ficiently intense to create in every point of the liquid a velocity higher than that of sedimentation of the catalyst and originate a mixing action that will keep the mixture uniform. Second, a turbulence regime must be established that will provide appropriate mass transfer for the over-all chemical reaction. These two fundamental conditions affect to a different degree the various steps that constitute the process and are dependent upon the disposition of the equipment, its dimensions, the liquid level, and the rpm. The interrelations among these variables will determine the type of agitation obtained and how it will satisfy the process requirements.

For every set of conditions it was possible to demonstrate the existence of a maximum in the curves of liquid level vs. rate constant. Considering that this effect was observed for two different oils and that it has already been reported in a previous work with a third oil (4), it should be accepted and used for the proper design of hydrogenators.

With dead-end hydrogenation the need for agitation requires internal agitation of the liquid for an appropriate transport of reagents and products to and from the reaction zone; provision of an appropriate suspension of catalyst; and inclusion of hydrogen from the zones where it is available. Internal mixing, at a level above the optimum, will mean that the same power must be distributed in a larger volume, thus diminishing the intensity of agitation. If the liquid level is continuously decreased, agitation efficiency diminishes until the impeller is practically at the liquid surface and becomes inoperative. Similar arguments can be used with respect to the suspension of the catalyst.

It is inferred that a certain impeller position will provide the best conditions for gas-liquid contact and the improvement in the rate of solution of hydrogen. This can be seen upon inspection of the two ways in which hydrogen may enter the oil. First, as to the feed at the bottom of the autoclave, the emerging bubbles are carried away by the flow created by the turbine. They are finely subdivided with the corresponding increase in contact area and retention time. An impeller placed low within the liquid and/or at a high level will favor this manner of gas dissolution. Second, the space above the liquid is occupied by hydrogen that has travelled through the oil without being absorbed. When the impeller approaches the liquid surface, its capacity of hydrogen suction will vary according to the rpm, relative height, and geometrical dimensions.

If the process is controlled by the chemical resistance, the hydrogenation process requires a certain minimum residence time of the elements in order that it may take effect. The hydrogen fed by bubbling is enough, and a maximum is attained in which a low agitation is sufficient. The impeller is located at about 55% of the liquid height. Increasing the liquid level decreases the available hydrogen, and lowering the level augments the agitation per unit volume with a corresponding decrease in the reaction rate.

For fast reaction rates, that is, for conditions of low chemical resistance, there is a need for large amounts of hydrogen and the transport of the gas from above the liquid surface predominates. This causes a change of the relative position of the impeller toward the liquid surface. The turbine is now located at about 75% of the liquid height.

It is also assumed that, at low rpm (900) and intermediate liquid levels (0.9 kg), translational and distributional movements are favored above those of local turbulence. The bubbling hydrogen is absorbed with ease while the upper volume does not contribute in a significant degree to the hydrogen availability.

The experimental results indicate that at 145C the large inflection in the curves of oil weight vs. k' occurs for turbine positions between 65 to 90% of the liquid height. These data indicate that, by comparison with general practice, the turbine is located near the surface.

JAIME WISNIAK
SAVA STEFANOVIC
Department of Chemical Engineering
Universidad Católica de Chile
Santiago, Chile

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A Compact, Convenient Stand and Heater for Soxhlet Extractors

FAT EXTRACTIONS have been carried out in this laboratory by using Soxhlet extractors. Use was made of a hot plate with a support rod, but this was considered unsatisfactory. Consideration was given to the purchase of a six-unit commercial heater, but this type was bulky and heavy. Since we were not using the extractors routinely, it was necessary that the heater be readily movable to and from valuable bench space. Also, the considerable problem of the numerous condenser hoses would still be present, plus the need for the necessary plumbing connections. After consideration of the above problems and our particular needs, the device described herein was designed and constructed. It has been in use in our laboratory for more than a year to our complete satisfaction.

To eliminate the usual jumble of condenser hoses, all the water connections are in the center of the unit, permitting completely free access to each extractor. Cold water is delivered through the center tube, which also serves as the support rod, and the condenser drains are individual tubes ringed around it.

Throughout this description, reference is made to Figure 1 as each part is considered. For clarity, only the basic parts are shown in the drawing. The description for each, plus the photographs of the complete unit in Figure 2, should be sufficient to permit construction.

Item A. This is a laboratory triangular base standard with 6½-in. legs, which has been drilled and tapped in each foot to receive $\frac{1}{4}$-in.-20 leveling screws.

Item B. This is the water delivery tube-support rod. The bottom end is fitted with a solid brass plug made from 1-in. hexagonal bar stock. The lower end of this is turned and threaded $\frac{1}{2}$-in.-13 to match the threads on the base. The upper end is drilled $\frac{1}{2}$-in. in diameter to a depth of $\frac{1}{2}$-in. with intersecting $\frac{5}{16}$-in. holes from two sides. The delivery tube-support rod is a $\frac{1}{2}$-in. OD, $\frac{1}{16}$-in. wall brass tube 20-in. long. This and 2-in. lengths of $\frac{5}{16}$-in. OD copper tubing, which serve as water inlets, are soldered into the plug. The open end of the delivery tube is threaded $\frac{1}{2}$-in. 20.

