

TABLE III  
Bleaching Tests for Uniformity of New Lot of Earth  
Manufactured in June 1964

Cottonseed		
Laboratory	Bag number	Bleach
Paymaster	62	2.7-2.7
	101	2.7-2.8
	224	2.7-2.6
	230	2.7-2.7
	327	2.6-2.7
	447	2.6-2.6
Anderson, Clayton	56	2.7-2.8
	101	2.8-2.8
	181	2.7-2.7
	285	2.6-2.6
	364	2.6-2.6
	450	2.6-2.6
Barrow-Agee	56	2.9-2.8
	128	2.9-2.8
	181	2.9-2.9
	285	2.8-2.7
	364	2.5-2.7
	450	2.7-2.7
Soybean		
Laboratory	Bag number	Bleach
A.D.M.	12	1.6-1.7
	110	1.7-1.6
	182	1.6-1.6
	291	1.5-1.7
	341	1.6-1.7
	418	1.7-1.6
A. E. Staley	56	1.7-1.7
	128	1.6-1.6
	182	1.6-1.6
	230	1.6-1.6
	327	1.6-1.6
	418	1.6-1.6
Central Soya	62	1.4-1.4
	128	1.4-1.4
	224	1.4-1.4
	230	1.4-1.4
	341	1.4-1.4
	418	1.4-1.4
Procter & Gamble	12	2.0-2.0
	110	2.0-2.0
	182	2.0-2.0
	291	2.0-2.0
	327	2.0-2.0
	447	2.0-2.0

into 3 portions and one portion sent to each of three committee members. Nine committee members were selected and each was sent 6 bag samples. These six samples were one from each of the 75 bag lots. The committee members were to make bleaches in duplicate by the Official AOCs Method on each of these samples. In this experiment they were to use their own refined oil on all tests.

Following are tabulations on the bleaching tests on cottonseed and soybean oil showing the percentages earth used and the Wesson colors obtained. Also the bleaching tests for uniformity giving the bag number and the results. On Mr. Coleman's spectro curve dated 6-6-64 the "Proposed Lot" is the 1964 lot of

### • Letter to the Editor

## A Molecular-Still Sample Reservoir Offering Precise Flow Control

**M**OLECULAR DISTILLATIONS are often complicated by imprecision and contamination introduced by conventional stopcocks used to admit a sample into the still. Ordinary vacuum stopcocks become difficult to adjust accurately when under high vacuum used with such distillations. Even slight rotations of the stopcock admit much more sample than can be efficiently handled by the apparatus.

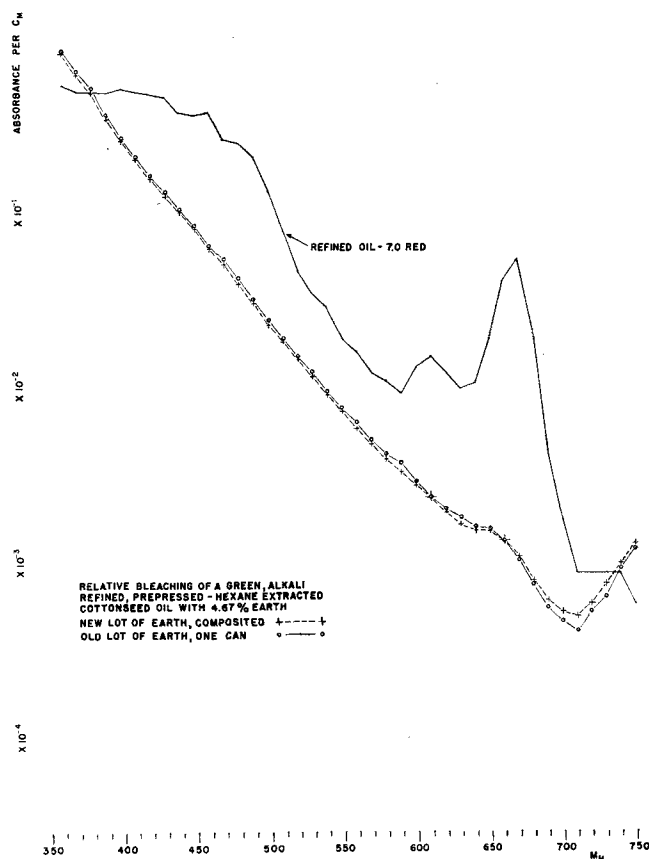


Fig. 1

earth, the "Present Lot" is our 1954 standard earth (Fig. 1).

In the testing of various lots of natural earth it was noted that the pH of the earth was one of the determining factors in obtaining a bleach that was satisfactory. The pH test was made on 10 g of earth and 40 ml of water. It was found that for an earth to be equivalent to the current standard the pH should be about 4.0.

