

Use of Thin-Layer Chromatography (TLC) for Identification of Aircraft Engine Oil Components

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ABSTRACT

Thin-layer chromatography (TLC) using a silica gel adsorbent on a plastic backing, and n-heptane-acetone (95:5) developing solvent, permits useful separation of (a) esters derived from pentaerythritol, dipentaerythritol, and dibasic acids, (b) silicones, and (c) petroleum-based fluids. Esterification mixtures of both pentaerythritol and dipentaerythritol could not be resolved into their respective homologs. With benzene developing solvent, the caproate and valerate of pentaerythritol could be separated; in other respects, the solvent was less satisfactory than n-heptane-acetone because of its tendency to cause streaked chromatograms. Dibasic acid and trimethylolpropane esters could not be differentiated except by saponification and resolution of the liberated acids with benzene-acetone solvent (85:15).

TLC shows some promise for rapid, specific separations, e.g., esters and silicones from petroleum-based oils and hydraulic fluids. Even with additional refinements of the method, however, results as precise or complete as those obtained with gas-liquid chromatography (GLC) or nuclear magnetic resonance (NMR) appear improbable.

PROBLEM STATUS

This is an interim report; work on this problem is continuing.

AUTHORIZATION

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USE OF THIN-LAYER CHROMATOGRAPHY (TLC) FOR IDENTIFICATION OF AIRCRAFT ENGINE OIL COMPONENTS

INTRODUCTION

There is a need for rapid and relatively simple means of identifying the chemical type and the possible contamination or mislabeling of aircraft engine oils. In a very few instances, determinations of simple properties, e.g., color, odor, or viscosity, could be adequate. But more often, such sophisticated techniques as absorption spectroscopy, nuclear magnetic resonance (1), or gas chromatography (2) are required. This report surveys the potential of a relatively simple technique—thin layer chromatography (TLC)—to differentiate between base stocks for the formulation of Specification MIL-L-7808 oils (3) (usually dibasic acid and trimethylolpropane esters) and MIL-L-23699 oils (4) (mixtures containing dipentaerythritol, pentaerythritol, and trimethylolpropane esters), petroleum-based lubricants, hydraulic fluids and silicones.

TLC involves a moving liquid phase in equilibrium with a stationary phase; the mixture to be separated is distributed between the two phases. When the stationary phase is a simple liquid, or a liquid held on a solid support, the process is termed partition chromatography; when it is a solid, and the forces are adsorptive in nature, the process is adsorption chromatography. This investigation is primarily concerned with the latter type of separation.

Within the last few years, the applications for which TLC has proved useful have burgeoned, together with a corresponding increase in the relevant literature. In the present study, considerable information on TLC background, techniques, applications, and bibliography was obtained from technical texts on the subject (5-7).

EXPERIMENTAL PROCEDURE

A variety of adsorbents are available for the solid phase films, e.g., silica gel, kieselguhr (diatomaceous earth), alumina, and cellulose. Exploratory work was done with freshly prepared films on a glass substrate. The data of this report, however, were obtained on commercially available coatings of Silica Gel G (silica gel with a plaster of paris binder) on a flexible polyester base (8). This material was conveniently reproducible; it bypassed the time-consuming coating and drying process, as well as the nuisance and possible health hazard of handling dry silica powder. A kit, supplied by the manufacturer, permitted rapid equilibration of the vapor with the solvent, thus speeding up development of the chromatograms.

Application of samples in suitable solvents was accomplished by touching a loaded glass capillary (ca. 1-mm I.D.) to the plate; this is termed "spotting." Sample weights ranged from 15 to 100 micrograms. Too large a sample resulted in streaks or large spots which could not be satisfactorily resolved; too small samples caused minor oil components and additives to be missed. So, the choice of proper sample size was often a matter of trial and error.

The chromatograms were developed by the ascending method in which the "spotted" plate is set edgewise in the developing solvent until the latter has risen a standard

distance (100 mm in this case). Distances traversed along the plate by individual components, relative to that by the solvent, are termed " R_f " values. The reproducibility of these values varied from approximately ± 10 percent in the lower range to ± 5 percent in the higher range, and it was deemed advisable to include, where possible, internal standards in all runs.

