Laboratory deodorization of vegetable oil

The following article describes the glass deodorizing apparatus developed at the U.S. Department of Agriculture's (USDA) Northern Regional Research Center (NRRC) and NRRC's method to prepare oils for taste-panel evaluation. It was written by K.J. Moulton Sr., research chemical engineer in vegetable oil research, USDA, Agricultural Research Service, NRRC, Peoria, Illinois.

Just as the "proof of the pudding" is in the tasting, so is the proof of a good salad or cooking oil. Comparing taste and other qualities in research requires that deodorization, the last chance to improve edible oils, is effective and standardized. Through decades of research at USDA's Northern Regional Research Center (NRRC), we have developed equipment and methods enabling us to reliably produce in the laboratory many quantities of oils to be evaluated by taste panels.

As it would be helpful for researchers and processors in the edible oil field to have standardized equipment and a standardized procedure to prepare oils for taste-panel evaluation, I have prepared this article to describe the multiple-flask glass deodorizer and operating procedures used in our research.

The original design and operation was published in *JAOCS* in 1948 by A. W. Schwab and H. J. Dutton (1). It has been used as an integral component of the NRRC oil evaluation program since then with some design modification and improvements in deodorization and deacidification operational techniques. The complete setup, including an enclosed oil bath, now most closely resembles an isothermic deodorizer proposed by D.S. Sarkadi (2).

Deodorizing in a standardized, reproducible manner is done with up to four different oils in a single-flask or multiple-flask apparatus. Simultaneously deodorizing the oils overcomes variable results due to changes in operating conditions that can alter the effectiveness of deodorization by continuous flow. This uncertainty is particularly troublesome when the goal is to compare different oils or oils processed differently.

Quality control features of the deodorizing apparatus include tubing that allows introduction of citric acid or other antioxidants into the oil without exposing the oil to air. Another feature is precise temperature control of the oils undergoing deodorization. Electric heating coils heat an oil bath quickly, a thermostat controls bath temperature ± 2°C and a cooling coil cools the bath quickly with cold water.

An illustration of a typical application of multiple simultaneous deodorization of vegetable oils with varied composition is described in the article, "Flavor and Oxidative Stability of Continuously Hydrogenated Soybean Oils," published in 1985 (3). In that study, we deodorized several series of three test oils and a control oil in an all-glass four-flask deodorizer for the same deodorization time, temperature, pressure and percentage steam. They included soybean oil; commercial nickel-hydrogenated and winterized soybean oil (4.6% linolenate); nickel-hydrogenated soybean oils (2.7% and 0.4% linolenate, respectively); copper-hydrogenated and winterized soybean oil (0.5% linolenate); and copper-hydrogenated soybean oils (2.4% and 0.4% linolenate, respectively). Each of these oils was treated with citric acid (CA) alone, CA + MS (dimethylpolysiloxane), CA + TBHQ (tertiary butylhydroquinone) and CA + TBHQ + MS. Because of their identical deodorization conditions, these oils could be compared for flavor, initially and after storage for eight days at 60°C, and for oxidative stability by peroxide value and induction period. Similar flavor and oxidative stability studies by Moulton et al. (4) and Mounts et al. (5) described the contribution of this all-glass multiple-flask deodorizer.

Laboratory apparatus

The complete deodorization setup consists of glassware, a vacuum distillation system and a controlled heating bath.

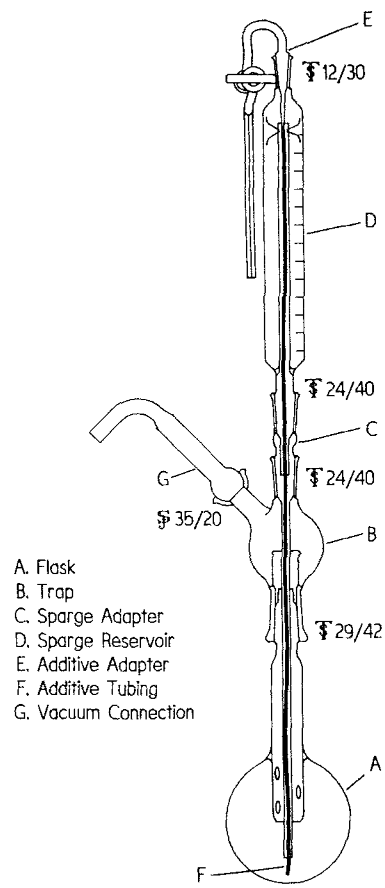


FIG. 1a. Diagram of the components and assembly of a single-flask deodorization apparatus.

