	3	.1

[a] Pin and disc, 19 HP, 3,000 RPM, 400,000 psi, 200°F

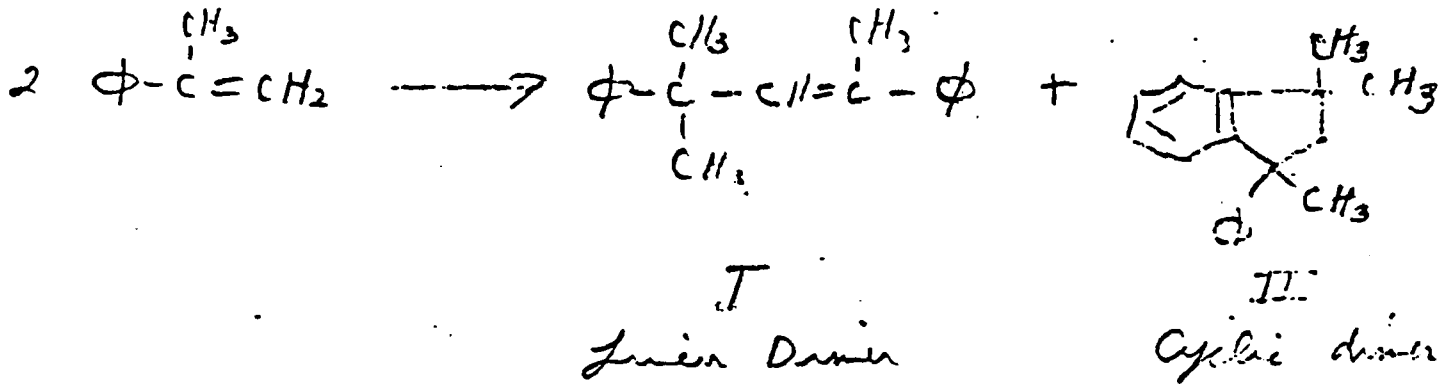
[b] Supercooled liquid; melts at ~68°F.

On the other hand, a "linear" octyldecalin (Item 3) made by alkylation of naphthalene with octene-1, followed by hydrogenation, had better O & C than the branched octyldecalin but considerably poorer traction. A dodecylcyclohexane prepared by hydrogenation of dodecylbenzene (Linear Alkylate 215) for investigation as a blending agent has good low temperature viscosity, but poor traction.

The hydrogenated linear α-methylstyrene dimer compares favorably with hydrogenated dicumyl (MCS 614). It is almost as good in coefficient of traction, and (surprisingly) is as good in the O & C. Its 210°F viscosity is lower, but it is better in low temperature properties, since MCS 614 melts at about 68°F. The new material has not crystallized so far, at 0°F or at -40°F.

Synthesis

Linear α-methylstyrene dimer. As reported last month, the linear α-MS dimer is obtained by allowing α-MS to stand at room temperature for a long time with 0.2% iodine. After 8 days, work-up gives the linear dimer I in 70% yield, with 48% conversion. This procedure gives a product with 2% or less of the cyclic indane dimer II.



Same polymer is formed.

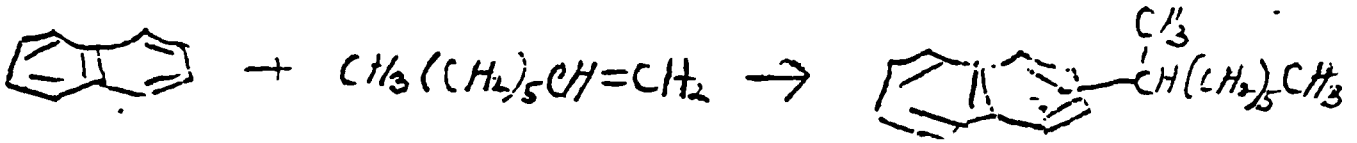
Hydrogenation of the linear dimer proceeded smoothly over Raney nickel to the fully saturated product.

Running the iodine-catalyzed dimerization at 50°C instead of room temperature did not increase the reaction rate, over a 3 day period.