Respectively submitted,

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The reservoir diagrammed in Figure 1 affords critical adjustment of sample flow so that distillation on a drop-by-drop basis may be performed with ease. The 1¼ mm needle valve A (Catalog No. 795-609-0114, Fischer and Porter Company, Warminster, Pa.) used as a flow controller is leakproof at pressures down to 10<sup>-4</sup> mm Hg. If required, vacuum grease may be applied well behind the valve seat. Such application

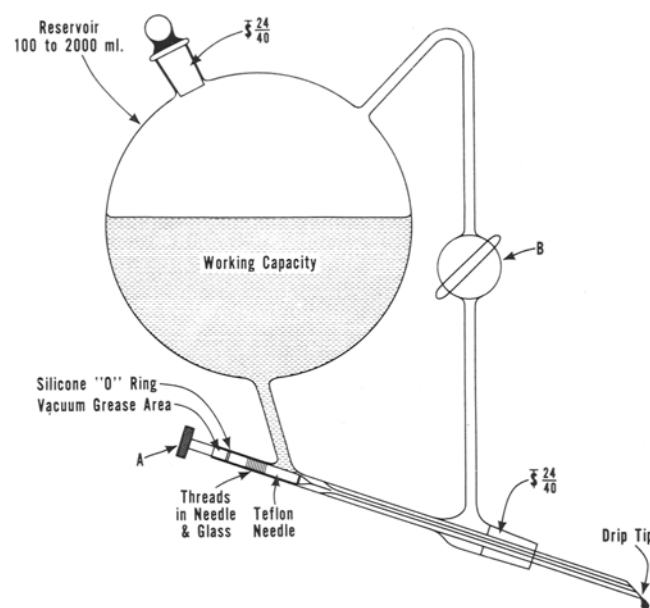


FIG. 1. Diagram of reservoir showing important features (Parts not drawn to uniform scale).

offers a distinct advantage over conventional stopcocks which invariably contaminate the sample by allowing it to flow over the lubricated area.

Samples may be degassed before distillation through vacuum stopcock B. When extremely high vacuum is not required, traces of solvent may be degassed from the sample during distillation. The completely accurate and fine flow control of the needle valve makes this operation possible. With conventional stopcocks it is often impossible to adjust flow if degassing is not carried out before distillation.

Reservoirs based on this design can save much time and frustration during molecular distillations. These savings, combined with low cost and ease of fabrication, make them particularly attractive for either small samples or large quantities sufficient for a full day's operation.

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## Modified Silver Ion Thin-Layer Chromatography

SINCE THE INCEPTION of the technique, many papers have appeared in the lipid field citing the use of the silver ion in thin-layer chromatography (TLC), countercurrent distribution, and column chromatography. The results presented here demonstrate that ammoniacal silver ion plates have advantages over those prepared from aqueous solutions.

Commercially available Silica Gel G (30 g) was slurred with 60 ml of 12.5% silver nitrate in 28–30% ammonium hydroxide. The procedure of Barrett et al. (1) was used for preparing aqueous silver nitrate layers. Two parts of 12.5% aqueous silver nitrate solution were mixed with one part silica gel (wt/wt). Uniform 0.25-mm layers were then spread on 2 × 20-cm and 20 × 20-cm glass plates with a Colab No. 2810 applicator (Colabs Lab., Inc., Chicago Heights, Ill.) modified in this laboratory (2). After the chromatoplates had air dried for 30 min, they were activated in an oven for 30 min at 110°C. Approximately 40 μg of the fatty acid methyl esters (obtained from the Hormel Foundation, Austin, Minn.) in 5 μl of chloroform were applied approximately 1 cm from the lower edge of the plate. Development of the chromatoplates was carried out in a chamber saturated with chloroform-ethanol 99:1 (v/v). The separations were visualized either by spraying with a 0.2% solution of 2', 7'-dichlorofluorescein in ethanol, or by charring according to the procedure of Privett and Blank (3) or Barrett et al. (4). The charring procedure was used when the results were to be documented by photography. Because of uncontrollable environmental factors, such as relative humidity, the ammonium hydroxide and aqueous prepared plates were always handled simultaneously from preparation through development to insure the validity of the comparison. Solvents and other reagents were reagent grade and used without further purification.

Methyl stearate, methyl oleate, and methyl linoleate

were used for comparison of the resolution obtainable on each of the different plate preparations. Approximately 40 μg of each methyl ester was spotted on the plates and developed by the ascending technique. Figure 1 shows the resolution of the esters on Silica Gel G plates impregnated with the ammonium hydroxide solution of silver nitrate (right) and with the aqueous silver nitrate solution (left). Resolution on the ammonium hydroxide silver ion plates was always superior to that of the aqueous prepared silver ion plates; in addition, the resolving power of the plates was maintained for a longer period. Silver ion plates prepared with aqueous solution lost their resolving power, presumably through the absorption of water, approximately twice as fast as the ammoniacal plates under the same conditions. On the silver ion plates, without ammonia, the distance of separation between the saturates and the monoenes was much smaller than the distance between the monoenes and the dienes (Fig. 1). With the ammoniacal silver ion plates, these distances were more nearly equal. No hydrolysis on the plate was observed in either case, as judged by the absence of polar hydrolytic products appearing as spots near the origin. Plates prepared by both methods appeared to have equal loading capacity as determined with methyl linoleate (10–100 μg). An increase in concentration was accompanied by an increase in  $R_f$  value.

Special precautions should be taken to protect silver ion TLC plates from moisture, air, organic matter, and light (causing them to darken). After exposure to direct sunlight for 1 hr, no noticeable darkening of the ammoniacal silver ion plate was observed; however, plates prepared without ammonia turned brownish black during the same period. Although the rate of discoloration is greatly reduced in the ammoniacal silver ion plates, it is not prevented: after two or three days unprotected plates on a laboratory bench