Glassware

A single deodorization setup is shown in Figures 1a and 1b; a multiple setup is shown in Figure 2. Common to both setups is a flask (A) (Fig. 1a), a trap (B) to prevent oil entrainment, a sparge reservoir (D) and adapter (C) to supply the stripping steam, and an additive adapter (E), with tubing (F) extending into the oil to administer citric acid and additives into the processed oil.

The flask [Fig. 1a (A)] may be 500-cc, one-liter or three-liter capacity. Protruding knobs fused to the neck of the flask, shown in Figure 3, allow the technician to grasp the flask with a firm grip when disconnecting the flask from the trap. The flask has an internal baffle, as shown in Figures 1a and 3, to prevent splashing the contents into the vacuum system or contaminating the oils in adjacent flasks. A standard taper (s.t.) 29/42 glass male joint joins the flask to the trap. A lip extends beyond the joint (See Fig. 3) and acts as a trap for condensed or splashed oil when assembled. The opening of this lip must be large enough to accommodate a large-bore funnel when filling the flask with oil at the start of a deodorization. Celvacene *Heavy* high-vacuum grease (CVC Products Inc., Rochester, New York), in liberal amount, covers the glass joint. Use of a male joint on the flask assures that the deodorized oil is not con-

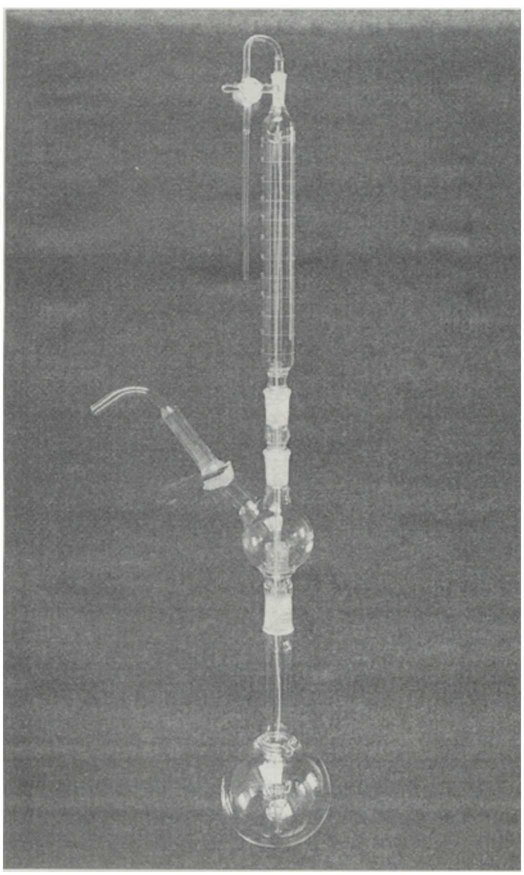


FIG. 1b. Photo of a complete single-flask deodorization apparatus.

