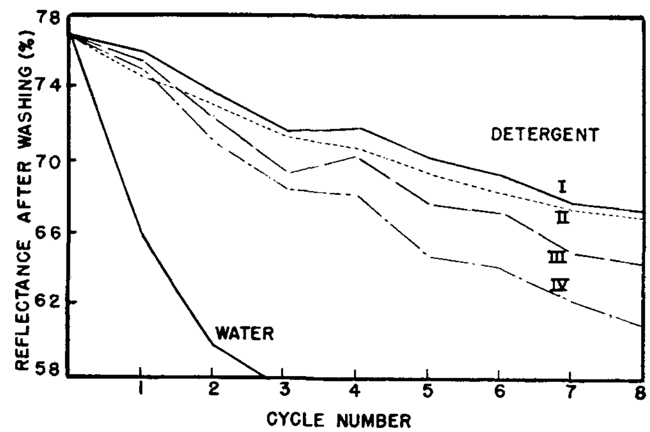

FIG. 5. Typical results obtained with new multicycle procedure.

four detergents are the same in all runs and in good agreement with the results of the roll-towel program.

The new test outlined above admittedly deals with the slow build-up of moderately light soils whereas the housewife may be more concerned with the immediate removal of stubborn dirt, such as on collars and cuffs. There is good reason to believe however that these more difficult soils are not different in composition from the lighter ones but are simply more deeply ingrained due to high moisture conditions during soiling. Since the new test procedure outlined here was designed primarily to measure removal of ingrained soil, it would be expected that detergents rating high in this test would be capable of removing tenaciously held natural soils.

TABLE V

Final Reflectances After Five Soil-Wash Cycles

Detergent	Run No.		
	1	2	3
I	70.3	70.7	69.0
II	69.3	69.7	68.3
III	67.7	68.0	67.0
IV	64.7	66.3	65.0

In order to demonstrate this some swatches were soiled on the machine under very high moisture conditions. After drying, these soils were found to be quite difficult to remove in a single wash, but again the four detergents described above were found to be ranged in the following decreasing order of effectiveness: I, II, III, IV.

It is therefore concluded that the new cotton detergency test described here shows considerable promise as a method for accurately evaluating detergents on a laboratory scale.

Acknowledgments

The authors wish to thank Marvin Kopp of this laboratory for his valuable assistance in carrying out the experimental work involved; also H. F. Schiefer of the Bureau of Standards for his advice on the abrasion machine, and C. E. Stevens of this company for designing the oscillating washer used here.

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Two Useful Accessories for the Beckman Spectrophotometer Model DU[1]

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THE slide carrier which comes with the Model DU Beckman Spectrophotometer for cells of various sizes is somewhat inconvenient to use especially for oil chemists. In the determination of fat compositions it often becomes necessary to change the cell lengths frequently. If the holder supplied with the instrument is used together with the regular Beckman cells, it becomes necessary to unscrew the partition and move it over when changing from one cell length to another.

To overcome this difficulty and to facilitate the use of various cells rapidly and sequentially a special holder was designed. The original suggestion for a similar design came from B. A. Brice of the Eastern Regional Research Laboratory of the U. S. Department of Agriculture. He had designed a holder for the demountable type of cells similar to the one described here. The present design was developed to hold the circular cells regularly supplied with the Beckman instrument.

A top view of the holder is shown in Figure 1. The cells are placed in the "V" grooves and are put in place in the light beam by means of the locking adjustable screws. The hole through the center axially was made merely to reduce the weight of the carrier.

Figure 2 shows a bottom view of the carrier. The small "V" on the left rides on the supporting rod in the same manner as the regular Beckman circular cell holder. The "step" on the right side of the carrier rests on the supporting rod closest to the phototube compartment. To relieve further the weight of the carrier some sections were removed from the bottom as shown in the picture.

[1] Presented November 2 at the 1949 Fall Meeting of the American Oil Chemists' Society, Chicago, Ill.

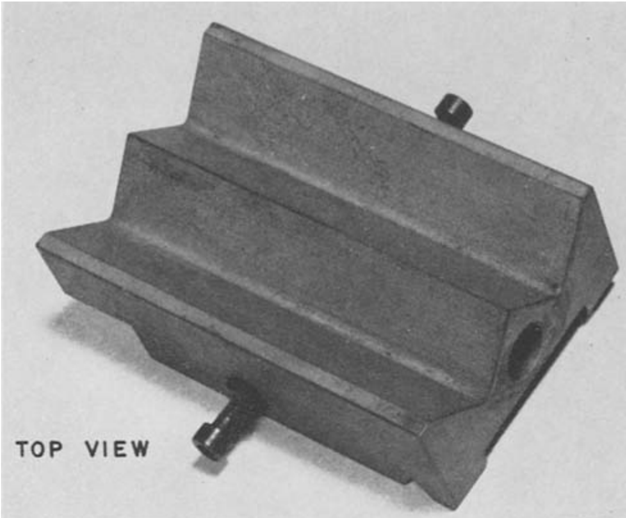

FIG. 1. Top view—Cell holder for Beckman spectrophotometer.

