The Flavor Problem of Soybean Oil. III. A Four-Sample, Glass Laboratory Deodorizer[1]

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THE desirability of having a number of samples deodorized simultaneously under identical conditions of time, temperature, pressure, and steam flow prompted the design of a multiple laboratory deodorizer. Because in studies on the flavor problem of soybean oil it has frequently been necessary to compare four samples (1), a four-unit deodorizer was constructed. Bailey and Feuge (2) have described a single all-glass laboratory deodorizer and Sanders (3) has described a multiple stainless-steel type. Features from both of these installations have been combined in the current design along with certain other improvements. Because of the deleterious effect of metals upon the stability of edible oils (4) and because of the advantage of visibility, an all-glass assembly was favored over metal types. The potential hazard of breakage of glass equipment during the deodorization process is minimized by conducting the entire operation in a hood provided with a safety-glass window. An oil bath serves both for heating and cooling the flasks. Provision is made for the measurement and control of the rate of steam generation. The deodorization flasks are equipped with baffle plates to prevent the bumping over and mixing of the samples.

Apparatus. One of the important features of the apparatus design (Fig. 2, 3) is the steam generator which permits the measurement of rate of steam flow. It is constructed from a 250-ml. graduated cylinder. A 10/30 standard taper ground-glass joint is sealed to its top and a piece of 7-mm. tubing, ring sealed into the bottom of the graduate, extends nearly to the top of the graduate. The lower end of the 7-mm. tubing ends in an inner 24/40 standard taper ground-glass joint. Three-mm. capillary tubing is sealed to the bottom of this ground-glass joint and extends to the bottom of the deodorizer flask. Water to be vaporized is placed in the graduated cylinder. The rate of steam generation is adjusted by controlling the temperature of the water. If a different rate is desired for each of the four samples, the rate can be regulated by winding a few strands of Nichrome wire around each cylinder and connecting the wires to autotransformers. However, for most deodorizations the same rate of steam flow is desired in each flask. This rate is adjusted by controlling the air temperature of the hood in which the generators stand (Fig. 1).

Another significant feature of the deodorizer is the construction of the flasks containing the oil samples (Fig. 2). These flasks are designed with perforated baffles which effectively prevent the bumping over of oil from one flask to another. The baffles are ring sealed into 3-liter flasks at the base of the neck. Samples of oil up to 1,000 ml. have been deodorized in

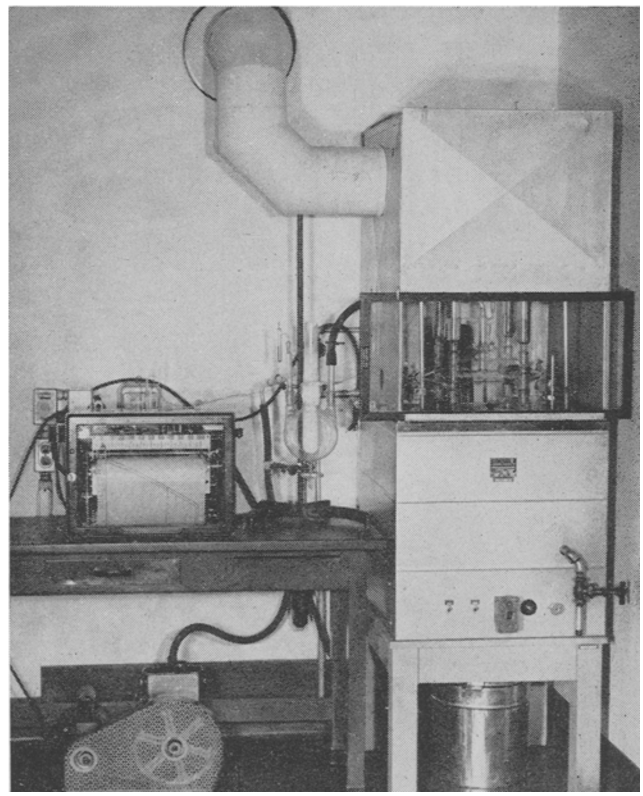

FIG. 1. Complete assembly with hood, oil bath, condenser, and temperature recorder.

these flasks without difficulty from bumping. The four flasks are connected to an H-shaped exhaust manifold by means of 24/40 standard taper joints. A 50-ml. bulb is blown in the manifold at the intersection of each arm of the cross member. These traps serve to collect condensate. The 5-mm. glass tubing, which is sealed to the 24/40 standard taper joints on the exhaust manifold, permits the introduction of an inert gas at the completion of the deodorization. Before sealing on these tubes, a hole was bored into the outer ground-glass joint. A corresponding hole was bored in the inner ground-glass joint of each steam generator. By rotation of the steam generators the two holes may be aligned and the inert gas can enter any one or all of the steam generators (Fig. 3). "Breaking the vacuum" at this point prevents the oil from being drawn back into the steam generator.