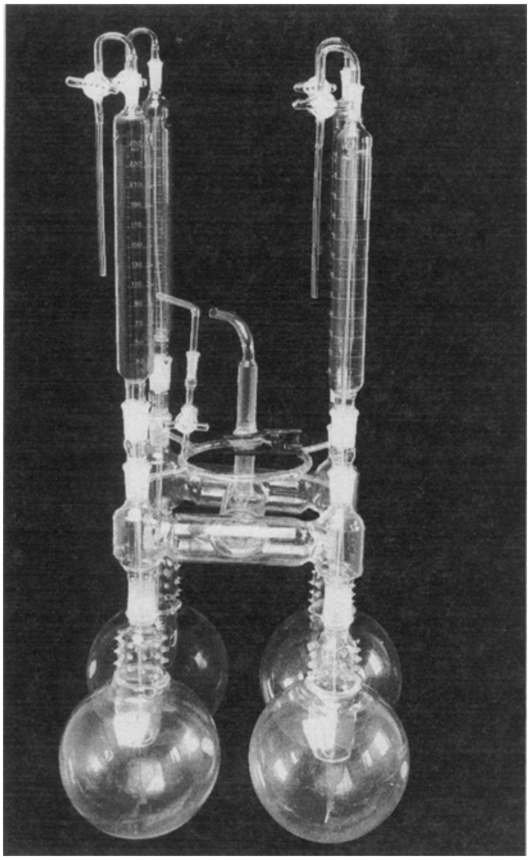


FIG. 2. Photo of complete four-place glass deodorization apparatus.

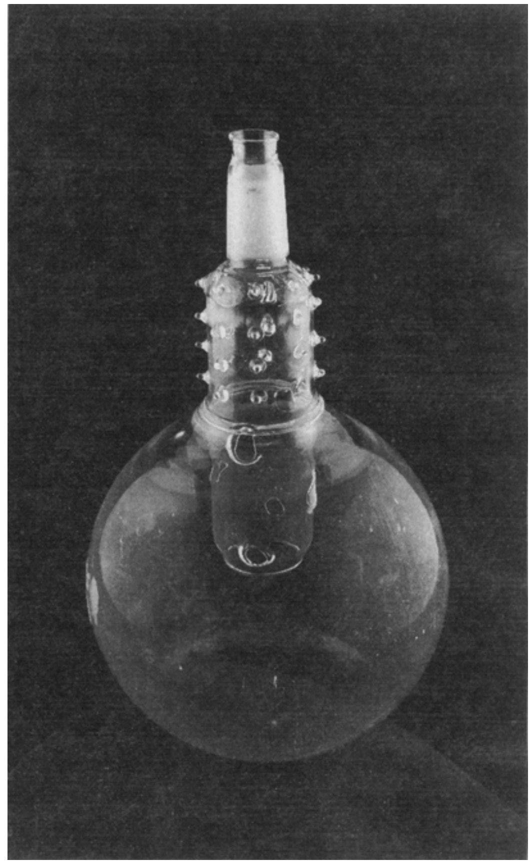


FIG. 3. Photo of a deodorization flask showing the pour spout, protruding knob-gripping surface and the internal baffle.

taminated with vacuum grease and entrained oil when the final deodorized oil is poured from the flask.

Design of the single-flask deodorization trap (B) is shown in Figures 1a and 1b. A spherical joint (s.j.) 35/20 ball-and-socket joint provides an adjustable connection to the vacuum system. Celvacene *Medium* high-vacuum grease is used on this joint and all upper joints and stop cocks of the apparatus. Clamps hold the trap and connecting flask vertically in the heating bath. Design of the "H" manifold trap for multiple flasks is shown in Figure 4. A s.j. 35/20 joint, located on the center of the manifold, connects the trap to the vacuum system. A glass tubing ring, stop cock and s.t. 10/30 female joint connects the manifold to a low pressure nitrogen line, used only to equilibrate the system pressure before discharging the deodorized oil. Lower s.t. 29/42 female joints accommodate flasks of oil to be deodorized. When only two or three oils are to be deodorized, ground glass stoppers block off the upper s.t. 24/40 and lower s.t. 29/42 manifold joints. Lateral outside dimensions (11-inch × 11-inch) of the "H" manifold are such that, when four three-liter flasks are connected, the flasks do not touch each other or the sides of the heating bath.

The sparge reservoir adapter [Fig. 1a (C) and Fig. 5 (a)] is inserted into the s.t. 24/40 single or manifold trap with the bottom reaching to within one inch of the flask bottom when assembled. The sparge reservoir [Fig. 1a (D) and Fig. 5 (b)], filled to the proper level with distilled water for sparging, is inserted into the sparge reservoir adapter. A 10-mm glass tubing, open at the top and ring-sealed at the bottom and centered in the sparge reservoir, provides the route for water vapor to sparge the processing oil.