A possibly better catalyst for the dimerization is a $POCl_3$. H_3PO_4 mixture. This catalyst, described in an expired Dow patent, gave 68% conversion and yield of dimer in a few hours, at about 100°C. However, the content of cyclic dimer increased to about 10%. The effect of this small amount of cyclic dimer on the properties of the fluid will be determined. CRD investigated the hydrogenated cyclic dimer as a tractant. It is more viscous at low temperatures (34,000 cs at 0°F) and oxidatively less stable than the linear material.

Also to be determined is whether the crude α-MS from Chocolate Bayou can be used as raw material. If so, it will lead to a very low raw material cost. Possible difficulty here may be the effect on hydrogenation of the small amounts of sulfur compounds in the crude α-MS.

Octyldecalins. Alkylation of naphthalene with octene-1 using 7.8% $AlCl_3$ and 7.8% nitromethane in heptane solvent led to the isolation of an octylnaphthalene mixture in 52/ yield.



Isomers, formed by migration of the double bond along the chain, are also formed. Hydrogenation over Raney nickel gave the "linear" octyldecalin, item 3, Table I. This had good oxidative stability and low temperature viscosity, but rather poor traction.

Alkylation of naphthalene with the highly branched octene diisobutylene gave a t-octylnaphthane fraction, and also t-butylnaphthalene, and higher t-alkyl products. This type of reorganization is a well-recognized hazard in alkylation of aromatics with poly(iso-olefins). Using naphthalene and diisobutylene at 2:1 mole ratio, with 3.9% each $AlCl_3$ and nitromethane (on naphthalene) gave 41% yield of t-octyl naphthalene and 33% t-butylnaphthalene. Hydrogenation of the octylnaphthalene over nickel gave the branched octyl decalin (item 2, Table I). This is a good tractant, with fair O & C and viscosity properties. In another experiment, using about four times as much nitromethane, and working up the reaction 1/2 hour after olefin addition was complete, 29% t-butyl, and 55% t-octyl was obtained. However, it is suspected that the "t-octyl" fraction contains material of longer chain length; characterization of this product is not yet complete.

Spectroscopic evidence indicates that an alkyldecalin is a major constituent of the Sun toric fluid. Judging from the two examples so far prepared, this type compound appears of less interest than the linear α-MS dimer. Both have low raw material costs. The α-MS dimerization is simpler, however, and the properties of the fluid are better.

MCS 998, a -40°F fluid for Lycoming, is a 70:30 blend of MCS 757 with diisodecyl adipate, with 1% dioctyldiphenylamine as additional oxidation inhibitor. Although MCS 757 does not foam, the blend did, but foaming was controlled with 20 ppm of DC 200 silicone. Properties of this fluid are given in another section of the summary. The first two gallons of MCS 998 sent to Lycoming were lost in shipment; a second two gallons were blended and shipped.

Used MCS 9612 - A sample of used MCS 9612 from Gates Rubber Co. in Denver was received. It had run for 25 hours at 200-275°F, and for 1 hour at 330°F bulk oil temperature. The oil contained a fine black sludge, which was found to be metallic wear debris. Viscosity and composition (by VPC) of the oil were essentially unchanged; acid number was 0.49.

Naphthenic Oils - An attempt to relate differences in traction coefficient among GMR 1204, Solnus 55, and Paratex 75, all naphthenic oils, to differences in degree of unsaturation showed no observable correlation. NMR and IR spectra were similar - there is no aromatic unsaturation. No unsaturation toward bromine was detected in the Paratex 75; the other two gave turbidities with bromine, probably due to reaction with additives.

MCS 998 (RLG)

The -40°F tractive fluid, MCS 998, was tested for viscosity, foam, and 4-Ball wear before shipment to Lycoming. The results are:

Viscosity at -40°F = 22,250 cs
100°F = 17.40 cs
210°F = 3.49 cs

Foam

Blender Foam at 200°F/200cc/2 min
Initial height of foam = 1/4 in.
Time to break foam = 20 sec.

ASTM D892-58T
Tendency 15 ml
Stability 8 sec

Four Ball Wear 200°F/1200 rpm/2 hrs/52100 on 52100
Scar diameter = 0.87 mm.

The pour point data on the "running" test conditions was measured and found to be between -60°F and -55°F.

Based on these results and previous O & C and traction testing, MCS 998 is judged to be:

1) equivalent to F50 in traction under the slip velocity conditions seen on the Lycoming LD 6

2) slightly worse than MCS 757 in foam but better than Versilube F50

3) a formulation that will cold start in the LD 6 at -40°F since it is equivalent in viscosity to MCS 757 at -20°F which passed our cold start pump test (memo 2/16/70).