The manifold, flasks, and steam generator system are evacuated through a spherical ground-glass joint carrying a manometer. Rubber tubing connects this joint to a cold-finger condenser which is cooled with a solid carbon dioxide-acetone mixture. A second manometer is attached between the condenser and the vacuum pump. The two manometers indicate the pressure gradient through the condenser.

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The manifold with steam generators and flasks attached is mounted on a frame which may be placed in an electrically-heated and thermostatically-controlled oil bath (Fig. 1). It can be brought to an operating temperature of 210°C. in 120 minutes. A cooling coil consisting of 40 feet of ¼-inch stainless-steel tubing is located in the bottom of the oil bath. The bath temperature can be dropped from 210° to 38° in 100 minutes by running tap water (17°) through this coil. A complete record of temperature changes during the course of the deodorization is obtained from a multiple recording potentiometer, with thermocouples in the oil bath, deodorization flask well, and steam generator.

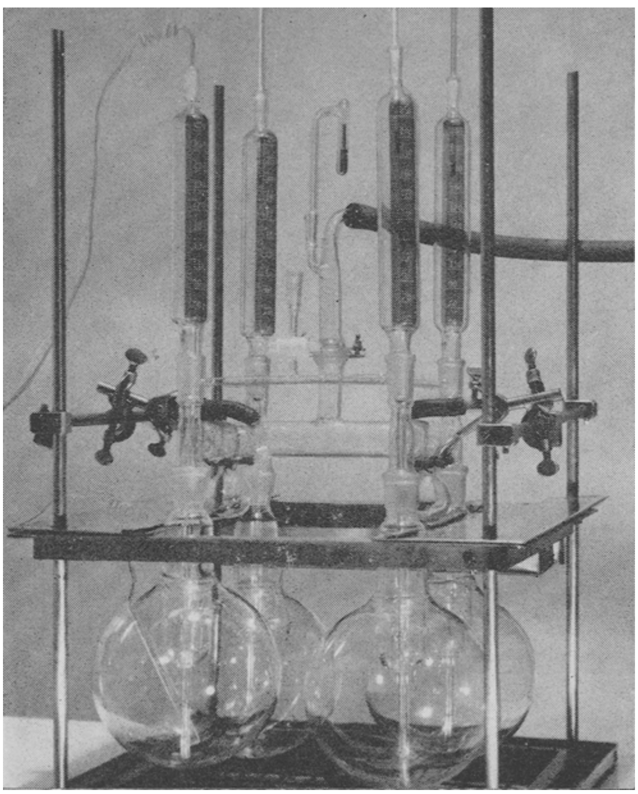

FIG. 2. Deodorizer assembly mounted on frame.

It should be noted that the present design permits the use of glass deodorizers with a degree of safety previously found only with metal deodorizers. When the deodorization is performed in glass on the laboratory table the vigorous ebullition of steam through oil at 210°C. under vacuum presents a potential hazard. Even more dangerous is the removal of the heater at the completion of the deodorization and the introduction of cooling water on the hot surface of the flask. In the present design the whole operation is conducted in a hood provided with a safety-glass window. The oil in the bath serves both to heat and to cool the flasks in a gradual manner.

Operation. The operation of the deodorizer proceeds as follows: First, the apparatus is charged with oil, water, solid carbon dioxide, and acetone and the vacuum pump is started. As the oil bath reaches the operating temperature it raises the air temperature of the hood. This temperature controls the rate of steam generation and is adjusted by a damper controlling the rate of air flow through the hood. Since the oil bath is thermostatically controlled, little attention other than filling the solid carbon dioxide trap is required until the deodorization is completed. At that time tap water is passed through the coils and the bath is cooled to 38° to 40°C. The vacuum pump is then stopped and the stopcock leading to the inert gas (nitrogen less than 0.002% 0_2) is slowly opened to "break" the vacuum.

The time of deodorization is a function of the temperature, pressure, and rate of steam flow (5). With the apparatus described, a temperature of 210°C., a pressure of approximately 1.0 mm. Hg, and a steam rate of approximately 1% of the sample weight per hour are used. Samples of a refined, bleached soybean oil were deodorized under these conditions for 0, ½, 1, 2, and 3 hours. They were submitted subsequently for organoleptic evaluation (1) and the results are included in Table 1. No significant dif-

