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WATER ABSORPTION OF FLUIDS/OILS.(U)
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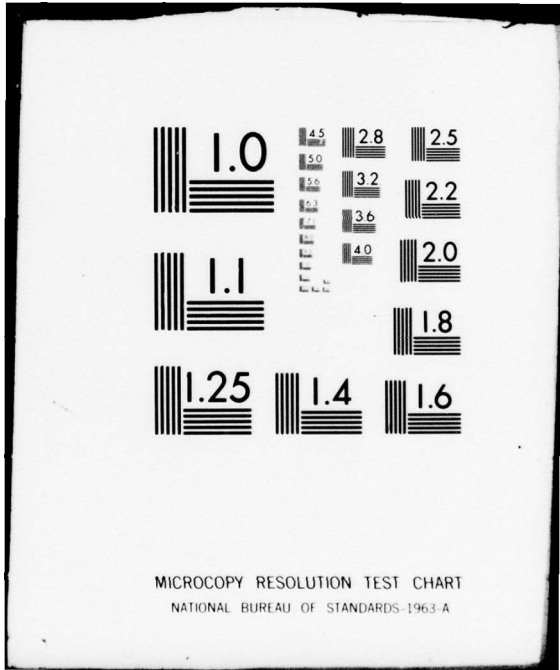
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WATER ABSORPTION OF FLUIDS/OILS

by
Robert G. Jamison

June 1978

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20. ABSTRACT (Continue on reverse side if necessary and identify by block number) The object of the investigation was to determine the effects of water absorption on the inhibitor systems and basestocks of various hydraulic fluids and engine oils. From this experimentation, a need was generated to develop a more accurate method for analyzing water in these fluids and oils. The results subsequently obtained verified the fact that absorbed water affects the stability of the inhibited fluid and oil systems.		

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WATER ABSORPTION OF FLUIDS/OILS

I. INTRODUCTION

All petroleum-based fluids can absorb trace amounts of moisture if exposed in a humid environment or by direct contact with water. Depending on the specific applications and the severity of water contamination, this absorption can result in deleterious effects to the intended function of the petroleum-based fluids. This contamination becomes more critical to fluids formulated with additives designed to impart or improve desirable properties or suppress undesirable properties in order to accomplish specific functions. These additives, dissolved in a predetermined formulation to meet specific performance requirements, are required to remain in solution under all environmental and storage conditions including those involving water contamination.

Unfortunately, this critical solubility equilibrium can be easily upset by small amounts of contaminants leading to a massive functional deterioration of the fluid. A case in point occurred in the turret and gun control hydraulic system of the M60A1 (AOS) tanks retrofitted with the new less-flammable hydraulic fluid MIL-H-46170 (FRH), which is based on a synthetic hydrocarbon (commonly termed alpha-olefin). It was determined that moisture reacted with a water stabilizer additive, causing the rust inhibitor (dinonylnaphthalene sulfonate) to precipitate from the solution.¹ Other investigators found that the presence of moisture in nonaqueous liquids such as turbine oils and hydraulic oils may constitute a serious problem.² These oils are designed for long-life operation and reuse in multifunctional applications where water is a normal or frequent contaminant.

The Project Manager's Office of FAMECE/UET (Family of Military Engineering Construction Equipment and Universal Engineer Tractor) had expressed a concern with the potential problem of water contamination/absorption and resultant emulsification tendencies of the formulated specification engine oils utilized in the hydraulic systems of these vehicles. It was noted that FRH fluids if adopted for FAMECE/UET would be able to tolerate and solubilize larger amounts of water than the MIL-L-2104C OE/HD010 oils, resulting in a system less sensitive to problems associated with water absorption.

¹ M. E. LePera, J. Messina, H. Mullinger, and C. E. Snyder, "Investigation of the Causes of Stuck Servovalves in U.S. Army Hydraulic Systems Using MIL-H-46170, Hydraulic Fluid, Rust-Inhibited, Fire-Resistant Synthetic Hydrocarbon Base." American Society of Lubrication Engineers, 32nd Annual Meeting in Montreal, Quebec, Canada. 9-12 May 1977.

² E. L. Armstrong, S. J. Leonardi, W. R. Murphy, and P. S. Wooding, "Evaluation of Water-Accelerated Bearing Fatigue in Oil-Lubricated Ballbearings." Vol. 34, 1, 15-21. American Society of Lubricating Engineers, 10-13 May 1976.

In another area of water contamination problems occurring in the field, Yuma Proving Ground (YPG) personnel requested an investigation of a subzero automotive engine oil (APG Purchase Description No. 1) which had been delivered to their facility in an unsealed 5-gallon container. The oil showed a precipitate and appeared to be contaminated by water. This evaluation required (1) the investigation of a new and uncontaminated oil sample for the water absorption effects on the product in question and (2) an assessment as to whether the absorbed water would produce any additive component precipitation as had occurred with YPG samples.

One additional area of potential concern involved the water absorption tendencies of the MIL-L-23699B aircraft gas-turbine engine lubricants. Water absorbed by these fluids was reported to be related to engine-bearing corrosion problems. More specifically, corrosion was evidenced in the areas where the ball-bearing made contact with the race during periods of short inactivity. As a result, an evaluation of water absorption tendencies was deemed necessary in an effort to determine the relationship of water absorption to bearing corrosion tendencies of aircraft gas-turbine engine lubricants.

In view of the above areas of immediate and/or potential concern, an investigation was judged necessary to define the water absorption and compatibility phenomena of lubricants and fluids found in the Military supply system. Further, with the increased emphasis on utilization of synthetically derived base stocks for fluids and engine oils, this need became more critical. This report described the results of the investigation to define water absorption characteristics of Military specification fluids and engine oils.

II. TEST PROCEDURES

Individual fluid/oil samples evaluated during this investigation initially were subjected to the humidification test and, subsequently, were analyzed for water content by use of a newly developed gas chromatographic procedure, or the Beckman K-4 Aquameter (ASTM D-1744). Additionally, samples were analyzed by infrared spectroscopy after testing to ascertain the amount of additive separation. In the humidification test described in Appendix A, a desiccator is charged with Ammonium Sulfate to yield a relative humidity (RH) of 80 percent.³ A 100-milliliter (ml) sample of fluid/oil is then exposed to the conditioned atmosphere by placing the sample into an uncovered test jar in the center of the desiccator. Samples of fluids/oils can be withdrawn at specified intervals through a stopper in the desiccator lid, allowing the RH to be constant during the test duration.

³ Federal Specification VV-B-680B, "Brake Fluid Automotive," 20 Jul 72.

The Gas Chromatography (GC) was employed to determine the water content in the fluid/oils wherein the Beckman K-4 Aquameter (ASTM-1744) results were suspect because of additive chemistry interference.^{4,5,6,7} The instrument used was a Hewlett-Packard 5834A with a 18850A terminal (Appendix B). The column used was a 2-m (78.7-in.) by 3-mm (OD, 1/8-in.) diameter copper tubing packed with Porapak Q (Walter Associates, Inc.). The following are conditions under which the tests were performed:

Detector -- Thermal Conductivity	Test Condition
Detector Cell Temperature (° C)	295
Helium Flow Exit (cc/min)	30
Initial Temperature (° C)	120
Initial Isothermal Time Interval (min)	1
Programmed Rate for Temperature Rise (min)	5
Final Temperature (° C)	200
Final Isothermal Temperature Time Interval (min)	2
Injection Port Temperature (° C)	300
Max. Oven Temperature (° C)	250
Recorder Chart Speed (in./min)	1
Recorder Chart Attenuation	1
Recorder Chart Slope Sensitivity	0.03

With an infrared spectrophotometer⁸ equipped with a 0.25-mm Sodium Chloride Cell, fingerprint spectra were recorded of the fluid/oil before and after the humidification tests. In this manner, changes in infrared spectra could be monitored to indicate potential additive-water-fluid incompatibility.