Three gallons of MCS 998 were blended and two gallons were shipped to Lycoming with two gallons of MCS 463 for flushing purposes.

11332

Tractive Fluids

The coefficient of traction on many fluids was measured by a new procedure on the twin disk test at two loads and two temperatures. The semi raw data is being reduced to traction coefficient vs. slip velocity. Qualitatively four synthesized fluids are equivalent to MCS 460 in traction. They are

DCCDC
Hydrogenated linear dimer HLD
Octyl Decalin (OD-3)
H27D4 Hydrogenated still bottoms

Viscosities, 4-Ball wear, and O & C were run (see JCW MS). Blends are being made to meet the 0°F GM Toric Fluid specification of 2000 cs.

Automotive Fluids (1338459-QET)

Air conditioner lubricant candidates MCS 994 and 995 submitted last month to Ford performed exceptionally well in their system except under simulated idle (low rpm) conditions. The difficulty resides in the relatively low viscosity of our products. We have reformulated a high viscosity lubricant now designated MCS 1005. This material has the following composition and properties.

MCS 1005

Composition: ~33% Carbowax 200
~67% phenyl bis(3-hydroxyphenyl) phosphate
0.2% tolyl triazole

Viscosity:

at 210°F 29.75 cs
at 100°F 775 cs
*at 165°F ~80 cs (extrapolated)
* 80 cs at 165°F desired by Ford)

Pour Pt. about 5-10°F

Freon insolubility: better than MCS 994 + MCS 995

Santoflush - Sewage Transfer Fluid (QET)

A request by Chrysler for a safe, low density, water immiscible, high flash point, fluid to be used on shipboard sewage systems was investigated. The best candidates appeared to be in the order DIDA > DOA > Santicizer 148 + mineral oil. Accordingly we sent Chrysler a sample of DIDA designated MCS 997. Their response was immediately favorable and they have now placed a $300 order for larger quantities of MCS 997.

PIONEERING (EOS)

Rub-block on Rotating Ring Friction and Wear Tester:

Following the receipt of Capital Authorization for this project, it has been placed in the hands of the Instrument Development Group who are finalizing the design. In this they are being aided by Prof. J. Georgian of Washington U., particularly in regard to calculations of the dynamics of the tester. Completion of the device is expected by the end of June. This machine is to be used in the contract for Thermo Electron to measure the lubrication of thiophene/F-50 blends for the Rankine Cycle automobile engine.

High-speed, High-temperature Bearing Tester:

In anticipation of its use in two different projects under consideration by NASA, steps are being taken to modify the ERDCO Universal Gear and Bearing Rig. The first step will have to be the gear-box change to produce speeds up to 30,000 rpm. ERDCO now estimates delivery in less than 24 weeks. We shall try to place the order for this item as soon as possible. ERDCO has looked into the feasibility of operating the standard 100 mm roller bearing test head at speeds up to 30,000 rpm and has concluded that substitutes for both the Rollway test bearing and the loading/shaft-support ball bearing in the test head would have to be found to permit operation over the rated maximum speed of 10,000 rpm. They are currently preparing an estimate of the cost of doing this. The high-speed ball bearing test head is also being considered. MAIC has been contacted regarding the practicability of their designing a test head which is compatible with the ERDCO test drive. This will be feasible. Furthermore, they will probably be able to design a test head which will accept either 80 or 100 mm ball bearings, giving us a chance to attain $dN=3 \times 10^6$ without having to replace the entire test head. Who is to build the new test head is not yet decided. A visit to MAIC to finalize the specifications for the test head is planned for May 11, when Alastair Cameron will be available. NASA-Lewis will then be visited on the 12th to see what they think of our plans.

NASA-Lewis Contract No. NAS3-12418: A request for a no-cost time extension fo this project is being planned and will be discussed on May 12 during the visit to NASA-Lewis. Prof. Cameron has contracted with his students who help man this project through June of '71.

Type III Jet Lubes - Lubricity (FSC)

New Additives - The following compounds were screened in our slow speed four ball test in MCS 524 (.1%). Those underlined show improved lubricity.