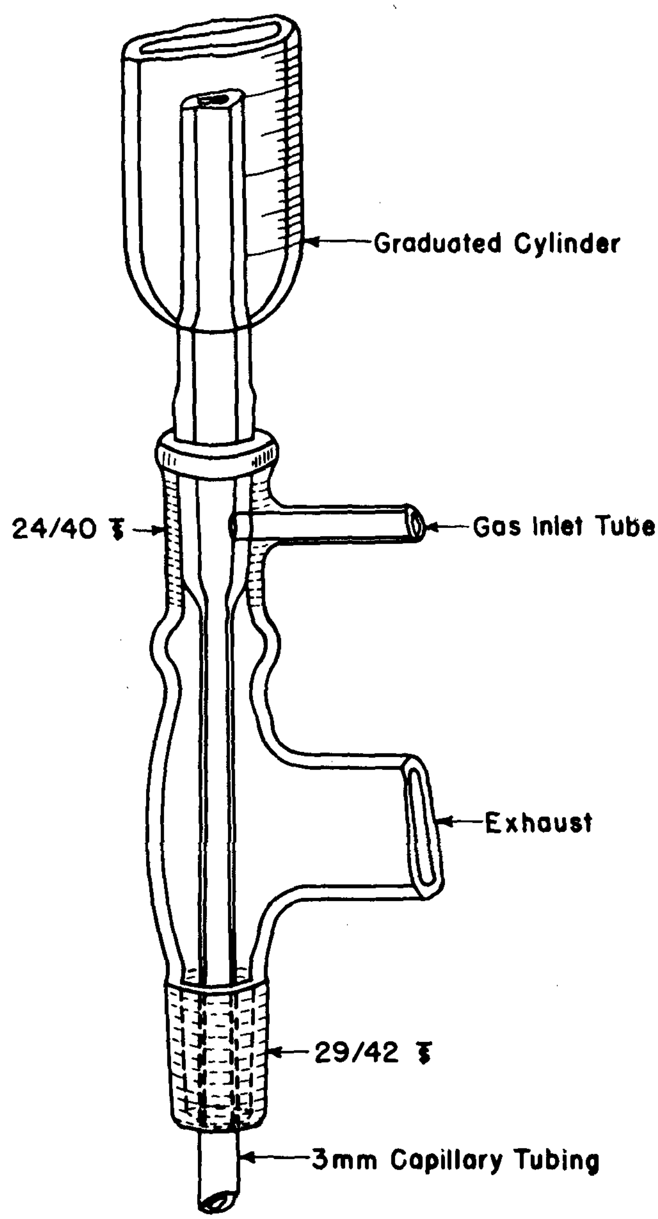

FIG. 3. Diagram showing details of construction of manifold and flask including steam generator, and method of introducing inert gas.

TABLE 1

Flavor Scores and Oxidative Stabilities of Deodorized Oils as a Function of Deodorization Time

Deodorization time at 210°C.					Significance	Probability
3 hours	2 hours	1 hour	½ hour	0[a] hour		
0 time						
8.8 (0.44)[b]	8.3 (0.33)	8.4 (0.39)	8.6 (0.22)	8.1 (0.34)	+[c]	0.26 to 0.74
Storage—3 days, 60°C.						
5.4 (1.0)			5.1 (1.2)		+	0.70
5.7 (1.3)				5.3 (1.5)	+	.49
5.3 (1.3)		4.6 (1.5)			+	.20
Peroxide values (A.O.M. conditions, 8 hours)						
18.2	14.6	20.6	15.0	14.6		

[a] Heated to 210°C. and cooled.

[b] Values in parentheses are peroxide values at the time of tasting.

[c] + No significant difference. (For definition of statistical terms see ref. 1)

ferences in flavor scores were indicated between the 5 samples by the taste panel, either immediately after deodorization or after 3 days' storage at 60°. From these results it is concluded that a satisfactory deodorization is obtained in this apparatus in less than 1 hour. The deodorization time indicated in the table is the time the samples were maintained at 210°. Actually deodorization occurs during the time the temperature is being raised to 210° and lowered to 40°.

TABLE 2

Constancy of Flavor Scores and of Oxidative Stabilities of Oils Processed in the Four-flask Deodorizer

Flask 1	Flask 2	Flask 3	Flask 4	Significance	Probability
0 time					
7.9 (0.74)[a]	8.2 (0.81)	7.8 (0.84)	8.3 (0.69)	+[b]	0.3 to 0.9
Storage—4 days, 60°C.					
5.1 (9.1)	5.5 (8.5)			+	0.57
5.7 (6.5)		5.1 (12.5)		+	.42
5.0 (8.2)			4.8 (12.2)	+	.75
	5.4 (8.8)	6.0 (9.7)		+	.47
	5.6 (8.0)		6.1 (8.0)	+	.11
		5.5 (6.5)	5.9 (5.7)	+	.61
Peroxide values (A.O.M. conditions 8 hours)					
52.7	54.5	54.0	53.5		

[a] Values in parentheses are peroxide values at the time of tasting.

[b] + No significant difference. (For definition of statistical terms see ref. 1)

A check of the constancy of oils produced in the four flasks was made by simultaneously deodorizing four samples of the same oil. These samples were submitted for organoleptic evaluation (1) and the results are listed in Table 2 along with the peroxide values after 8 hours in the Swift stability apparatus. Both chemical and organoleptic evaluations show no significant difference among the samples.

Summary

A glass laboratory deodorizer has been described which permits the simultaneous deodorization of four samples under nearly identical conditions of time, temperature, pressure and rate of steam flow. The design includes provision for measuring and controlling the steam flow, for preventing bumping, for "breaking" the vacuum, and for heating and cooling the deodorizer. Successful deodorizations have been made in relatively short periods of time. Upon deodorization of the same alkali-refined bleached oil in each of the four flasks no difference has been found either in the initial quality or in the stability of the samples.

Acknowledgment

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