Item C. This is the plate which supports the heater plate and the six drain tubes and also serves as an anchor point for the electric cord. It a 3 in. in diameter disc of $\frac{1}{4}$ in. thick brass and is shown as viewed from the top in the auxiliary drawing in Figure 1. On a $1\frac{1}{4}$-in. radius there are three No. 27 holes 120° apart for supporting the heater plate. On a $\frac{5}{8}$-in. radius there are six holes spaced as shown for the condenser drain tubes, which are 24-in. lengths of $\frac{5}{16}$-in. copper tubing. In the center a $1\frac{1}{2}$-in. long heavy wall tube of $\frac{1}{2}$-in. ID is soldered in place along with the condenser tubes which extend 4 in. below the plate. The dotted lines show holes drilled and tapped: one for the set screw against the water tube and one for the cord anchor.

Item D. This is the heater plate, which is attached to the support plate by means of three 1-in. standoffs and 6–32 screws. Ceramic standoffs would be preferable for insulation purposes; however metal ones have worked on our unit. The plate itself is an 8-in. in diameter circle of $\frac{1}{4}$-in. thick aluminum with a 2-in. diameter hole in the center. A ring heater (No. R-650 steel sheath or No. R-650M Monel sheath (Watlow Electric Manufacturing Company, 12001 Lackland Road, St. Louis 41, Mo.) is attached to the bottom of the plate by using six $\frac{1}{4}$ in. × 1 in. aluminum bars at 60° intervals. Monel sheaths permit higher wattages to be used.

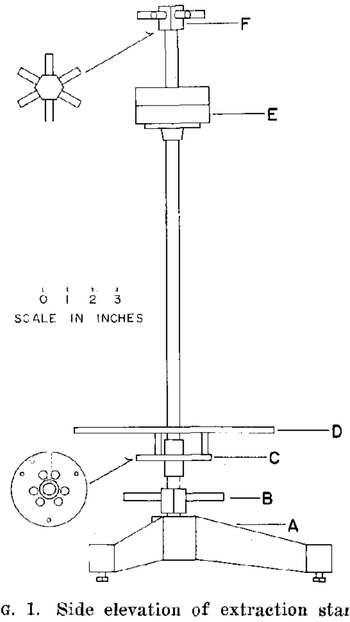

FIG. 1. Side elevation of extraction stand.

Item E. This consists of a series of six spring clamps to hold the condensers. They are attached in two alternating layers on a 3-in. in diameter wooden block, which has a standard lab foot attached. The latter permits it to be fastened to the water tube.

Item F. This is the distributor head for the cold water supply. It is a 1-in. length of 1-in. hexagon brass bar stock, which has been drilled and tapped $\frac{1}{2}$ in.–20 to match the threads on the $\frac{1}{2}$-in. tube. On each face of the bar a $\frac{5}{16}$-in. hole was drilled through to the center, and 1-in. lengths of copper tubing were soldered in place. Teflon tape was used on the threads to give a water-tight seal to the water tube.

During assembly of items B and F, care must be exercised to avoid any restricting of the water passages, which in turn would affect the efficiency of the condensers. Both inlets to B are connected to the water supply if more than three condensers are used.

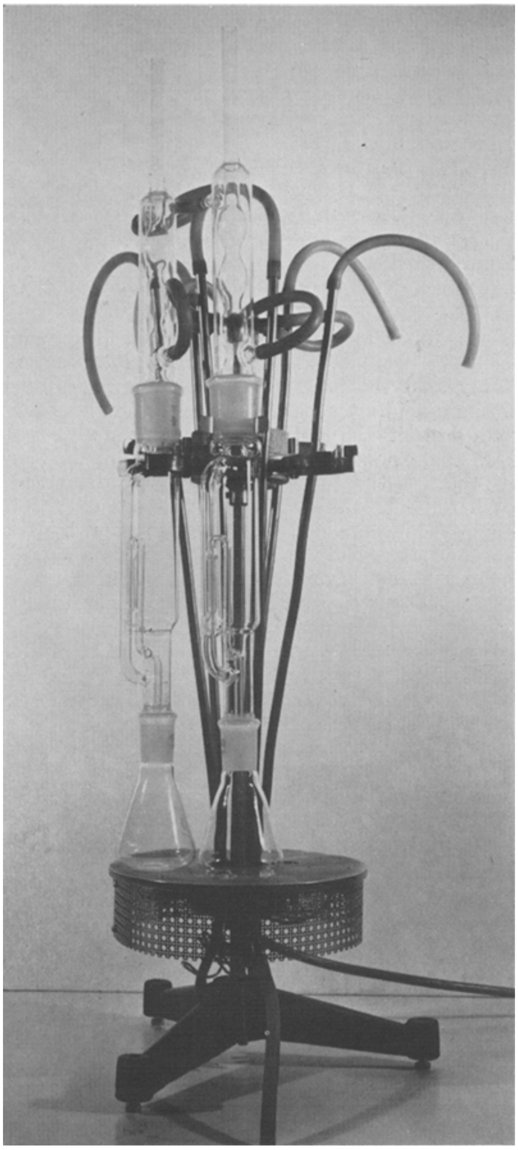

FIG. 2. View of extraction stand with two Soxhlet extractors in place.

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. Benzene	Solvents, ml. Acetic acid	Sterculate found, %
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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