III. RESULTS

Eight samples (described in Table 1) of synthetic subzero automotive engine oil, with which YPG encountered problems, were subjected to the humidification test. The results in terms of water absorbed after 14 weeks of exposure are presented in

⁴ ASTM-D-1744, "Water in Liquid Petroleum Products by Karl Fisher Reagent."

⁵ Serm Zweig, Handbook of Chromatography. Vol. 1 and 2. CRC Press, 1972.

⁶ D. Nogare & Juvet, Gas Chromatography, Interscience, April 1966.

⁷ Robert G. Jamison, "Determination of Contaminants in Less-Flammable Hydraulic Fluids." MERADCOM Report 2192, September 1976.

⁸ K. E. Stine, "Modern Practices in Infrared Spectroscopy." Laboratory Manual, 1975.

Table 1. Water Absorption Levels in Test Arctic Engine Oil Samples^a

Specification Type	Source	Sample	Basestock Type	Exposure to Humidification Test For:				
				4 wk	6 wk	8 wk	10 wk	14 wk
APG PD 1 ^b	Company C	CCL874	Polyalkylated Benzene	0.28	0.32	0.28	0.32	0.35
APG PD 1	Company E	CCL875	Diester	0.17	0.28	0.23	0.22	0.24
APG PD 1	Company E	CCL876	Diester	0.34	0.29	0.24	0.25	0.27
APG PD 1	Company E	CCL877	Diester	0.34	0.31	0.25	0.24	0.31
MIL-L-46152	Company H	CCL878	Diester	0.51	0.45	0.39	0.43	0.48
Candidate MIL-L-46167	Company M	CCL725	Polyalpha Olefin	0.32	0.33	0.37	0.42	0.25
Candidate MIL-L-46167	Company R	CCL807	Diester-Mineral Hybrid	0.44	0.46	0.46	0.50	0.31
MIL-L-46152	Company C	CCL813	Polyalkylated Benzene-Mineral Hybrid	0.30	0.32	0.32	0.33	0.16

^a Water levels determined via ASTM D-1744 using a Beckman KF-4 Aquameter.

^b Aberdeen Proving Ground Purchase Description No. 1 for "Lubricating Oil, Internal Combustion Engine, Subzero."

Table 1 and are illustrated in Figure 1. After 14 weeks, the subzero automotive engine oil (APG Purchase Description No. 1) became cloudy and a fine, white precipitate was observed at the bottom of the test jar. This sample exhibited the same conditions as the deteriorated contents of the 5-gallon container received from YPG, proving the assumption that excessive water contamination had caused the additive precipitation.

Previous to this study there was no acceptable test method to determine or measure the amount of water absorbed by a variety of formulated oils and fluids that differed not only in composition but also in chemical structure. Therefore, it was necessary to develop such a method. Six samples of engine and hydraulic oil were exposed to the humidification procedures and sampled for the water content every 30 days, using the Karl Fisher technique for measuring the weight percent water. The six fluids selected are shown in Table 2. The measured amount of absorbed water in the synthetic hydrocarbon base (MIL-H-46170) material was extremely high. The Karl Fisher Method (ASTM D-1744) is accurate only for small amounts of water (ASTM limits 50 to 1000 p/m). Figure 2 illustrates the results of determining water content by GC as interfering compounds are elated to distort the quantitative analysis. Table 3 shows the comparison of absorbed water measured by Karl Fisher (ASTM D-1744) and GC versus the difference in weight percent between the methods. The Karl Fisher reagent apparently reacts with the dispersant additives in engine oils to give higher results, whereas the GC is not affected by this and only determines water as a singular peak. The resulting water content of the samples in Table 3 did not affect a precipitate; therefore, the difference between the absorbed water (after 30 days of exposure) and one percent total water was investigated. After the addition of free water, the samples were shaken vigorously for 30 seconds and then measured. After 2 hours, it was evident that all samples except silicone produced a precipitate, indicating gross water incompatibility.

To determine whether additives precipitated or reacted with absorbed water after exposure to the humidification procedure for 114 days, the infrared spectrophotometer was used to identify possible additive depletion. Figure 3 illustrates the spectra of three different hydraulic fluids which were exposed to the humidification procedure; in all three there is a slight "OH" band at 3 microns, typical of the hydraulic fluids tested.

The water absorption tendencies of MIL-L-23699B gas-turbine lubricants are of interest in view of recent efforts to develop a corrosion-inhibited version. Six QPL samples of MIL-L-23699B were obtained from the Naval Air Propulsion Center (NAPC). In addition to these six products, one petroleum solvent-extracted-neutral basestock (450SUS) was evaluated concurrently for reference purposes. In order to permit direct comparison with other fluids and oils, five additional specification products are shown in Table 4. The results are shown for a test period of 2, 4, 8, 16, and 30 days after exposure to the humidification test.