Chromatographic patterns were visualized by spraying air or oven-dried plates with appropriate agents. Dichlorofluorescein and ultraviolet illumination gave satisfactory results with oils. Acid components of esters were isolated by saponification (2) and were visualized with bromocresol green adjusted to pH 4.0 to 5.5. Both visualizing agents were employed in 0.2-percent concentration in ethanol and were applied with an air brush.

Several solvent systems were screened for their efficiency in separating the oils chromatographically; they are listed in Table 1 together with brief summaries of their characteristics. Composition changes in mixed systems due to loss by preferential evaporation of the more volatile components caused poor reproducibility when a mixed solvent was employed to develop more than one chromatogram. Mixed solvent systems were therefore renewed immediately before each run. No solvent system provided completely satisfactory separation of all of the fluids. The most generally useful systems were n-heptane-acetone (95:5) and benzene.

Chromatograms of the acid components of esters, isolated by saponification as previously indicated, were developed with benzene-acetone (85:15).

Table 1
Solvent Systems Investigated for TLC Use With Esters,
Silicones, and Petroleum Oils

Solvent	Remarks
Benzene	Acceptable separations, some streaking
Monochlorobenzene	Fair separations, some streaking
Benzene: Acetic Acid 99:1	Fair separations
Benzene: Acetic Acid 98:2	Fair separations
Benzene-Acetone 95:5	High R_f values for most oils
n-Heptane	Poor separations
n-Heptane-Acetone 95:5	R_f zero for all esters
n-Heptane-Acetone 90:10	Acceptable separations
n-Heptane-Ethyl Ether 99:1	Acceptable separations
n-Heptane-Ethyl Ether 94:6	R_f zero for all esters
n-Heptane-Ethyl Ether 80:20	Poor separations
n-Heptane-Ethanol 95:5	Fair separations but ether component excessively volatile
n-Heptane-Acetic Acid 95:5	Poor separation of esters
Ethyl Ether	Acetic acid component wetted only lower portion of plate
Acetonitrile	Excessively volatile
Acetonitrile: Benzene 50:50	Esters and silicones followed solvent front, petroleum fluids did not migrate
	All fluids followed solvent front

RESULTS

Oils Studied

The oils examined by TLC are listed with their appropriate abbreviations and code names in Tables 2 and 3. A summary, from Refs. 1 and 2, of the compositions of commercial MIL-L-23699 formulations and base stocks is also included.

Table 2
Characterized Fluids Studied by TLC

Fluid	Code
Pure Esters:	
Diisooctyl Azelate	DOA
Bis(2-ethylhexyl)sebacate	DOS
Pentaerythritol Tetravalerate	PETV
Pentaerythritol Tetracaproate	PETC
Dipentaerythritol Hexacaproate	DPHC
Silicones:	
Polydimethylsiloxane*	DMS
Polymethylphenylsiloxane †	MPS
Petroleum-Based:	
Jet Engine Lube	1010
General Purpose Lubes	2135
Steam Turbine Lube †	V-78
Hydraulic Fluid AN-VV-O-366b	366b

*Viscosity at 100 °F ca. 40 cs.

†Viscosity at 100 °F ca. 250 cs.

‡ Contains ca. 3% monoester.

Table 3
Identification of Commercial Esters and Formulations Studied by TLC

Code	Type	Ester Proportions	Main Acid Components	Average Acid Chain Length
L-1	MIL-L-7808 formulation	Adipate base	-	-
C-1	Base Stock	TMP‡ ester	C ₉	9
NRL-S-300*	MIL-L-23699 base stock†	PE§ (100)	C _{5,6,7,8,9}	6.3
NRL-S-303*	MIL-L-23699 base stock†	DP§	C _{5,8,10}	6.4
NRL-S-304*	MIL-L-23699 base stock	PE, DP (96:4)	C ₅	5.1
NRL-S-305*	MIL-L-23699 formulation†	PE, DP (82:18)	C _{5,6,7,8,9}	6.1
NRL-S-306*	MIL-L-23699 formulation†	PE, DP, TMP (3:23:74)	C _{5,7,8,10}	7.5
NRL-S-307*	MIL-L-23699 formulation†	PE, DP, TMP (87:10:3)	C _{iso-5,5,7,8,10}	6.6
NRL-S-309*	MIL-L-23699 formulation†	PE, DP (87:13)	C _{iso-5,5,9}	6.4

*Data summarized from Refs. 1 and 2.

†Esterification mixture.