Additive tubing [Fig. 1a (F)] attached to the additive adapter [Fig. 1a (E) and Fig. 5 (c)] is fed through the open 10-mm glass tubing and extends into the processed oil. Size 14 Teflon thin-wall spaghetti tubing (Chemplast Inc., Wayne, New Jersey) was found to be a satisfactory tubing.

A completely assembled four-place deodorization setup is shown in Figure 2. The manifold trap rests securely on a horizontal platform (shown in Fig. 6) at the proper height above the heating bath medium. Care must be taken to ensure that the heating bath stirrer does not strike any of the submerged flasks during operation.

Vacuum distillate system

The vacuum distillate system consists of a laboratory vacuum pump, McLeod gauge, manometers and a cold

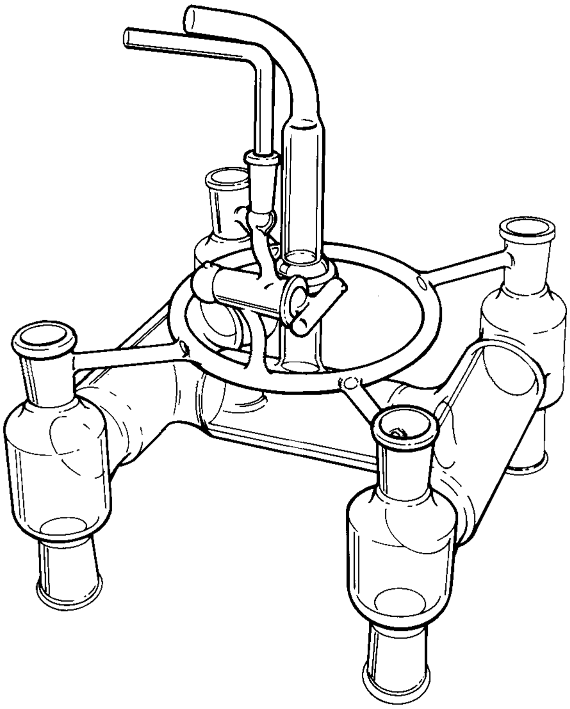
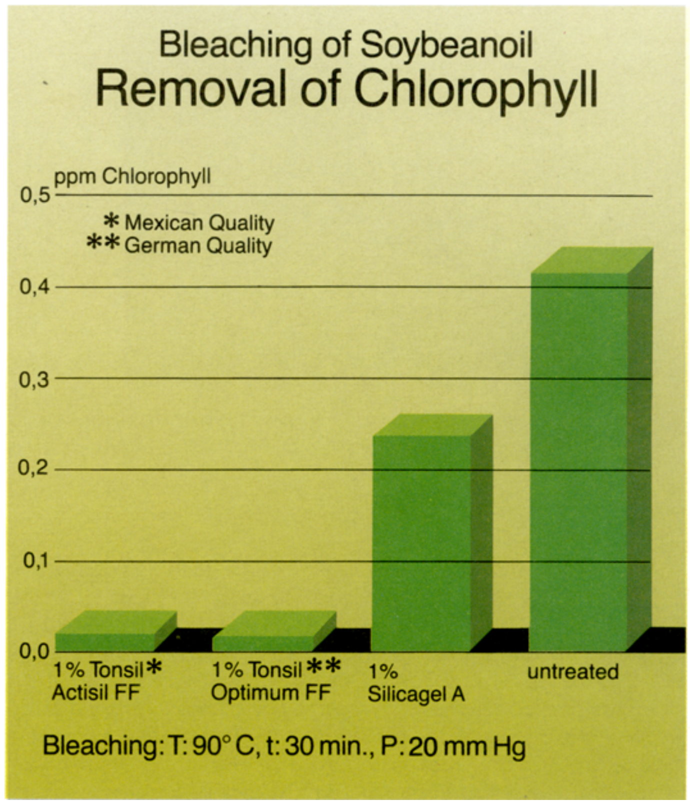


