water by volume was added to the sample before testing and then subtracted from the results measured.

The accuracy of the method for water was such that in the analyses of 6 known mixtures varying in water from 1 to 3.5 vol % the average deviation of the measured values from the expected values was 0.047 vol %. The maximum difference was 0.1 vol %.

The measured cyclohexane in the same 6 samples differed from the actual by an average of 0.22 vol %. The maximum deviation was 0.3 vol % for a single reading. All measured values were higher than expected values.

Two series of drying tests were made to ascertain whether any gossypol binding would occur in the oven during the drying operation.

Samples containing 11.7% moisture were placed in the oven in wire baskets for periods of 1 hr with the oven set at 180, 190, 200, 210 and 220F. An unheated sample was also analyzed for free and total gossypol.

None of the dried samples were found to contain more bound gossypol than the undried flakes.

In a second test the oven temperature was held constant at 180F and the drying period varied from 60 to 210 min. Here again, analyses of the dried flakes revealed no evidence of lowered free gossypol as a result of drying under these conditions.

Relationships between flakes moistures and water in miscellas when extracting with the binary and ternary azeotropes are given in Figure 1.

Patterns of water movement between the flakes and the solvent mixtures are shown in Figure 2.

Flakes were dried to a moisture content of 3% before the binary ceased to remove water from the flakes.

A point at which the flakes ceased to remove water from the ternary was not established.

ACKNOWLEDGMENTS

Appreciation is expressed to A. C. Wamble and S. P. Clark of the Cottonseed Products Research Laboratory for technical counsel received.

REFERENCES

1. King, W. H., and V. L. Frampton, JAOCS *38*, 497–499 (1961).
2. Field, E. (E. I. duPont de Nemours & Co.), U.S. 2,302,608 (1942).
3. Frampton, V. L., Oil Mill Gazetteer *66*, 33–36 (1961).
4. Mann, G. E., F. L. Carter, V. L. Frampton, A. B. Watts and C. Johnson, JAOCS *39*, 86–90 (1962).

[Received June 6, 1965—Accepted September 2, 1965]

The Equivalence Test of the Earth to Become Known as Our 1964 Standard Earth

Composite samples were taken during the manufacturing of the 18 tons (450—80 lb bags) of earth that was manufactured by Bennett-Clark in June 1964. This sample was mixed, divided and portions sent to each of the 14 members of the committee. Tests were made on this earth to determine the exact percentage to be used. Each laboratory used its own refined oil, thus giving us a wider range of oils. Seven laboratories used soybean oil, and the other seven used cottonseed oil. Each laboratory made 4 bleaches in duplicate on the same oil. These bleaches were:

1. 4.67% new earth on c/s or 3.0% on s/b.
2. 5.67% new earth on c/s or 3.5% on s/b.
3. 4.17% new earth on c/s or 2.5% on s/b.
4. Standard percentage of the current standard (1954) earth (4.67% for cottonseed and 3.0% for soybean).

The above bleaching tests were made in accordance with the Official AOCS Method. Mr. Coleman and Mr. Clause were asked to run a spectro curve on tests No. 1 and No. 4.

A second experiment was made to determine if there was a significant difference between the bags of earth. This test was made in accordance with the procedure given by Mr. David McLean of the AOCS Statistical Committee. An outline of the procedure is as follows: The 450 bags were divided into six sections of 75 bags each. Three bags were sampled from each of the 75 bag lots. The number of the bag to be sampled from each lot was determined by placing slips of paper numbered from 1 to 75 and drawing three numbers at random. The same procedure was used for the other 5—75 bag lots. The sample from each bag was mixed well and divided