Ultraphos 12

P & G Additive #3

phenylboric acid

triallyl phosphate

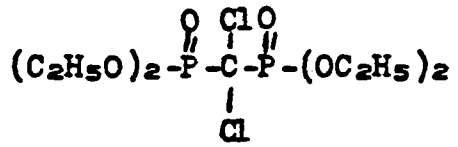
Ultraphos 12

This liquid is an anionic phosphate ester surfactant manufactured by Witco Chemical Co. It gives an excellent friction curve on M50 steel. It has not been run on any other metallurgy.

This phosphate gives less deposits in an O & C test than MCS 293 but has a higher viscosity increase (8.3% vs. ~4%).

P & G Additive #3

This chemical is tetraethyl perchloromethylene phosphonate



At 0.5%, this phosphonate does not change the boundary friction of MCS 524 on M50 steel or on hard anodized aluminum. But it has an excellent curve on aluminum metal. This is the first additive in MCS 524 which gives a curve on aluminum comparable in magnitude and general shape to Mobil Jet II. A comparison curve is shown with following graph.

Phenylboric acid

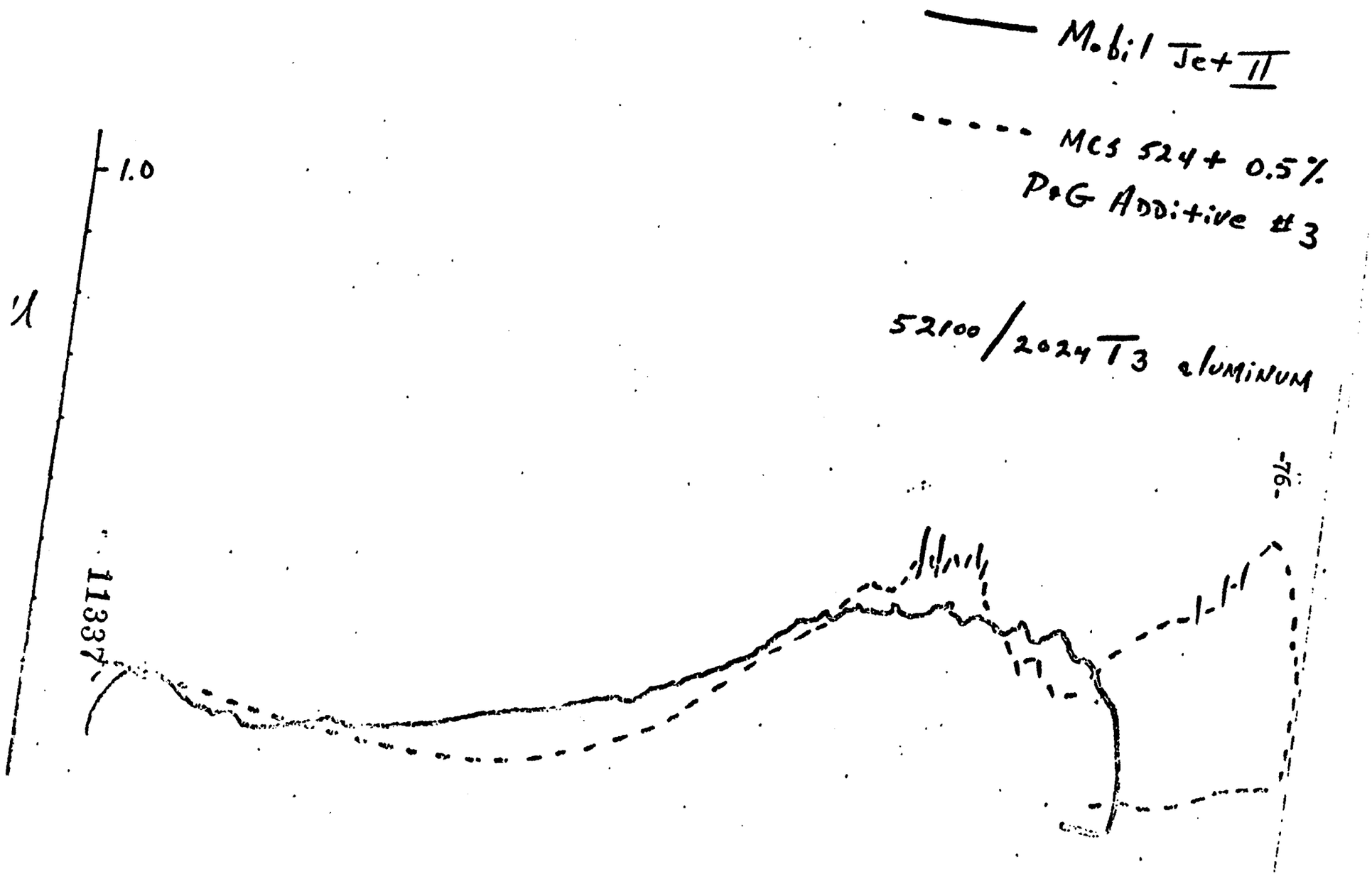
This acid did not improve the boundary friction on M50, hard anodized aluminum, or aluminum metal. It is interesting that many of our acid additives change the friction of steel or anodized aluminum but don't change the friction of aluminum metal. This metal will react instantaneously with acids as weak as water. And lubrication theory predicts the generation of fresh metal under boundary conditions. Perhaps the additives react but don't produce lubricating soaps. Perhaps the metal is so reactive it reacts with the base stock giving metal corrosion.