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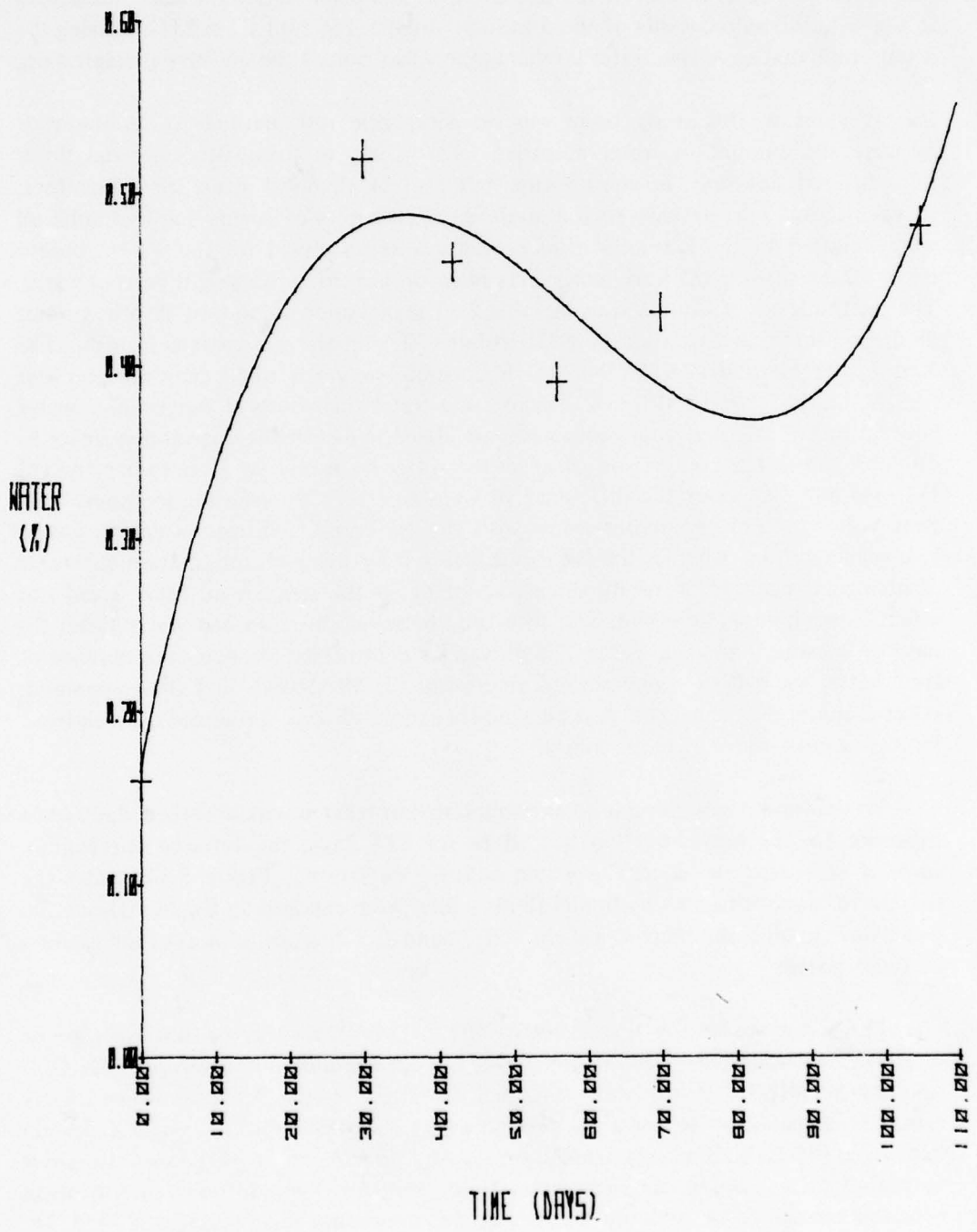