‡TMP = Trimethylolpropane.

§PE = Pentaerythritol ester.

§DP = Dipentaerythritol ester.

Separations with n-Heptane-Acetone Solvent

Figure 1 summarizes the R_f values obtained with the n-heptane-acetone solvent system. The lengths of the major axes of the ellipses are indicative of the reproducibility of the R_f values. Figure 1(a) indicates that the system provided useful separations of laboratory preparations of dibasic acid esters, pentaerythritol esters, and dipentaerythritol esters from each other. There was, however, no differentiation between closely related homologs, e.g., PETV and PETC.

R_f values of commercial esters and lubricant formulations are given in Fig. 1(b). Those of the trimethylolpropane ester and the adipate-based MIL-L-7808 formulation (C-1 and L-1) were almost identical, so these esters are not separable. The triester contained a minor quantity of an unidentified component with an R_f value of about 13.

Commercial base stock esters of pentaerythritol or dipentaerythritol with mixed acids could not be resolved into their component compounds; instead, their chromatograms were somewhat elongated continua. This is not surprising when it is considered that the reaction of pentaerythritol with a mixture of four acids can result in as many as thirty-one esters. Esterification mixtures of pentaerythritol were readily separated from those of dipentaerythritol, e.g., NRL-S-303 and 304.

R_f values of the MIL-L-23699 formulations reflected the presence of the component esters. Thus, chromatograms of NRL-S-305, 307, and 309 confirm that they were based primarily on pentaerythritol esters, with lesser concentrations of dipentaerythritol esters. NRL-S-309 contained a minor component, possibly tripentaerythritol ester, with a R_f value of 8. On the other hand, the chromatogram of NRL-S-306 revealed dipentaerythritol esters to be present, together with either dibasic acid or trimethylolpropane esters; the minor concentration of pentaerythritol ester known to be present (Table 1) did not manifest itself. The procedure for differentiating esters of dibasic acids from those of trimethylolpropane will be discussed in a subsequent section. All MIL-L-23699 formulations showed the presence of two additives.

Oxidation of MIL-L-23699 formulations at 400°F for 48 hr produced no change in the chromatograms of their component esters. All of the oxidized additives, however, were so strongly adsorbed that their R_f values were zero.

Figure 1(b) lists some examples of formulations which differ sufficiently in composition that they are separable (unlike NRL-S-305 and 309, for example). The MIL-L-7808 formulation L-1 was readily separated from its mixtures with either PETC or its related esterification mixture NRL-S-300, and also from DPHC. A mixture of DOA and NRL-S-305 was also easily resolved.

Figure 1(c) lists the results obtained for silicones. R_f values for the polydimethyl- (DMS) and polymethylphenyl (MPS) siloxanes did not differ materially, and a combination of the two could not be resolved. Chromatograms of phenyl-containing siloxanes streaked badly. Mixtures of DMS with DOS, C-1, or PETC and DPHC gave good separations.

Chromatograms of petroleum-derived oils all followed the solvent front and could not be separated from each other (Fig. 1(d)). The chromatogram of the steam-turbine lubricant (V-78) showed two minor components to be present, one of which is believed to be a monoester. Differentiation of petroleum-based fluids from those of nonpetroleum origin was unambiguous because of the much larger R_f values of the former.

