

Waters

Lab Highlights

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AN/PP,OS/MD,QC/PL/OT

LINSEED MODIFIED ALKYD RESINS

The combination of linseed oil with alkyd resins is a very popular consumer coating formulation. The term alkyd usually refers to polyesters that are modified with a triglyceride oil (or acid). In general, alkyd resins are the reaction product of a polybasic (phthalic acid*), a polyhydric alcohol (glycerol) and a fatty acid/oil (linseed). The term alkyd can be broken down into "al" pertaining to the alcohol and "kyd" which refers to the acid (changed from "cid"). The basic function of the oil is to control the drying rate (and therefore the crosslinking rate), increase sample solubility and to increase the flexibility of the total mixture. Gel permeation chromatography (GPC) can be an extremely valuable analytical tool to the coatings formulator.

LINSEED OIL

Linseed oil is obtained via solvent extraction from flaxseed which is approximately 35% oil. The drying rate of linseed oil is rated high as compared to other typical coating oils due primarily to the high degree of unsaturation (linolenic acid, 3 double bonds). The drying rate of the oil can be increased by adding metallic driers (cobalt and magnesium naphthenates) with an increase in temperature (not boiling). A linseed oil that has been bodied via metallic driers is considered a "boiled" linseed oil even though this name is misleading. Figure 1 compares the GPC analysis of three linseed oils. The raw linseed oil, which is 90% C₁₈ fatty acids which only differ by the amount of double bonds (oleic, linoleic, linolenic), consists of one large peak. A more exact profile of the raw linseed oil may be obtained using a reversed-phase method with a fatty acid analysis column. However, in the case of bodied linseed oils (Figure #1) GPC can be very useful. Comparison of linseed oil #1 to linseed oil #2 reveals that linseed oil #2 has a larger amount of higher molecular weight material which would affect the drying rate of modified alkyd resins.

MODIFIED ALKYD RESINS

Figure #2 is the comparison (overlay) of two consumer oriented linseed modified alkyd resins. A summary of the calculated molecular weight averages is given below:

SUMMARY OF CALCULATED MOLECULAR WEIGHT AVERAGES**

<u>Resin</u>	\bar{M}_n	\bar{M}_w	\bar{M}_z
A	3,308	9,405	22,114
B	3,667	13,985	48,985

Differences between the two resins can be seen in both the polymer region and the low molecular weight region. These differences can affect viscosity, crosslinking rate (drying), brushing ability, hardness and other important properties which could result in a "bad" product.

See reverse side for chromatograms.

* The phthalic acid (PA) is added to the system in the form of phthalic anhydride which forms PA during reaction.

** These calculated molecular weight averages are based upon linear, narrow distribution polystyrene standards.

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FIGURE 1

CONDITIONS:

Columns: 500, 100Å ULTRASTYRAGEL™
Sample Concentration: 0.5% w/v
Injection Size: 100 µl
Mobile Phase: THF
Flow Rate: 1 ml/min
Chart Speed: 1 cm/min
Detector: RI, 4X

LINSEED OILS

Raw Linseed Oil: Solid Line
Boiled Linseed Oil #1: Broken Line
Boiled Linseed Oil #2: Dotted Line