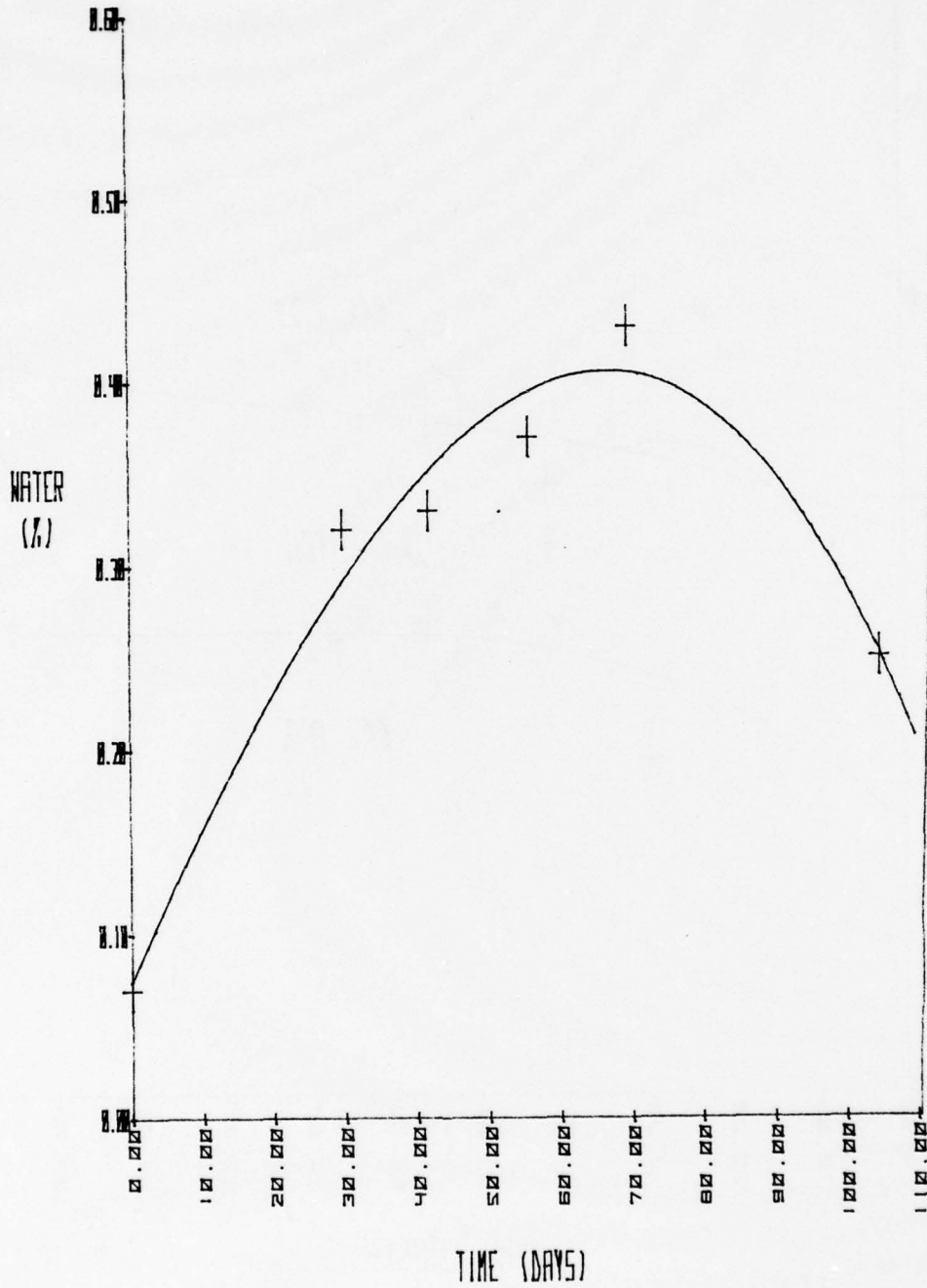
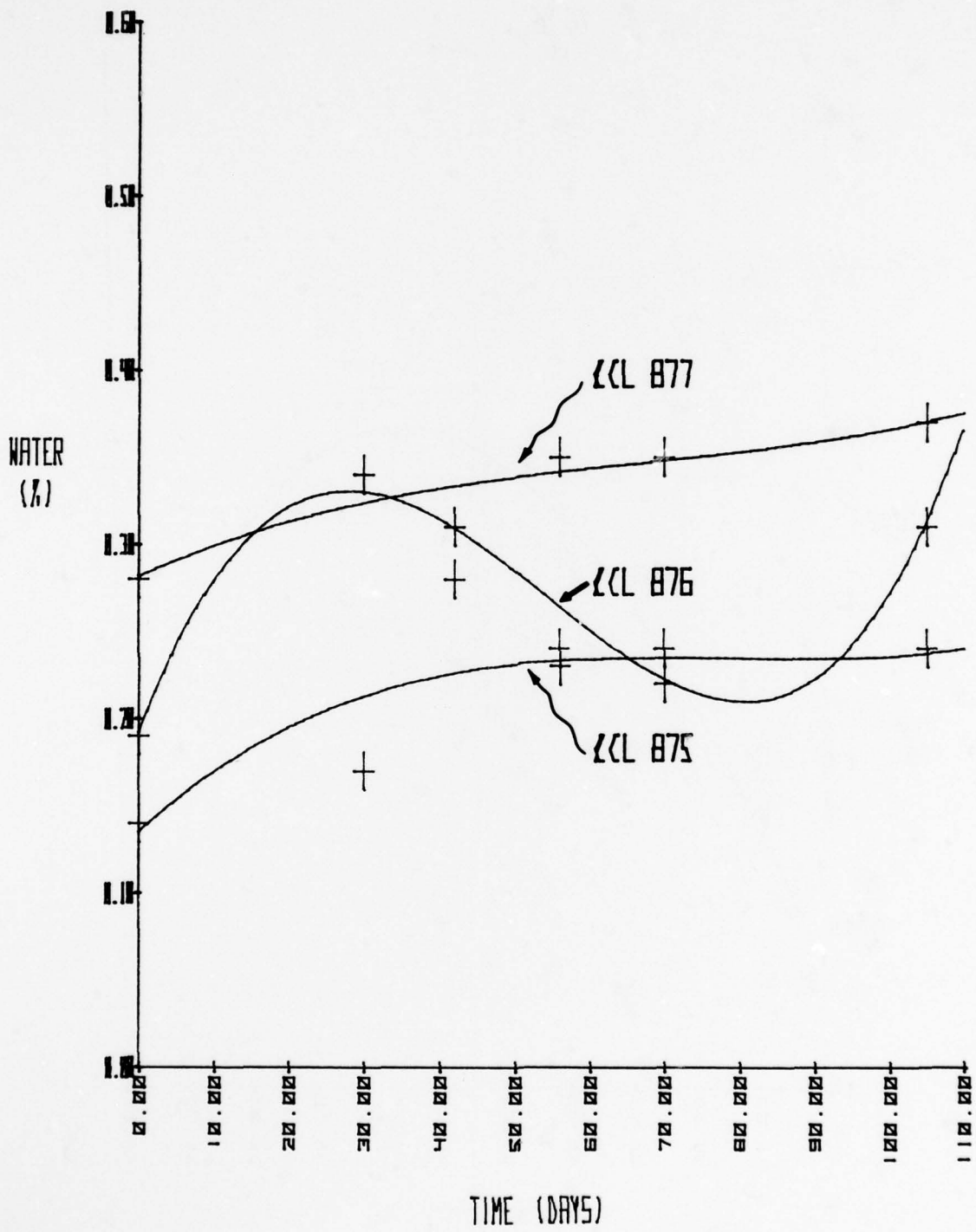
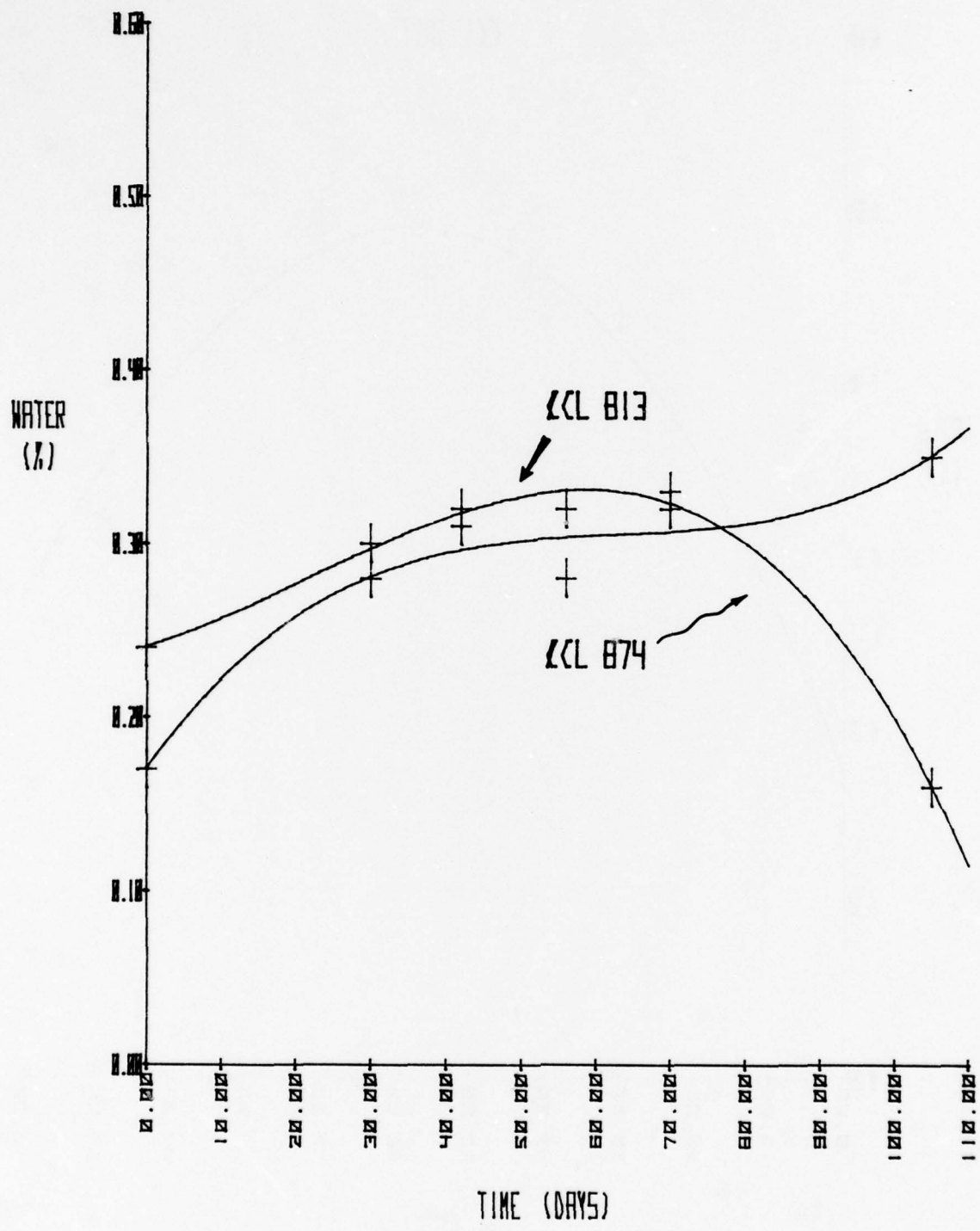