Triallyl Phosphate

This additive did not change the boundary friction on M50, anodized aluminum, or aluminum metal.

New Base Stocks

Most of the additives which we forwarded to SNECMA in MCS 524 were screened in MCS 963 base stock in the slow speed four ball. MCS 963 is



meta ØSØSØ

+

ortho ØSØSØ

+

meta ØOØSØ + antifoam

These tests were run to see what the additive response would be in a base stock chemically similar to 524 but with a different viscosity. Also, to obtain patent coverage.

The additive friction curves in MCS 963 were very similar to those in MCS 524. Thus:

phenylphosphinic acid: large μ reduction on hard anodized aluminum; large μ reduction on M50 steel from 200-400°F followed by a μ spike; some μ reduction on aluminum metal.

perfluoroglutaric acid: worse high temp. μ on M50 steel; large μ reduction on hard anodized aluminum.

Also screened were MCS 963 + monopotassium oxalate

MCS 963 + bis(chloropropyl)-2-propene-phosphonate

and MCS 963 + A-88 + trichloroacetic acid

Only the oxalate had a different pattern in the lower viscosity base stock. It had a significant friction lowering on M50 steel in MCS 524 but only a small friction lowering in MCS 963.

Other additives

CP 64600 - This is Ludwig Maier's phosphosulfide. A 500°F O & C test was run on samples of MCS 524 plus 0.1% and 0.05% of this sulfide. Generally, these blends gave reasonably clean tests with fewer deposits than MCS 293. Specifically:

In the presence of copper, the formulated fluid had less black deposits at both concentrations than MCS 293. Some vapor coking was observed.

In metal attack the copper corrosion was comparable to MCS 293. We increased the silver corrosion.

The viscosity increase was 9% vs. 4% for MCS 293.

In the absence of copper the deposits were as above. The metal attack had increased silver corrosion. The visc. increase was comparable to MCS 293. All in all not a bad test, considering the reactivity of the additive.

SKF-NASA Report on MCS 2931

The final report on the bearing tests with MCS 2931 has been written. The authors are L. A. Peacock and W. L. Rhoads of SKF. It has some discrepancies with the information given us by Bob Johnson. The report states that MCS 2931 ran two successful tests out of six with two runs giving fluid related spalling failures. Our call report says 2931 passed five out of six tests with one spalling failure which was not fluid related.

It is interesting that the lubricity failures in the report are surface initiated spalling failures, i.e. fatigue failures. This mode of failure can be initiated by water. The trichloroacetic acid additive used in MCS 2931 did contain ~1% water. We should watch water content for possible correlation with fatigue in any future use of this product.

The authors use a dimensionless parameter h/σ to characterize fluid performance. This is the ratio of the film thickness, h, to the composite surface roughness. A high value implies a good film and puts the fluid in the higher EHD region (i.e. near hydrodynamic lubrication). A value below 2 implies lots of contact, i.e. a more boundary like EHD regime. The correlation of this parameter with bearing performance is not too good.