FIG. 4. Diagram of the "H" manifold trap used for multiflask deodorization.

finger trap connected to the vacuum connection [Fig. 1a (G)] on the deodorizer trap [Fig. 1a (B) or Fig. 4]. Dry ice/acetone in the cold finger condenses the fatty acids, volatiles and stripping steam. A McLeod gauge measures the instantaneous pressure in the deodorizer system. Closed-end manometers, connected in the vacuum system, are used when breaking the system vacuum with nitrogen to indicate when the system reaches atmospheric pressure.

Controlled heating bath

With the flasks submerged in the heating bath, the oils to be deodorized are heated to deodorization with an electrically heated, thermostatically controlled heating bath (Fig. 6). The bath used (fabricated by Precision Scientific, Chicago, Illinois), has a 17.5-inch × 17.5-inch × 15-inch stainless steel tank that contains about 16 gallons (112 pounds) of commercial cooking oil (5.5-inch outage when cold). Heating is by three 1-kw submergible heating elements and one 1-kw Calrod heating element. Each element has its own switch and is controlled by a thermostat capable of holding the oil bath temperature to within ± 2°C (Fig. 7). With all elements on, the bath temperature can be raised from room temperature to 220°C in two hours. A stirrer in the rear



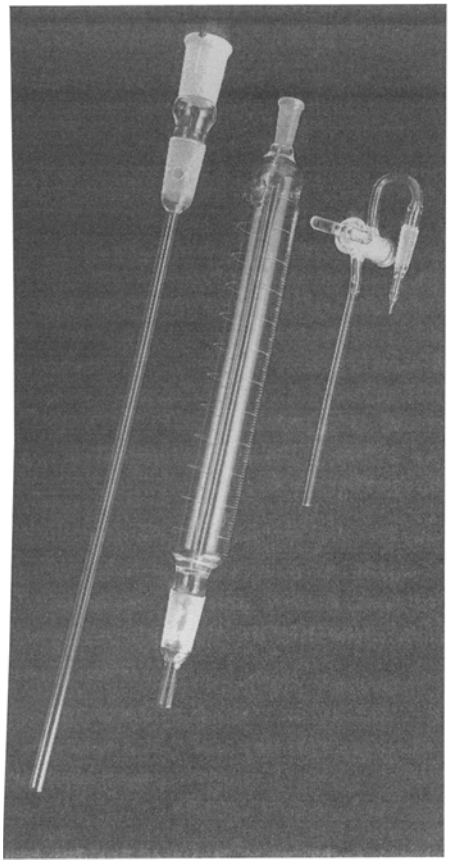


FIG. 5. Photo of the sparging and additive injection components: (a) sparge adapter; (b) sparge reservoir; (c) additive adapter.

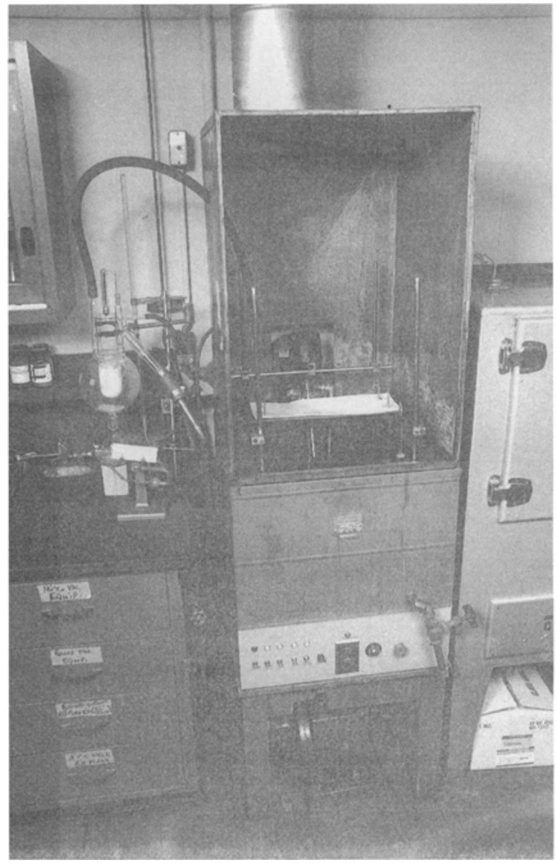


FIG. 6. Photo showing the controlled heating bath, platform to support multiple-trap manifold and vacuum-trap system.