Figure 1. Water absorbed by the eight test samples after 14 weeks of exposure.

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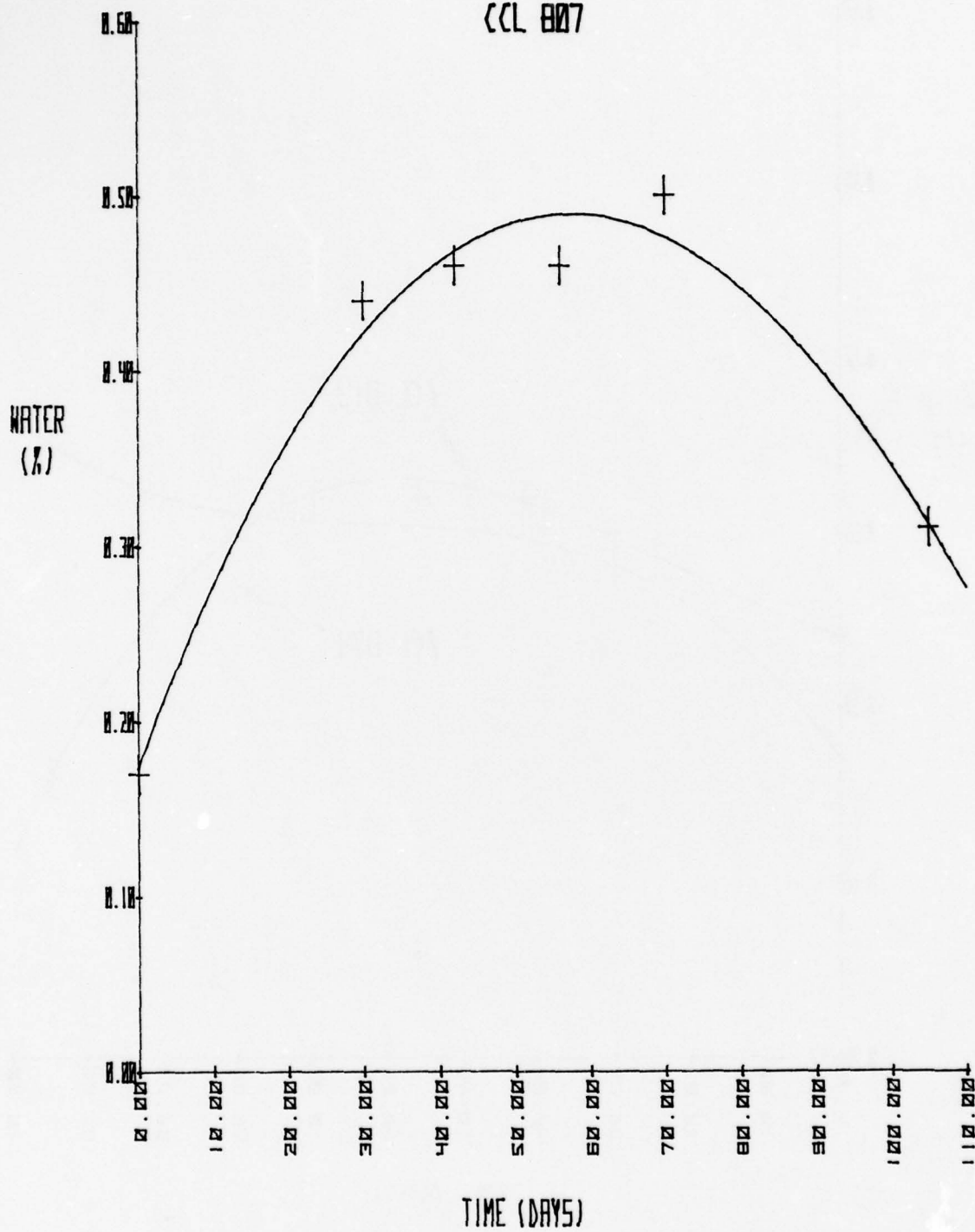
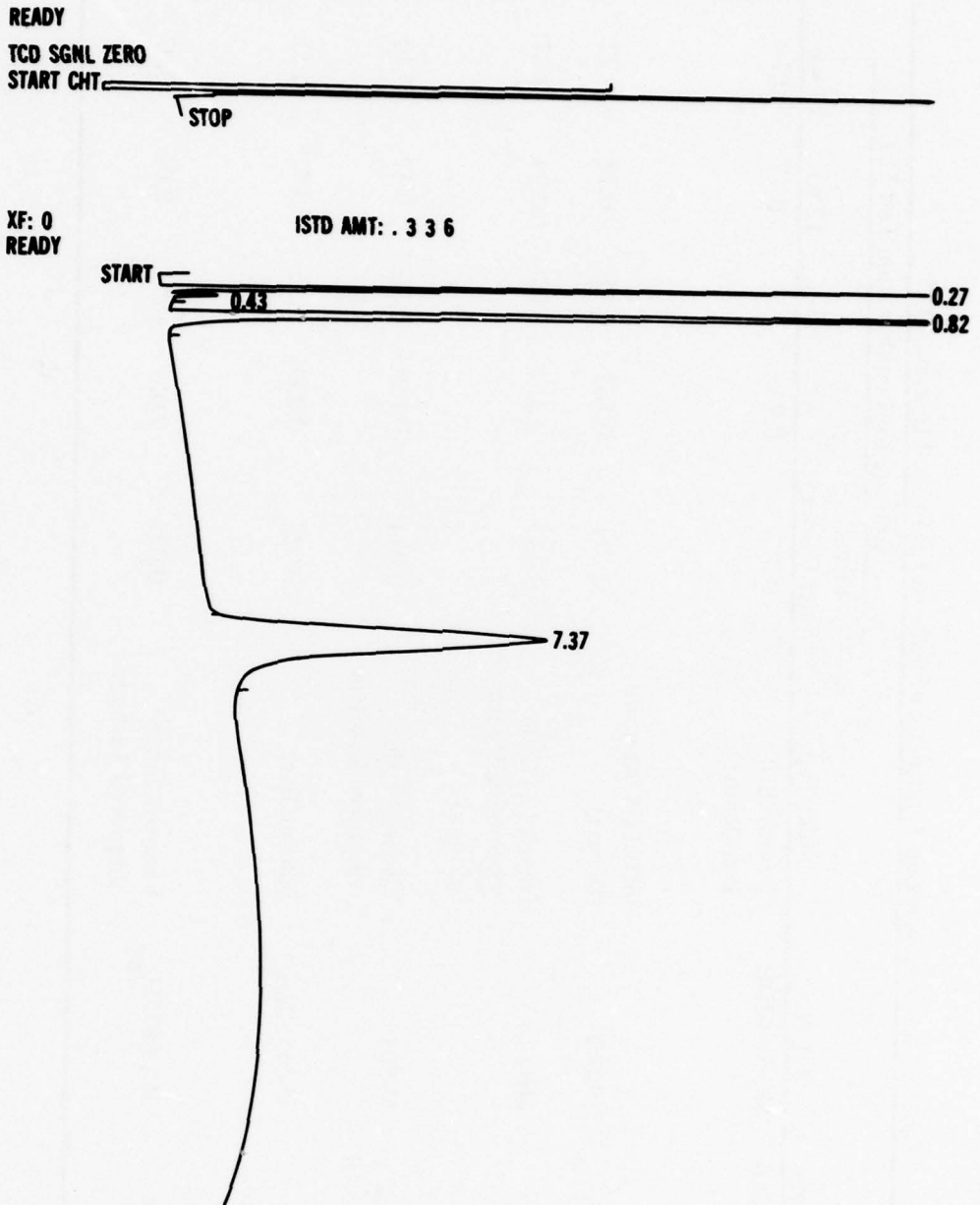


Table 2. Water Absorption Tendencies of Selected Engine and Hydraulic Oils

Specification Type	QPL No.	Fluid Type	Exposure to Humidification Test For:				
			Initial Water Content	4 wk	8 wk	12 wk	14 wk
MIL-L-2104C, Tactical Engine Oil Grade OE/HDO-10	MC-573MOD	Conventional Petroleum Oil	0.473	0.975	1.37	0.77	0.78
APG PD No. 1 Arctic Engine Oil	3908D	ISODECY Azealate (Diester)	0.194	0.362	0.45	0.21	0.22
MIL-H-46170, Less-Flammable Hyd Fluid	MF2	Polyalpha-Olefin (Synthetic Hydrocarbon)	0.037	0.314	0.47	0.24	0.27
MIL-H-6083D, Rust-Inhibited Operational	M-5056	Viscosity-Index Improved Kerosene	0.044	0.159	0.26	0.12	0.13
Candidate MIL-B-46176 Auto Brake Fluid	U-265523-1	Silicone Base	0.027	0.239	0.36	0.41	0.55
MIL-H-5606C, Aircraft Hyd Fluid (OHA)	MA-687759	Viscosity-Index Improved Kerosene	0.021	0.05	0.11	0.02	0.03