SUMMARY OF TEST RESULTS

[illegible]	FLUID (S)	MEASURED h/σ AT 300°F	TYPICAL BEARING CONDITION
EXCELLENT	1. [illegible]	3.6 - 4.0	GOOD.
GOOD	2. [illegible] POLYETHER.	3-4 (INITIALLY) <1.0 (LONG-TERM)	MINOR GLAZING AND MICROPI[illegible].
	3. [illegible] FLUOROSILICONE.	<1.0	
ACCEPTABLE	4. [illegible]	<1.0	[illegible]
	5. [illegible] POLYPHENYL ETHER.	<1.0	
UNACCEPTABLE	6. [illegible]	2.[illegible] (INITIALLY) <1.0 (LONG-TERM)	
	7. [illegible]	3.5-4.0	[illegible]
	8. [illegible] ESTER.	<1.0	

11339

Of the fluids rated equal or better than MCS 2931, only the Krytox 143 AB and possibly the Dow fluorosilicone could be used in air operations.

Phenylphosphinic acid - This acid has one striking friction property. That is, its μ- temp profile up to about 450°F is very similar regardless of the metallurgy. This is shown graphically on the following figure. Perhaps this tells us something about the mechanism of lubrication by this acid.

Phenyloctadecanoic acid - This material (Eastman) is a general boundary additive for MCS 524. Last month I stated that it contained no terminal phenyl group. The NMR people now say this is an error. The sample is a mixture of

$$\text{Ø-}(CH_2)_{17}\text{-}CO_2H \text{ and}$$

$$CH_3(CH_2)N\begin{matrix} H \\ C \\ | \\ CO_2H \end{matrix}-(CH_2)_{15}-\overset{CO_2H}{N}$$

It would be interesting to see if the pure terminal acid is more effective than the mixture.

Aryl Phosphate Lubricity

Several additives were evaluated as steel lubricity agents for aryl phosphates. Specifically, they were tested in MCS 941 (36% CPDPP + 37% TPP + 27% CDP). Our interest in this base stock has dropped since we are now making blends based on NCPDPP. However, MCS 941 is a good reference phosphate with poor steel lubricity. The screening test was the Chrysler Falex Test. This uses a hard steel (Rc 20) on a soft steel (Rb 85). The results were as follows:

Additive (.1%)	Falex Test Failure Load psi
none	1000
2-propyl benzenephosphonite	4250 (passes test) 3270
H-1128	4250 (passes test) ~2700
L-77	~2100
CP64600	900

The results with the first two additives are not bad at all.

phenyl phosphonic acid in HCS 524

0.1 % —— Mild Steel

0.1 % —— Admiralty

0.1 % —— Al

0.075 % —— stainless steel

11341

2-propyl benzenephosphonite $\phi\text{-}\overset{\overset{O}{\|}}{\underset{H}{P}}\text{-}OC_3H_7$

This is not an acid but is probably easily hydrolyzed. It should be screened in the Republic Test.

H-1128 - This compound is

$$(\underline{i}\text{-}C_3H_7O)_2\text{-}\overset{\overset{S}{\|}}{P}\text{-}S\text{-}CH_2\text{-}\overset{\overset{CH_3}{|}}{CH}\text{-}S\text{-}\overset{\overset{S}{\|}}{P}(O\text{-}\underline{i}\text{-}C_4H_9)_2$$

It was obtained from the Oil Additives Group.

L-77 (Carbide)

This is a silicone-ethylene oxide block polymer. It is advertised as a "super" wetting agent for stainless steel, teflon, aluminum, and glass. Since this polymer probably isn't reacting at the surface, the increased load carrying is probably due to increased wetting. Blends with a higher concentration will be run.

CP64600

This is Ludwig Maier's phosphosulfide.