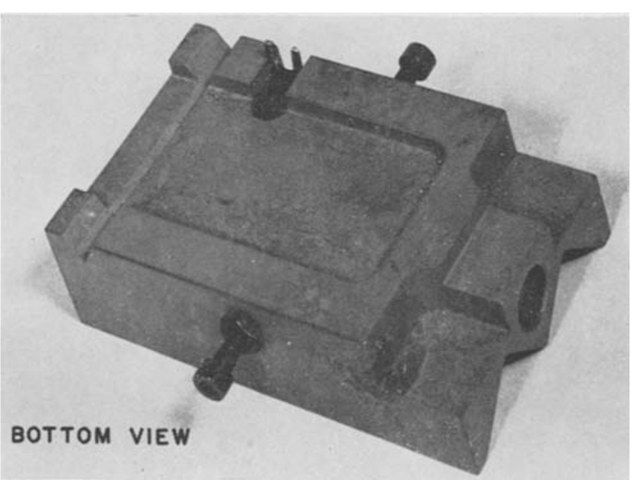

FIG. 2. Bottom view—Cell holder for Beckman spectrophotometer.

To facilitate insertion of the carrier into the compartment the small clip shown at the top of Figure 2 was attached to the bottom of the holder. This fits on the regular plunger as supplied with the Beckman instrument. This carrier can then be used by lifting out the regular Beckman carrier and setting this one down in its place.

The dimensional details of the carrier are shown in Figure 3. The material used for the present holder was bronze, chemically blackened. Other lighter materials such as aluminum could be used. In addition to the carrier changes, the only change made on the compartment itself was the removal of a small amount of metal from the boss holding the light tight plunger so that the adjustment screw on that side would clear properly. This was blacked over again before use.

In this carrier the click stops are not utilized, but the cells are centered by means of the adjustable screws. The click stop was not removed. In operation it is merely ignored. Centering the cells is easily accomplished by opening the slit wide, passing a suitable beam of light through it and the cell, and on to a piece of white paper held in front of the phototube compartment. The screws are then set and locked in place. The carrier described above has been in satisfactory operation in our laboratories for almost a year now.

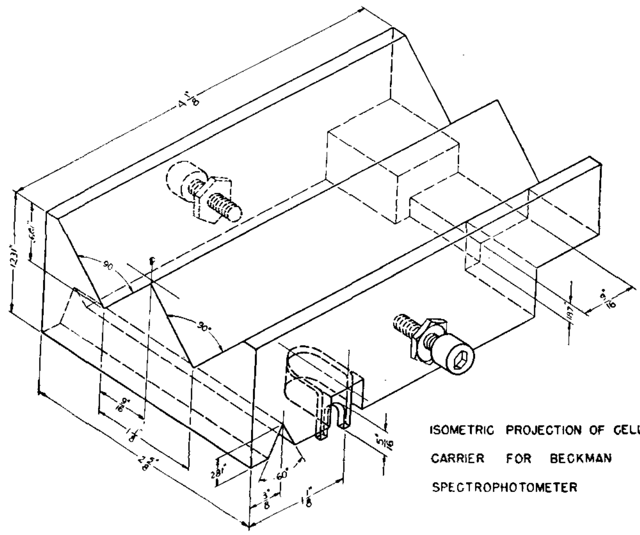

FIG. 3. Isometric projection of cell carrier for Beckman spectrophotometer.

ANOTHER useful accessory for the Beckman Spectrophotometer, which we had to design by necessity, is a holder for plastic fat samples such as margarine, lard, or shortening on which the reflectance is to be read.

The holder is shown in Figure 4. It was made of 1/4" transparent Plexiglass sheet stock and consists of two parts. The bottom section shown in the center has four small steel pins on the corners which fit into the holes drilled through the top piece. In addition, the top piece has a center hole cut out to hold the fat sample. This hole was made approximately the same size as the hole in the black metal cover of the reflectance sample holder. When assembled together to hold a fat sample, the appearance is as shown on the left. Notice that small notches are cut on one side properly to line up the pegs in the correct openings. In the picture the container is holding a sample of margarine.

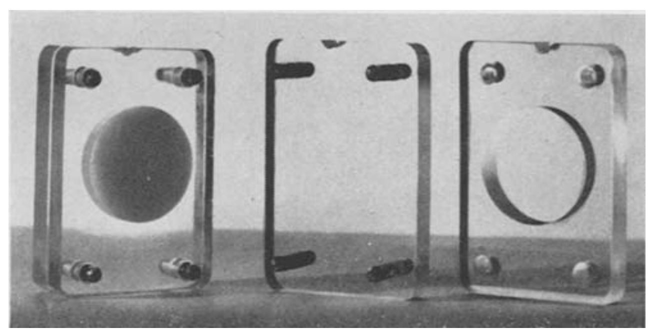

FIG. 4. Plastic fat sample holders for reflected light measurements.