AMERY - APG-PD-1 AFTER 111 DAYS @ 80% RH



hp 5830A
ISTD

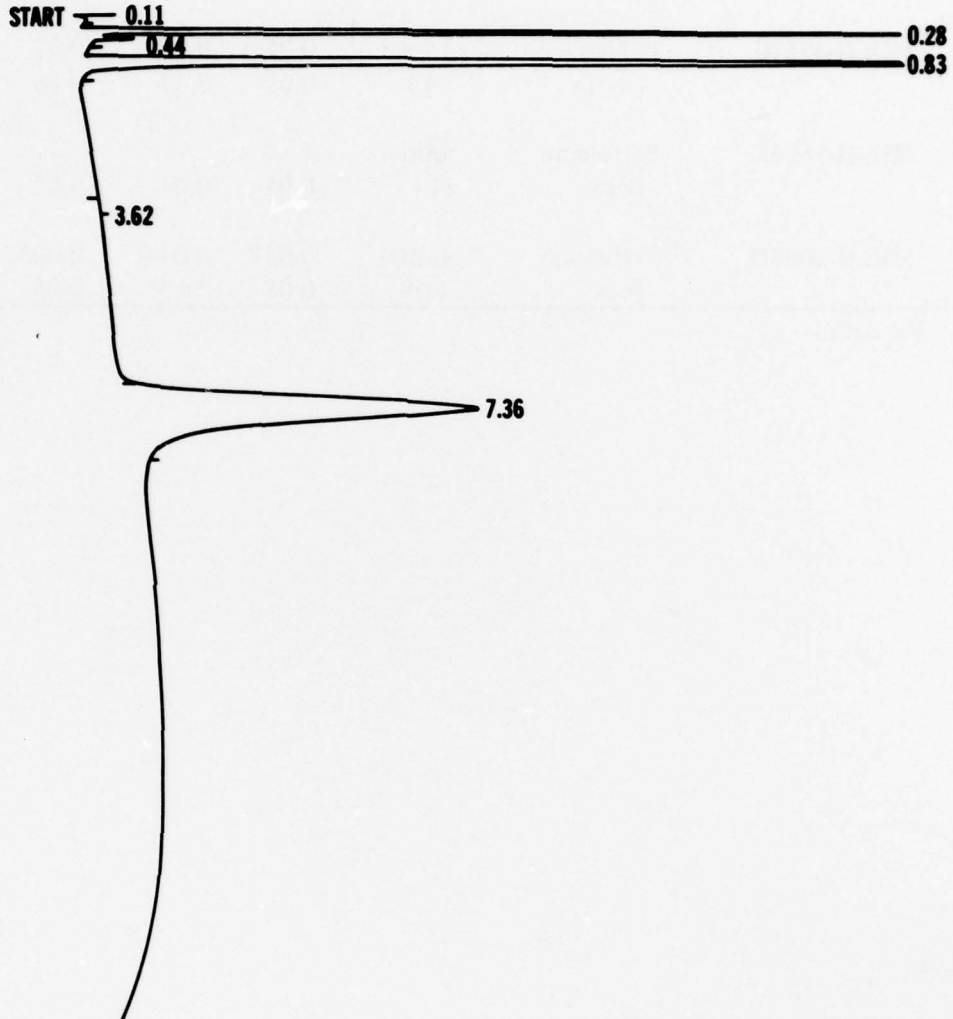
RT	EXP RT	AREA	CAL #	AMT
0.82	0.84	3671	2	0.171
7.37	7.37	7218	R 1	

XF: 1.0000 E+ 0 ISTD AMT: 3.3600 E- 1

Figure 2. Results of determining water content by CG.

TCD SGNL ZERO
START CHT

STOP



hp 5830A
ISTD

RT	EXP RT	AREA	CAL #	AMT
0.83	0.83	3613	2	0.170
7.36	7.37	7160	R 1	

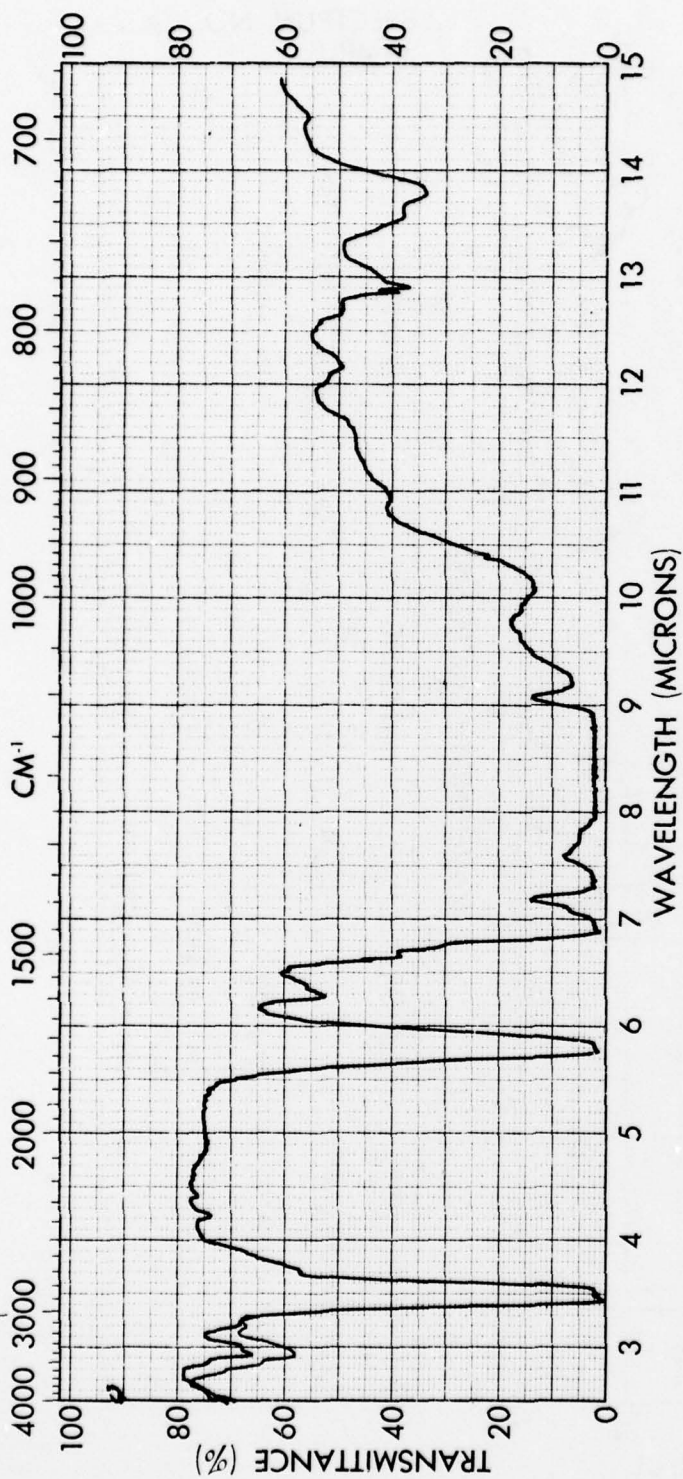
XF: 1.0000 E+ 0

ISTD AMT: 3.3600 E- 1

Table 3. Comparisons of Absorbed Water (Gas Chromatograph vs. Karl Fisher)

Specification Type	Fluid Type	Humidity Test Time (Days)	Water Content (%)		
			GC	D1744 KF	Difference GC vs KF
APG PD No. 1	Diester	Initial	0.046	0.19	0.144
Arctic Engine Oil	Diester	114	0.171	0.22	0.049
MIL-H-46170	Polyalfa- Olefin	Initial	0.02	0.04	0.02
		114	0.09	0.27	0.18
MIL-H-5606C	Petroleum Base	Initial	—	—	—
		114	0.05	0.03	0.02
MIL-H-6083D	Petroleum Base	Initial	0.047	0.044	0.003
		114	0.08	0.13	0.05

— No data.