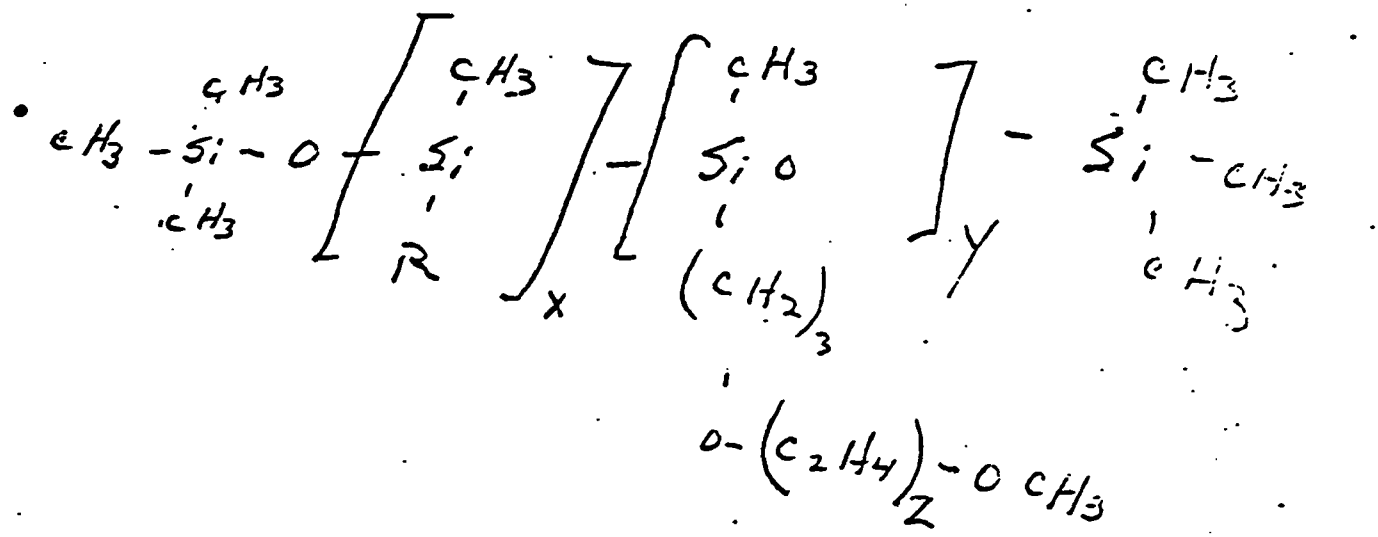
Pump-Flow Properties of Aryl phosphates

We are also starting to look at some flow properties of arylphosphates such as cavitation probe, and streaming potentials. A one gallon blend of NCPDPP plus 0.04% FC-98 was made up for the streaming potential measurement.

Water-Oil Emulsion Lubricity-
Water-Glycol Lubricity

One suggested approach to the lubrication of water-oil emulsions is to minimize the interrupted flow of two phases at the surface. My first thought to a chemical approach to minimum phase separation was to try Carbide's surfactant silicones. These are block polymers of silicone-ethylene oxides. Thus one tail of the molecule would be soluble in each phase and might tie them together. Also, by varying the ethylene oxide chain, the viscosity of the water layer might be matched with the oil layer.

I have since found a Carbide patent for water lubricants (U.S. Patent 3,457,173 issued 6-22-69). Water lubricants include emulsions and water-glycol blends. The feature of the invention is the use of silicone-ethylene oxide polymers in aqueous fluids. A typical structure for such a polymer is:



The lubricity of the block polymers is enhanced by emulsified polyolefins such as polymerized α-olefins. Examples are given of improved Falex loads for emulsions.

The use of these block polymers may explain the excellent pump wear properties of Carbide's Ucon Hydrolube 150 LT. We should evaluate these polymers in water blends of interest to us. Presumably Carbide has done considerable testing of these products.

NASA-Lewis Research

Thermal Decomposition (T_D) Runs

Bill Jones asked us to rerun two T_D's in the presence of M50 steel. This was because our T_D values were higher than they expected based on their experience with these fluids. He thought the metal might catalyze a lower T_D. This did in fact happen with one of the samples.

All of the vapor pressure and T_D curves are completed. The results have been phoned to W. Jones. He also requested copies of the curves for calculation of Arrhenius constants. Lew Fowler is preparing a graphic conversion scale so these can be read directly from the curves. This should be ready this week.

MCS 460 Samples

One quart samples of MCS 460 were sent to Bob Johnson at NASA-Lewis and Ward Weiner at Georgia Tech. I also sent them a preliminary data sheet for this compound.

Film Thickness Contract 11343

Work continued on the heat capacities and bulk modulus curves of the model compounds. Cameron recently has used

his film thickness measurements to study freezing in the contact zone. He has expressed an interest in thermodynamic measurements of glass transitions or pour points. This might be heat capacity (via DTA or DSC) or it might be density or modulus measured as a function of temp. This is still up in the air but we may make some further physical property tests on the fluids.