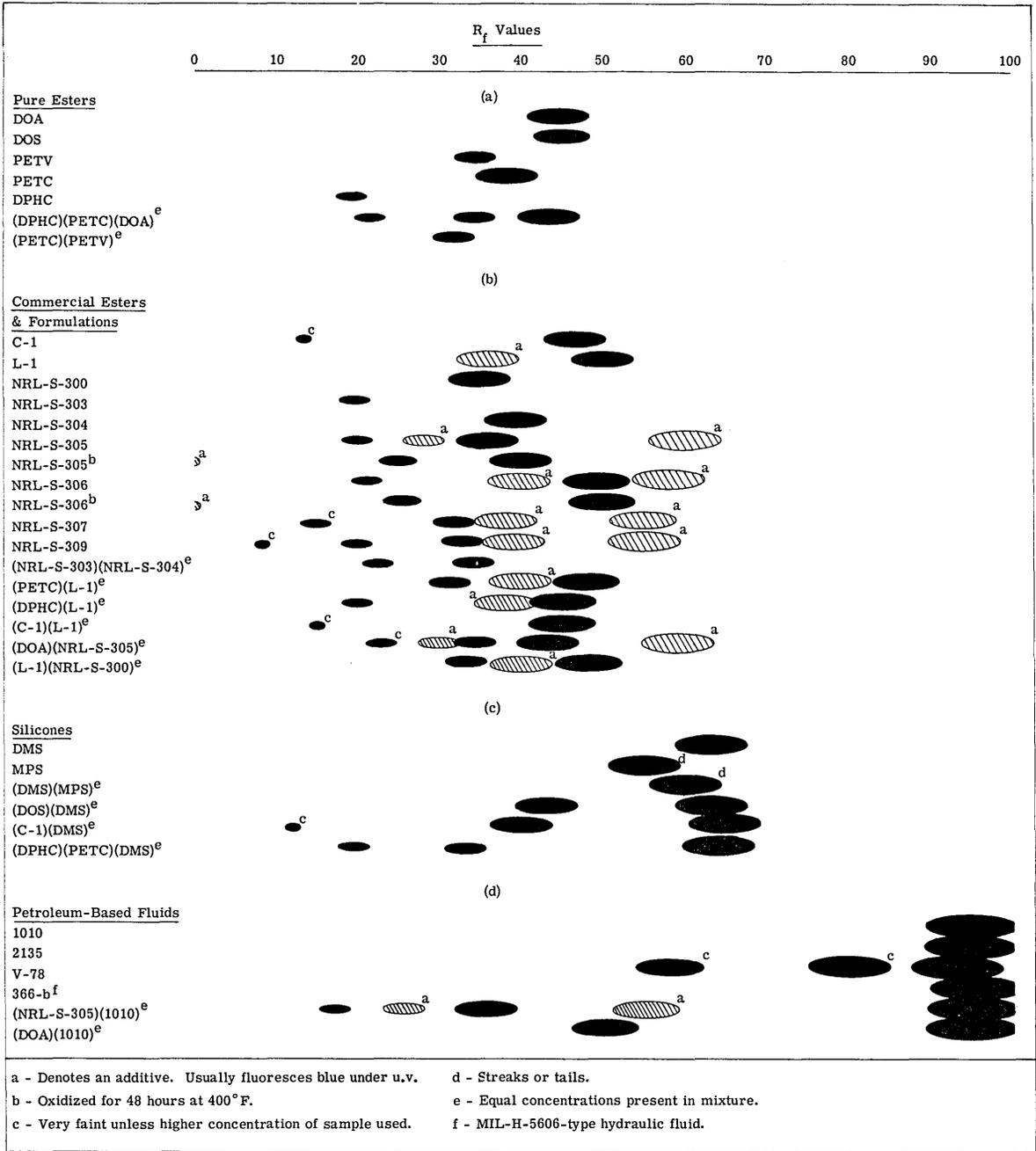


Fig. 1 - R_f values of test fluids (concentrations 1 to 3% in n-heptane) with n-heptane-acetone (95:5) solvent system

The second solvent with some usefulness in separating the oils was benzene. Its most serious deficiency was that it caused badly streaked chromatograms unless sample concentration was limited to 0.5 percent or less. Under these conditions, however, low concentrations of minor components and additives were often overlooked. Figure 2 lists some of the results obtained with simple systems.

Benzene-based R_f values for pentaerythritol and dipentaerythritol esters were smaller than with the n-heptane-acetone system. Despite this diminished response, a marginal separation of PETC from PETV was achieved; this had not been possible with n-heptane-acetone. R_f values for the dibasic acid and trimethylolpropane esters approximated those obtained with the previous system. The chromatogram of NRL-S-307 showed the pentaerythritol ester component, but not the dipentaerythritol ester. As with the n-heptane-acetone system, petroleum-based oils followed the benzene solvent front, but the minor components of V-78 were not observed, probably because of the small sample used. The behavior of the silicones differed markedly in the two solvent systems; whereas in the previous solvent R_f values were of the order of 60, they were 90 or higher in benzene.