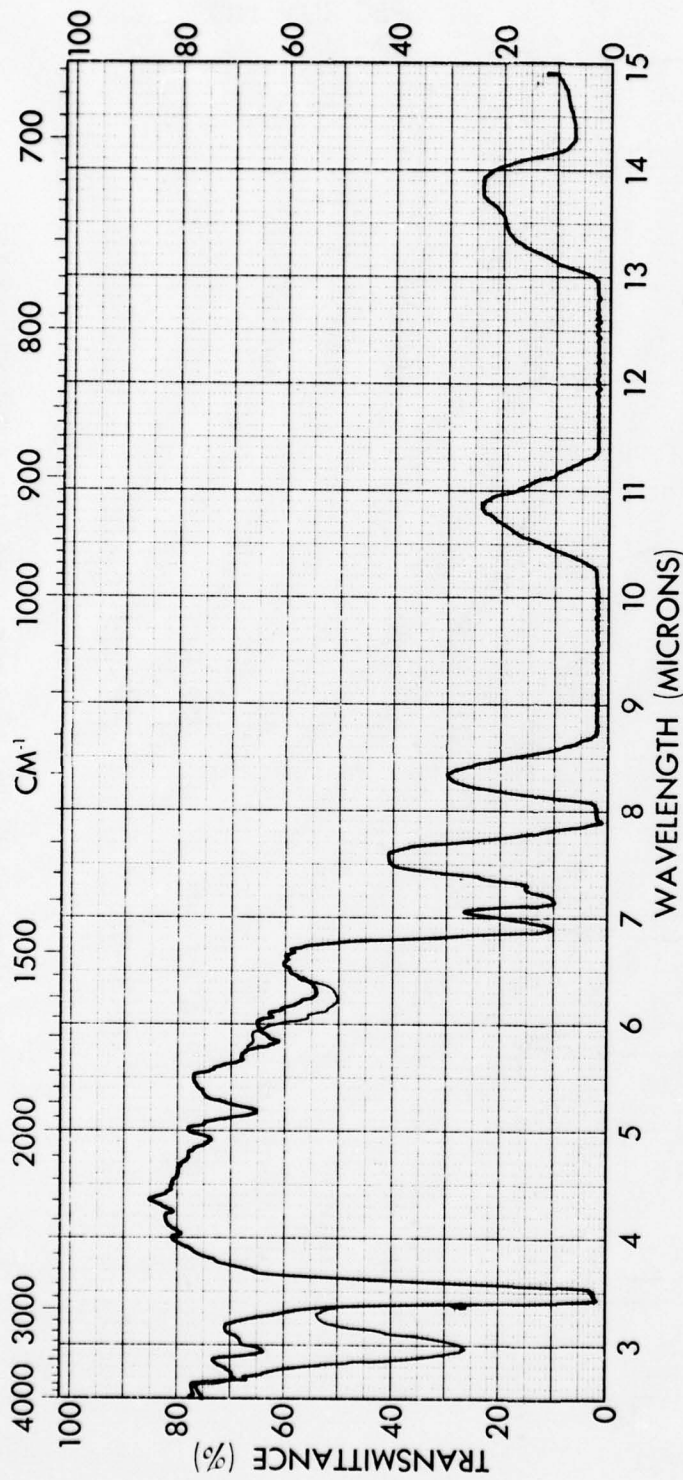


SPECTRUM NO. _____		LEGEND _____		REMARKS _____	
SAMPLE <u>APG PD 1</u>		1. _____		BLACK - AFTER TEST	
LUBRICATING OIL, INTERNAL		2. _____		RED - ORIGINAL	
COMBUSTION ENGINE, SUB-		DATE _____			
ZERO		OPERATOR _____			
		THICKNESS <u>0.25 mm</u>			

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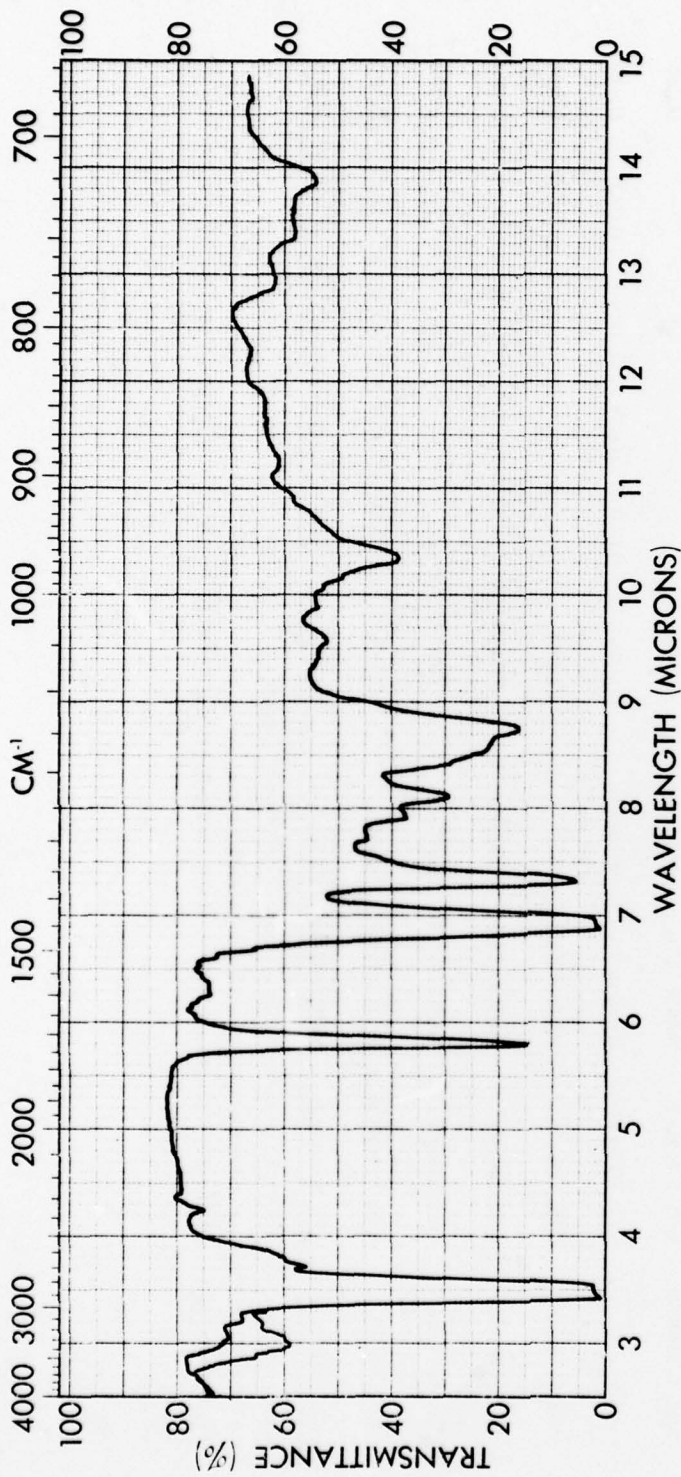
Figure 3. Spectra of three different hydraulic fluids which were exposed to the humidification procedure.