TABLE I
Bleaching Tests with Cottonseed Oil
Type and Percentage of Earth Used

Laboratory	AOCS Standard Earth 4.67%	June 1964 Earth 4.67%	June 1964 Earth 5.17%	June 1964 Earth 4.17%
Paymaster	2.8	2.8	2.8 (5.27%)	2.8 (4.27%)
	2.9	2.8	2.7 (5.27%)	2.8 (4.27%)
Milwhite	2.6	2.8	2.6	2.9
	2.6	2.8	2.6	2.9
Anderson, Clayton	2.8	2.7	2.6	2.8
	2.9	2.7	2.5	2.8
Barrow-Agee	2.8	2.8	2.7	3.0
	2.8	2.8	2.7	3.0
Filtrol	6.6	6.2	5.9	6.5
	6.5	6.3	6.0	6.6
Paymaster	5.2	5.2 (A very dark oil)		
Hahn	3.1	3.1	2.9	3.3
	3.1	3.1	3.0	3.3

TABLE II
Bleaching Tests with Soybean Oil
Type and Percentage of Earth Used

Laboratory	AOCS Standard Earth 3.0%	June 1964 Earth 3.0%	June 1964 Earth 3.5%	June 1964 Earth 2.5%
Votator	1.2	1.2	1.0	1.3
	1.2	1.3	1.1	1.4
A.D.M.	1.7	1.6	1.3	1.9
	1.6	1.7	1.2	2.0
A. E. Staley	1.6	1.6	1.4	1.8
	1.6	1.6	1.4	1.8
Central Soya	1.4	1.4	1.3	1.6
	1.4	1.4	1.3	1.6
Bennett-Clark	1.6	1.6 (9 times)		
	1.6			
Procter & Gamble	2.1	2.0	1.8	2.2
	2.1	2.0	1.8	2.2
Hahn	2.5	2.5	2.3	2.6
	2.5	2.5	2.4	2.7

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

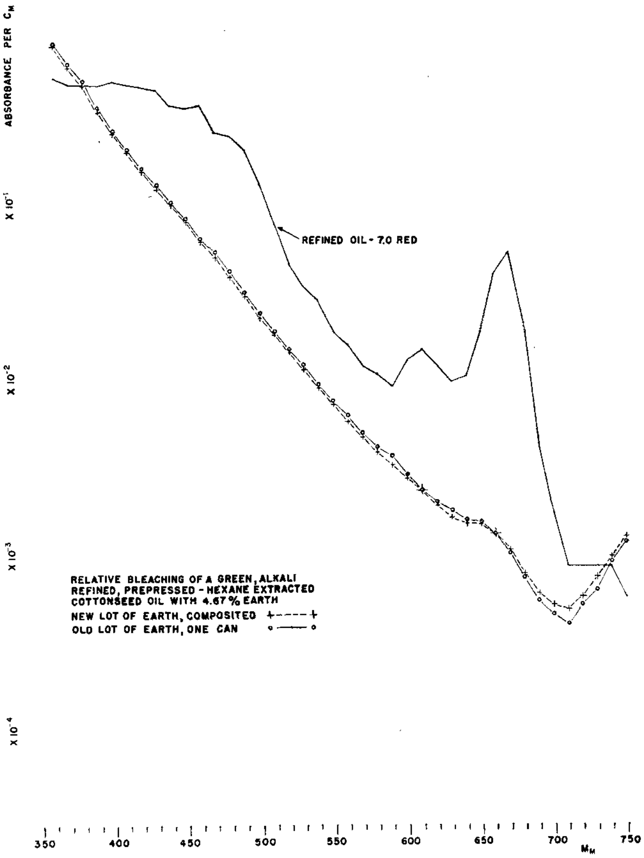

FIG. 1

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,

P. W. BATEMAN
R. J. BELL
R. T. CLAUSE
W. T. COLEMAN
ALEXANDER GREENTREE
L. H. HODGES
K. E. HOLT
W. J. JOHNSON
WALTER KITCHENS
J. W. MCEWAN
C. E. MCMICHAELS
A. D. RICH
E. H. TENENT, JR.
E. R. HAHN, Chairman

• *Letter to the Editor*

A Molecular-Still Sample Reservoir Offering Precise Flow Control

MOLECULAR 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.

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^{-4} mm Hg. If required, vacuum grease may be applied well behind the valve seat. Such application