The pegs protrude below the bottom plate also and are fitted into small holes drilled into the metal plate of the sample holder directly over the tension spring. This prevents the sample from sliding when the lid is pushed down. The holder was made from plastic

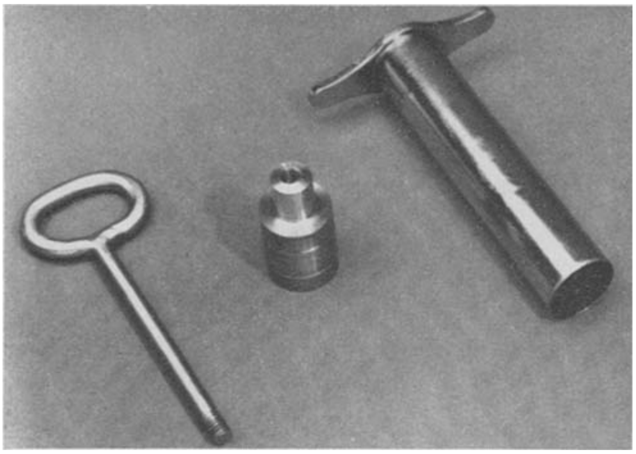

FIG. 5. Disassembled sampler for use with plastic fat sample holder.

to cut down heat radiation to or from the sample, which becomes quite a problem at times with metal holders. The above holder is also easily disassembled for cleaning.

The sample may be introduced into the sample holder in a number of ways. One such way is illustrated in Figure 5. The sampler was made from a cork borer of the appropriate size fitted with a piston and plunger. The piston has to be inserted from the cutting end because of the manner in which the cylinder is attached to the handle of the borer.

In using the above described sampler, the piston comes in contact with the top of the fat surface being sampled. This is satisfactory for samples where the condition of the original surface is not a prerequisite.

For persons desirous of obtaining a sample without disturbing the original surface the following procedure is suggested. The handle of the cork borer should be removed. This cylinder is then inserted into the fat, and the cylinder and fat core withdrawn and inverted. Insert the piston on the cutting side of the cylinder and push out the fat core. The first surface emerging will then be undisturbed and in its original condition.

The assistance of Mr. Garapolo, of the Engineering Department Experimental Shop, in the drawings and shopwork required for these units is gratefully acknowledged.

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Adsorption Analysis of Lipids. IV. Fractionation of Cholesterol and Ergosterol[1]

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PREVIOUS work (1, 2, 3, 4) has indicated that cholesterol and ergosterol can be separated by chromatographic adsorption methods. In no instance however have data of purity and recovery been sufficiently complete to assess the efficiency of the adsorption analysis technique as a practical tool for the isolation of sterols. Before the complex problem of the analysis of soybean sterols could be undertaken, information concerning the efficiency of adsorption analysis was required. The cholesterol-ergosterol system was chosen as a model system primarily because of availability of materials and ease of analysis of mixtures by physical methods.

This paper describes the fractionation of cholesterol, ergosterol, and mixtures of the two upon columns of alumina by use of the "flowing chromatogram" technique. The efficiency of fractionation was evaluated by determinations of weights, melting points, optical rotations, and spectral adsorption measurements on eluted fractions.

Materials

Cholesterol used was of commercial grade: m.p. 146-148°C.; $[\alpha]_D^{25°C.} = -39.76°$. The constants reported in the literature for pure cholesterol are as follows: m.p. 150-151°C.; $[\alpha]_D = -39.5°$ (5). The cholesterol purified by the procedure of Anderson (6) was also used: m.p. 148.5-149.5°C.; $[\alpha]_D^{25} = -39.87°$.

Commercial ergosterol (m.p. 147-151°C.; $[\alpha]_D^{25} = -119.80°$) was purified by recrystallizing twice from acetone, according to the method of Lamb *et al.* (7). Its spectral absorption coefficients (α = gms./liter^{1} cms.^{1}) were: 31.95 at 2.710 Å; 33.23 at 2,810 Å; 19.48 at 2,930 Å. Its melting point was 149-156°C., and $[\alpha]_D^{25°C.} = -119.53°$. Values reported in the literature are: 28.38 at 2,710 Å; 29.16 at 2,810 Å; 16.79 at 2,930 Å (8); m.p. 163°C.; $[\alpha]_D = -132°$ (5).

Aluminum oxide (Harshaw's Al-2 powder[3]) was used for adsorbent and was selected for its characteristics of filtration, fractioning power, and high recovery of solutes. It is in Class V of the Brockman Scale of adsorbent strengths. Petroleum ether (B.P. 50-60°C.) was purified by distillation and passage through a silica column to remove ultra-violet absorbing impurities (9).

Experimental Procedure

Considerable exploratory work was necessary before the conditions of adsorption were determined. This survey work was, of course, complicated by the colorless characteristics of the sterols. Choice of the adsorbent was a compromise between the fractionating power of strong adsorbents and the high recoveries of weaker adsorbents. Choice of solvents was

[1] Presented at fall meeting, American Oil Chemists' Society, October 31, November 1 and 2, 1949, in Chicago, Illinois.

[2] One of the laboratories of the Bureau of Agricultural and Industrial Chemistry, Agricultural Research Administration, U. S. Department of Agriculture. Report of a study made under the Research and Marketing Act of 1946.

[3] The mention of this product does not imply that it is endorsed or recommended by the Department of Agriculture over others of a similar nature not mentioned.