SPECTRUM NO. _____
 SAMPLE _____

SPECTRUM NO.	ORIGIN	LEGEND	REMARKS
SAMPLE SILICONE		1.	BLACK = AFTER TEST
BRAKE FLUID MIL-B-46176	PURITY	2.	RED = ORIGINAL
	PHASE LIQUID	DATE	
	THICKNESS 0.25 MM	OPERATOR	

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SPECTRUM NO. _____
 SAMPLE _____

SPECTRUM NO.	ORIGIN	LEGEND	REMARKS
MIL-H-6083		1.	BLACK = AFTER TEST
HYDRAULIC FLUID	PURITY	2.	RED = ORIGINAL
PETROLEUM BASE, FOR	PHASE LIQUID	DATE	
PRESERVATION AND TESTING	THICKNESS 0.25 MM	OPERATOR	

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Table 4. Water Absorption Characteristics of Lubricants

Specification Product	Sample	Origin	Basestock Fluid Type	Water, Wt %		Water, Wt % After Exposure ^a For:				
				Initial	2 Days	4 Days	8 Days	16 Days	32 Days	
MIL-L-23699B	QPL Sample A	NAPTC	Polyol Ester	0.030	0.219	0.254	0.274	0.276	0.269	
MIL-L-23699B	QPL Sample B	NAPTC	Polyol Ester	0.008	0.198	0.246	0.248	0.242	0.246	
MIL-L-23699B	QPL Sample C	NAPTC	Polyol Ester	0.060	0.201	0.268	0.268	0.271	0.270	
MIL-L-23699B	QPL Sample D	NAPTC	Polyol Ester	0.030	0.236	0.273	0.289	0.310	0.296	
MIL-L-7808G	QPL Sample E	NAPTC	Polyol Ester	0.020	0.205	0.205	0.201	0.236	0.221	
MIL-L-23699B	QPL Sample F	NAPTC	Polyol Ester	0.019	-	-	-	-	0.286	
	SAE 30 Grade	Basestock	Petroleum	0.019	0.019	0.015	0.019	0.012	0.019	
MIL-H-46170	QPL Sample	MERADCOM	PAO ^b	0.037	-	-	-	-	0.314	
MIL-H-6083D	QPL Sample	MERADCOM	Petroleum	0.044	-	-	-	-	0.159	
MIL-H-5606C	QPL Sample	MERADCOM	Petroleum	0.021	-	-	-	-	0.050	
APG PD No. 1 ^c	QPL Sample A	MERADCOM	Diester	0.194	-	-	-	-	0.362	
APG PD No. 1	QPL Sample B	MERADCOM	PAB ^d	0.170	-	-	-	-	0.280	

^a Samples exposed to humidification procedure (constant relative humidity of 80 percent).

^b Polyalpha-Olefin Basestock commonly referred to as Synthetic Hydrocarbon.

^c Aberdeen Proving Ground Purchase Description for Arctic Internal Combustion Engine Oil (NATO 0-183).

^d Poly-Alkylated Benzene Basestock.

- No data.

Table 5 provides a summary of water absorption tendencies of fluids/oils by generic type. Increases vary from 87 to 886 percent with Polyalkylated Benzene derivatives showing the least amount of water pickup, whereas Polyols give the greatest.

Table 5. Percent Water Increase in Basestocks after
30 Days Exposure at 80-Percent Relative Humidity

Basestock	Percent Water Increased by K. F.
Petroleum	106
Polyalpha-Olefin	748
Polyol	886
Silicone	785
Polyalkylated Benzene (Diester)	87
Hybrids	138

IV. CONCLUSIONS

Water determinations by Karl Fisher (ASTM D-1744) method gave consistently high results with formulated fluids and oils because of additive interference.

At a level of 1 percent added water, all fluids except the silicone produced a precipitate, indicating additive insolubility/incompatibility.

Upon exposure to 80 percent relative humidity for 14 weeks, the subzero automotive engine oil was the only material tested to show a precipitate.

Petroleum base fluids such as MIL-H-5606 absorb the least amounts of water. However, petroleum oils with a high level of additive treatment (MIL-L-2104C) pick up large amounts of water.

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MIL-H-5606, "Hydraulic Fluid, Petroleum Base, Aircraft Missile, and Ordnance,"
30 Sep 71.

MIL-H-6085, "Hydraulic Fluid, Petroleum Base, for Preservation and Testing," 5 Mar
73.

MIL-L-7808, "Lubricating Oil, Aircraft Turbine Engine, Synthetic Base," 10 Sep 71.

MIL-L-23699, "Lubricating Oil, Aircraft Turbine Engines, Synthetic Base," 22 Nov 71.

MIL-L-46152, "Lubricating Oil, Internal Combustion Engine, Administrative Service,"
20 Nov 72.

MIL-L-46167, "Lubricating Oil, Internal Combustion Engine, Arctic," 15 Nov 74.

MIL-H-46170, "Hydraulic Fluid, Rust-Inhibited, Fire-Resistant, Synthetic Hydrocar-
bon Base," 28 Mar 75.

MIL-B-46176, "Brake Fluid, Silicone, Automotive, All-Weather, Operational and
Preservative," 27 Mar 78.

PURCHASE DESCRIPTION

APG PD 1, "Lubricating Oil, Internal Combustion Engine, Subzero," 15 Jul 69.

APPENDIX A

HUMIDIFICATION PROCEDURE

Two bowl-form glass desiccators, 250 mm inside diameter, having matching tubulated covers fitted with No. 8 rubber stoppers, are charged with 450 ± 25 grams of reagent grade ammonium sulfate and 125 ± 10 ml of distilled water. The surface of the salt slurry shall lie within 45 ± 7 mm of the top surface of the desiccator plate. A corrosiveness test jar is placed in each of the desiccators, and the desiccators are placed in an area where temperature is controlled at $23 \pm 1.1^\circ\text{C}$ ($73.4 \pm 2.0^\circ\text{F}$) for 24 ± 4 hours. After the conditioning period, the rubber stoppers in the desiccator covers are carefully removed and 100 ± 1 ml of the test fluid is placed in each corrosion test jar by means of a pipette. The rubber stoppers are immediately placed back in the cover openings. The desiccators are left in the controlled temperature area for 30 or 60 days. On completion of the exposure to the humid atmosphere in the desiccators, the test jars containing the fluid are removed and tightly covered. Samples are removed by pipette, and water determinations are conducted.

APPENDIX B

GAS CHROMATOGRAPHY

The 5834A Gas Chromatograph is a keyboard instrument that houses a multi-function digital processor. Working with values entered via the keyboard on a special terminal unit, the processor establishes isothermal or programmed temperature control of the GC. The processor also analyzes integration data by basing its calculations on the method entered before a test run is started. During a run, the terminal traces the chromatograph and prints peak retention time and at the end of the test automatically prints the qualitative report.

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