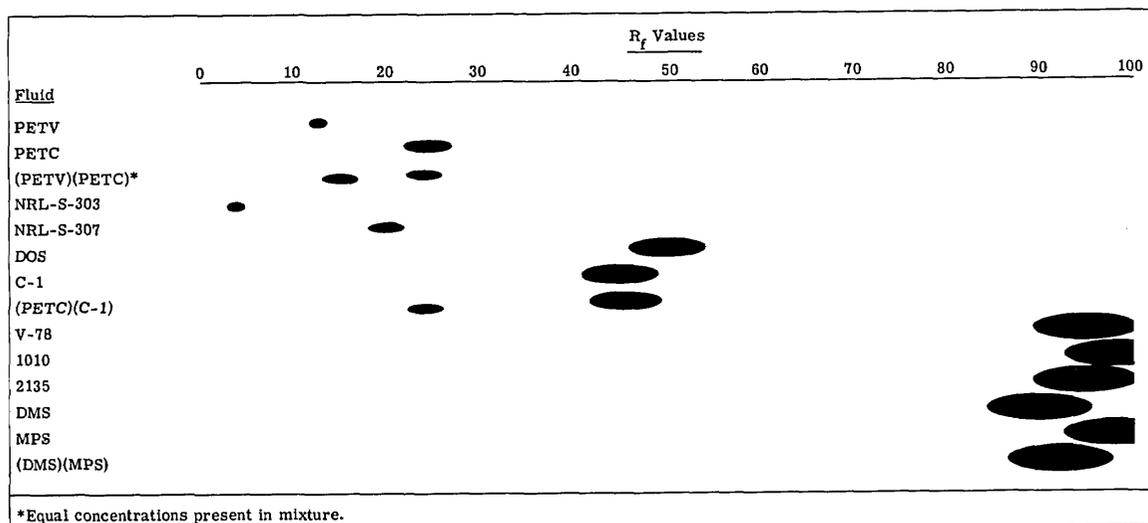


Fig. 2 - R_f values of test fluids (concentration 0.5% in n-heptane) with benzene solvent system

Determination of Acid Components of Esters

Independent determinations of the acid components of esters provide valuable additional information regarding the identity of the parent material. TLC is a useful tool for this type of determination and a number of special purpose systems have been reported, but none for the lower fatty acids. One such procedure investigated in the present work was based on "reversed phase" chromatography. This is essentially partition chromatography in which the silica gel support is saturated with polydimethylsiloxane. A highly polar developing solvent (acetic acid and water) is employed. Results were unsatisfactory because the acetic acid could not be released from the plate, even after heating for 3 hr at 100°C. The bromocresol green visualizing agent was thus unable to distinguish between the chromatograms and the background.

The procedure finally adopted (benzene-acetone (85:15) solvent and uncoated plates) was not without deficiencies, but nonetheless provided useful data. Solutions of the purified acids in ethanol were used for calibration; they are designated as $C_x^{H^+}$, "x" being the acid

chain length. Thus hexanoic acid is $C_6^{H^+}$. In 0.2-percent concentration, these acids gave excessively faint chromatograms when visualized with bromcresol green. Although one-percent concentrations gave visible chromatograms, these streaked to such an extent that meaningful R_f values had to be based on the leading edge of the streaks and not on the chromatogram midpoints. In general, reproducibility was not as good as with the oils. The acids released by saponification of the esters are coded according to their origin, e.g., DOS^{H^+} , $NRL-S-307^{H^+}$, etc. Averaged results are given in Fig. 3.

It is apparent that the polar characteristics of the monobasic acids in the $C_6^{H^+}$ to $C_9^{H^+}$ range do not differ sufficiently to permit satisfactory separation under the conditions described. Dibasic acids, on the other hand, were clearly delineated by their small R_f values. The saponification-TLC procedure is thus a useful method for determining the presence or absence of esters of dibasic acids in a lubricant sample.