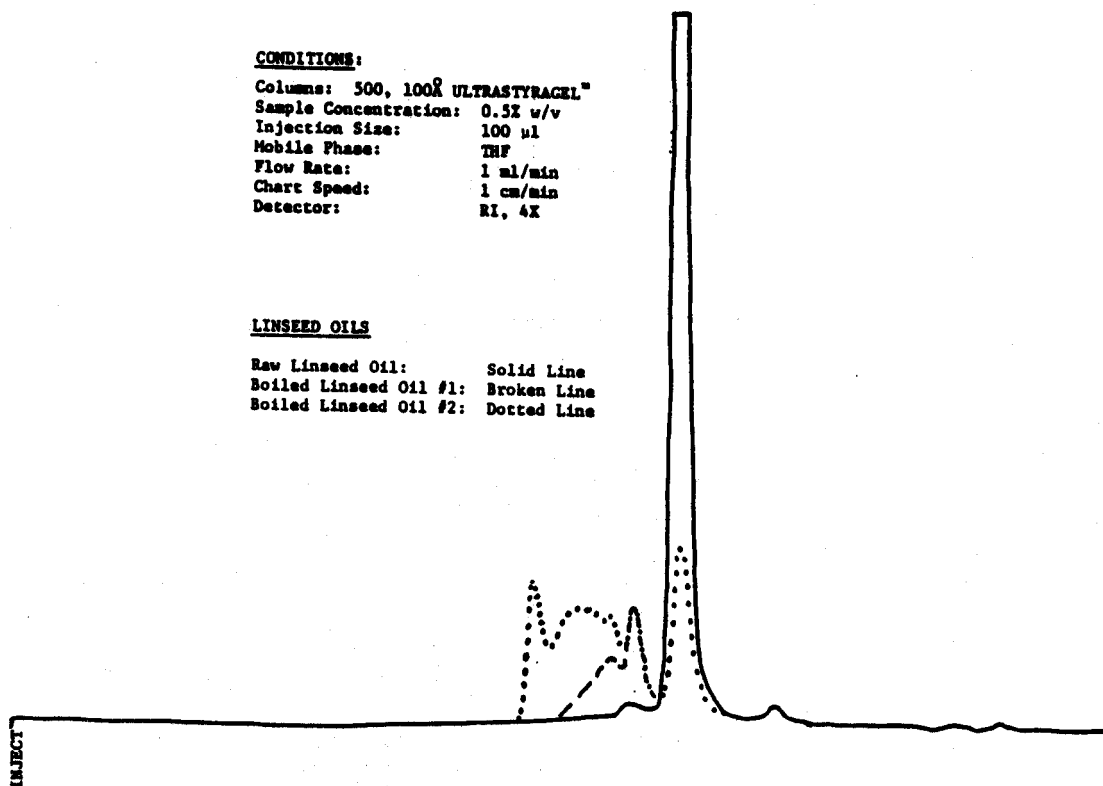
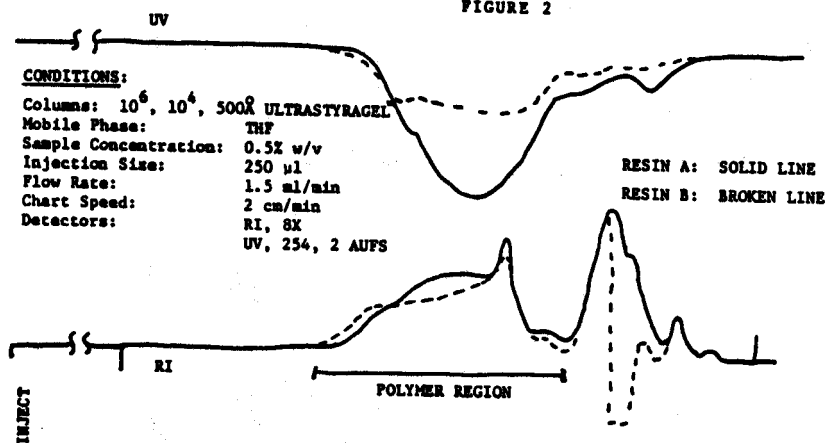


FIGURE 2

CONDITIONS:

Columns: 10⁶, 10⁴, 500Å ULTRASTYRAGEL
Mobile Phase: THF
Sample Concentration: 0.5% w/v
Injection Size: 250 µl
Flow Rate: 1.5 ml/min
Chart Speed: 2 cm/min
Detectors: RI, 8X
UV, 254, 2 AUFS

RESIN A: SOLID LINE
RESIN B: BROKEN LINE



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