of the bath circulates the bath oil to maintain a constant bath temperature. Cold water through a coil of three-eighths-inch stainless steel tubing cools the bath oil from a deodorization temperature of 220°C to 100°C (for addition of citric acid) in 35 minutes. A wire grid, four inches above the bath bottom, separates the heating elements and cooling coil from the flasks. The cabinet area above the oil bath is completely enclosed. A removable glass and metal front allows access to assemble and disassemble the deodorizer and visual monitoring of the deodorization. Heat and fumes are drawn off through an exhaust line in the top of the cabinet. A horizontal movable platform, mounted four inches above the stainless steel tank, supports the glass trap manifold. A thermometer in the heating oil bath indicates the oil temperature.

Deodorization technique

The following procedure is recommended for deodorizing oils in the apparatus just described:

Step 1—Weigh oil to be deodorized into the flask or flasks [Fig. 1a (A)]. About 1200 gm is the maximum amount of oil for a three-liter flask; 400 gm for a one-liter flask; 200 gm for a 500-ml flask.

Step 2—Add distilled water to the sparge reservoir (D) up to the 150 cc level. Because of the volume occupied by the internal tube, the graduations on the reservoir may not be accurate. If accurate measurement of water used is needed, each sparge reservoir should be recalibrated.

Step 3—Apply Celvacene vacuum grease to all ground glass joints.

Step 4—For *single deodorization,* assemble the single deodorization apparatus as in Figure 1a and clamp into the heating bath. For *multiple deodorizations,* fasten the multiple-trap manifold (Fig. 4) on the platform above the heating bath (Fig. 6) and assemble the remaining glassware as follows:

- Connect the required two, three or four flasks to the "H" trap manifold. Be sure to identify and record the contents of each flask. Verify identification again on discharge of the deodorized oil.
- Connect the sparge adapters [Fig. 5 (a)]. Rotate the hole in the s.t. 24/40 90° from the corresponding hole in the manifold joint.
- Connect the sparge reservoirs [Fig. 5 (b)].
- Connect the additive adapters [Fig. 5 (c)] with

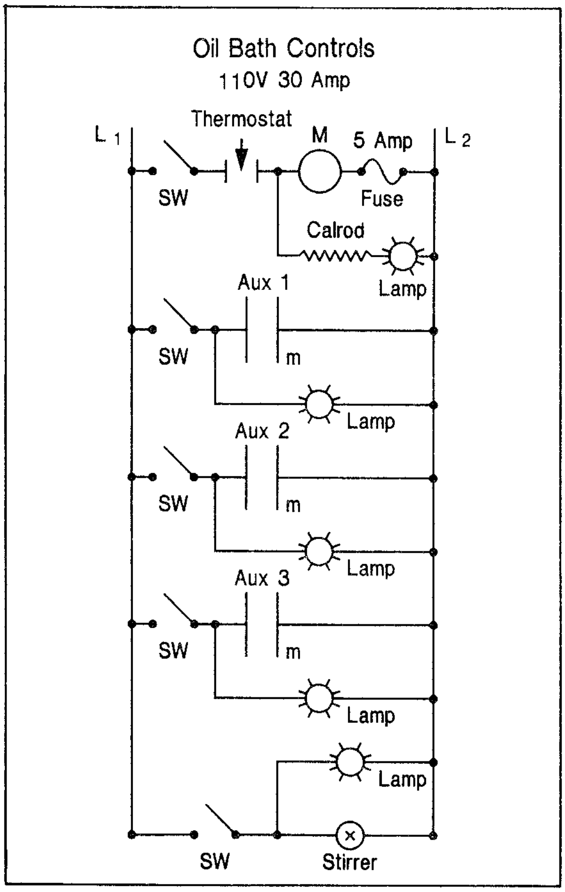


FIG. 7. Schematic of controls for the controlled heating bath. Relay (M) operates Calrod, Aux 1, Aux 2 and Aux 3 heating elements.