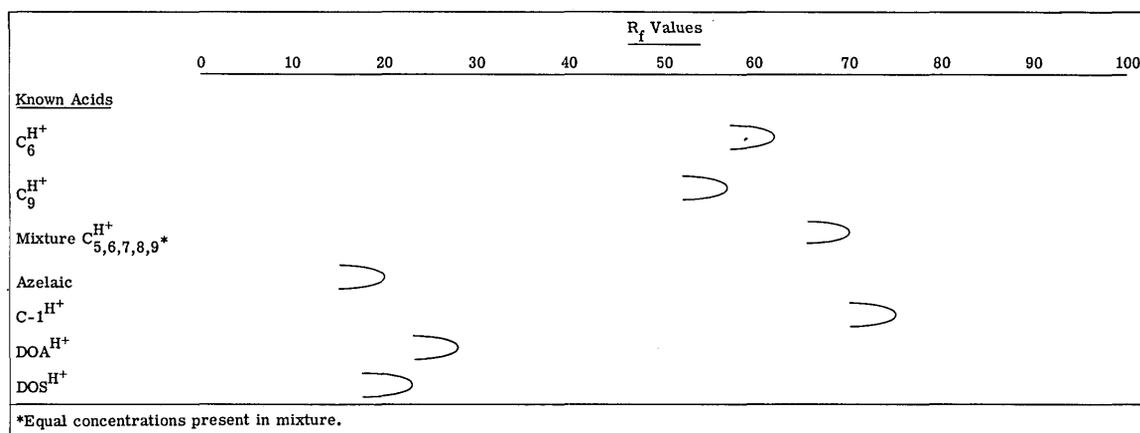


Fig. 3 - R_f values (averaged values calculated from leading edges of streaks) for acids (concentration 1% in ethanol) with benzene-acetone (85:15) solvent system

DISCUSSION

Although TLC has inherent limitations, it can provide useful information regarding the composition of a variety of lubricants. Given any binary, or most ternary systems, it is possible to separate and identify (a) esters from pentaerythritol, dipentaerythritol, trimethylolpropane, and dibasic acids, (b) silicones, and (c) petroleum oils. The technique will thus readily show the presence of about 5 to 10 percent concentration of a contaminant, such as petroleum in engine oils from which it is supposed to be absent. The technique is applicable to oxidized as well as to unused lubricants. It can be employed to "fingerprint" an unused synthetic lubricant formulation provided the composition is unique.

On the negative side, the TLC procedure used would not be particularly sensitive to moderate changes in component proportions made by the lubricant vendor, and addition or deletion of minor components might be overlooked entirely. Since it is impossible (or at least extremely difficult) to resolve esterification mixtures or the individual acids, changes in the proportions of the acid components of polyol esters would not be revealed. Except in unusual circumstances, formulations of lubricants conforming to the same specification cannot be differentiated in engine oils containing makeup additions from different vendors.

While a more intensive investigation of solvent systems, adsorbents, and special techniques would undoubtedly permit more definitive separations to be attained, the general limitations outlined above would still apply. Thus, TLC appears to be useful if it is employed for screening, rough classification, or for the quick recognition of contaminated oils. For definitive information, recourse must be had to such techniques as nuclear magnetic resonance (NMR) and gas-liquid chromatography (GLC).

SUMMARY AND CONCLUSIONS

An exploratory study was made of the potential of thin-layer chromatography (TLC) for separation and identification of the components of engine lubricants, particularly of the MIL-L-23699 type. Using commercially prepared plates of Silica Gel G on a flexible plastic backing, simple and mixed acid esters of pentaerythritol could be separated from those of dipentaerythritol with a n-heptane-acetone developer solvent system. The mixtures within each class, however, could not be resolved. Benzene permitted marginal separation of the valerate and caproate of pentaerythritol, but otherwise showed no improvement over n-heptane-acetone. Esters of trimethylolpropane and of dibasic acids could not be distinguished as such; the mono- and dibasic acids released by saponification could, however, be separated from each other by benzene-acetone, thus permitting identification of the ester class. Petroleum-based lubricants and hydraulic fluids followed the solvent fronts of both n-heptane-acetone and benzene, and were thus easily distinguished from the other fluids studied. Polydimethylsiloxane could be separated by n-heptane-acetone from esters of dibasic acid or trimethylolpropane, and from a mixture of pentaerythritol and dipentaerythritol esters. A polydimethylphenylsiloxane streaked excessively in the solvent mixture. No streaking occurred in benzene with either silicone, but both materials followed the solvent front and thus could not be differentiated.

TLC promises to be a convenient tool for the rapid preliminary characterization of several classes of lubricants and hydraulic fluids. It cannot, however, give information as precise or as complete as does gas-liquid chromatography or measurements of nuclear magnetic resonance.

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14. KEY WORDS	LINK A		LINK B		LINK C	
	ROLE	WT	ROLE	WT	ROLE	WT
<p>Chromatographic Analysis Engine and Motors Contamination Petroleum Esters Silicones Lubricants</p> <p>TLC (Thin-Layer Chromatography)</p>						

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