Otherwise the one leg may be capped off. Likewise hoses can be looped between two unused outlets of the distribution head F if all six outlets are not required.

In our particular case it was convenient to center the stand over an opening in a steam bath and allow the six exit tubes to drain into it. In other circumstances it would be necessary to arrange for disposing of the water by rubber tubing or some other means.

Electrical connections are made by using a three-wire cord and grounding it to part C. A clamp should also be used to anchor it at this point. To shield both the electrical connections and the hot plate, a 2-in. strip of perforated aluminum was attached to three of the heater clamp-down bars. Control of the heater output is made by either a variable transformer or a SCR motor speed control.

It should be pointed out that the clips by themselves will not hold the condensers. With the two hoses attached they will be prevented from sliding vertically through the clips.

R. R. LOWRY
Department of Agricultural Chemistry
Oregon State University
Corvallis, Oregon

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Improved Titration of Cyclopropenes

THE DISCOVERY that the positive Halphen test given by seed oils of many species of the order Malvales, including *Gossypium hirsutum* or Upland cotton, is associated with the presence of the biologically active cyclopropene acids, malvalic and sterculic, has created interest in rapid methods of analyzing for these compounds. Titration methods, based on a modification of the procedure for determining oxirane oxygen with the Durbetaki reagent, have been published (1,2). Essentially these methods consist of dissolving the sample in three parts glacial acetic acid and one part benzene, then titrating at 55C with 0.1 *N* hydrobromic acid in glacial acetic acid with crystal violet as indicator.

In this laboratory the most highly purified samples of methyl sterculate and methyl malvalate almost invariably analyzed 83% to 86% cyclopropenes by hydrobromic acid titration, yet other criteria of purity indicated that the values should have approached 100%. Experience with the titration method indicated that, as the concentration of the glacial acetic acid in the solvent was decreased, the purity of the sample as determined by titration increased. It was also found that benzene was superior to acetic acid as a carrier for hydrobromic acid. Not only was benzene a good solvent for hydrobromic acid, but the solutions were chemically very stable. By using a highly purified methyl sterculate which analyzed 84.5% by the usual titration method (1), a series of titrations was conducted at 55C with 0.1 *N* hydrobromic acid in benzene and with several ratios of acetic acid to benzene in the solvent for the sample. The results obtained are shown in Table I. Difficulty was encountered in titrating sample No. 5 because the crystal violet used as indicator would not remain in solution in the benzene, and the color at the end-point was very faint.

The products from titration Nos. 1, 2, and 5 as well as the product obtained by the usual titration were analyzed by thin-layer chromatography. Also analyzed was a portion of the original sterculate after refluxing with glacial acetic acid. The sterculate refluxed with glacial acetic acid produced two spots, and the sterculate titrated in benzene solution (no acetic acid present) produced one spot. Both the sterculate titrated in glacial acetic acid and that titrated in the usual manner produced three spots; in each case the major spot matched that which was found on titrating in benzene only; the two minor spots matched those produced by the reaction product obtained on heating the methyl sterculate with glacial acetic acid. The reaction product from titration No. 2 also produced three spots, but the two minor spots were quite small.

TABLE I

Titration No.	Solvents, ml.		Sterculate found, %
	Benzene	Acetic acid	
1	0	20	86.6
2	14	6	93.7
3	19	1	99.3
4	19	1	100.0
5	20	0	108.8(?)

The above is considered proof that the acetic acid used as a solvent in the usual hydrobromic acid titration of cyclopropenes actually reacts with the cyclopropenes and makes the determination inaccurate. Apparently, free hydrobromic acid catalyzes this unwanted reaction under the relatively mild conditions of titration.

In preliminary experiments it was found that the hydrobromic acid titration of methyl epoxystearate in the absence of acetic acid gave slightly higher contents of epoxystearate than did similar titrations in which acetic acid was substituted for most of the benzene. Hydrobromic acid reacted, of course, much more rapidly with methyl epoxystearate than with methyl sterculate.

An improved titration procedure which employs a solution of hydrobromic acid in toluene is now being developed and will be published. It is anticipated that the improved procedure will permit the accurate determination of both cyclopropenes and oxirane oxygen.

R. O. FEUGE
ZIGRIDA ZARINS
JOHN L. WHITE
RAIFORD L. HOLMES
Southern Regional Research Laboratory
U.S. Department of Agriculture
New Orleans, Louisiana

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