Teflon tubing attached to the sparge adapters. Feed the Teflon tubing through the internal glass tube.

• Clamp the vacuum connection [Fig. 1a (G)] to the "H" manifold.

• Connect a nitrogen line to the stop cock on the "H" manifold tubing ring (See Fig. 4).

Step 5—Fill the cold finger trap with dry ice and acetone.

Step 6—Turn on the exhaust fan.

Step 7—Turn on the vacuum pump to evacuate the apparatus.

Step 8—Turn on the stirrer in the heating bath oil.

Step 9—Turn on power to all heating elements (Calrod, Aux 1, Aux 2, Aux 3). Be sure cooling water valve is closed.

Step 10—Keep log of time, temperature (°C), pressure (mm), sparge water level (cc) and operation comments.

Step 11—When the desired deodorization temperature is reached, turn off Aux 1 and Aux 2 heaters, adjust the thermostat to control the bath at the deodorization temperature and record the time for starting the deodorization.

Step 12—After the required deodorization time (usually three hours at 220°C or one hour at 260°C), turn off all heaters, reset the thermostat to a low temperature and start cooling the bath with cold water through the cooling coil.

Step 13—If the deodorized product requires the addition of stabilizers (antioxidants and/or citric acid), turn off the cooling water when the oil bath reaches 100°C.

Step 14—Draw diluted solutions of stabilizers or citric acid into the deodorized oil in each flask through the additive adapter. (A 13 × 100 mm glass culture tube is suitable to contain the solutions.) Two rinses of diluent ensure that all stabilizers or citric acid have been added to the oil.

Step 15—Hold the bath temperature at 100°C for 10 minutes to assure thorough mixing of the additives and flashing off the solvent, then turn on the cooling water again to cool the bath to 70-80°C.

Step 16—Turn off the vacuum pump.

Step 17—Rotate the sparge adapter [Fig. 4 (a)] so the hole in the s.t. 24/40 joint is aligned with the hole in the "H" manifold joint.

Step 18—Open the stop cock in the "H" manifold tubing ring and slowly admit low-pressure nitrogen into the deodorizer flask until the manometer indicates atmospheric pressure is reached. Note: For a single-flask deodorization, nitrogen can be slowly admitted to the system through the additive tubing or through a tee in the vacuum connection.

Step 19—Disassemble the glassware in reverse order of assembling.

Step 20—Pour deodorized oil from the flask into a labeled bottle through a gauze-faced filter disk (Filter Fabrics Inc., Goshen, Indiana) and immediately cover oil with nitrogen before capping. Be sure product is labeled properly, corresponding to oil in the flask.

Step 21—Percentage water used for sparging can be calculated from the drop in level in the sparge reservoir (adjusted for the internal tube volume) or from the volume of melted cold finger contents, assuming an equal amount of water was delivered to each flask.

Deodorization should not be considered complete until all the glassware is scrupulously cleaned and ready for the next deodorization. The flask, sparge adapter and single trap can be adequately cleaned by soaking in a hot, strongly caustic and detergent bath followed by thorough washing in detergent, with at least three tap water rinses and two distilled water rinses. The multiple-manifold trap, sparge reservoir and additive adapter may not be cleaned adequately by washing and the possibility exists of retaining oil, caustic or detergent residue, which will affect subsequent deodorizations. These glass components can be cleaned by heating to 600°C in an annealing oven. At this temperature, all oil and stop cock grease are burned off, leaving the glassware clean.

ACKNOWLEDGMENT

Francis J. Castle was inspirational for the original design and the modifications of the glassware, as well as the glass-blowing fabrication of all glass parts of the apparatus.

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