

but the cleaned glasses were deleterious on foam retention of beer.

New work on hand soaps containing hexachlorophene antiseptics confirmed their effectiveness for maintaining a low count of resident bacteria on the skin (Browers—*Proc. Chem. Specialties Mfrs. Assoc. June 1950*, 90; Cade—*Ibid.* 92; Blank & Coolidge—*J. Invest. Dermatol.* 15, 257), and demonstrated that these antiseptics did not delay wound healing (Best *et al.*—*Arch. Surg.* 62, 895). Quaternary ammonium compounds as skin cleansers and antiseptics formed a film over the skin under which bacteria were retained (Blank & Coolidge—*J. Invest. Dermatol.* 15, 249).

Sewage processing engineers were concerned with the increased use of synthetic detergents because it may affect conventional methods of treating sewage. Sperry (*Sewage & Ind. Waste* 23, 1469) observed that the following effects were produced by the synthetics: they reduced the amount of suspended solids deposited, thus diminishing removal from tanks; gas to

be expected was diminished; grease tended to emulsify; and excessive frothing that occurred might be an annoying nuisance. Degens *et al.* (*Inst. Sewage Purif. J. and Proc.* 1950, 63) found that concentrations of five parts per million of four common synthetic detergents were lethal to tadpoles, sticklebacks, and *Daphnia*, whereas several other fauna and aquatic plants were unaffected. Some of the detergents decomposed under the conditions of the "biological oxygen demand" test. Waddams' (*Ibid.* 32) work on the problem included laboratory bactericidal tests and the reconciliation of results of these with washing practices and sewage treatments. He summarized the work as follows: The presence of 100 ppm. of synthetic detergents in sewage would be most unlikely; at 200 ppm. it did not affect settling of sewage; up to 500 ppm. it did not have bactericidal nor bacteriostatic action on bacteria common to crude sewage; it affected protozoan, *Euplotus patella*, at 100 ppm. only after 60 hours; it had no effect on sludge digestion below 750 ppm.; and flocculation of sewage by alum was not affected.

Rapid Test for Trichloroethylene in Vegetable Oils. Modified Beilstein Test

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A NEW modification of the Beilstein test is recommended for the semiquantitative determination of small amounts of chlorinated solvents in vegetable oils. The test will detect 0.01% trichloroethylene in crude vegetable oils. The technique should also prove valuable in increasing the sensitivity of other applications of the classical copper wire test.

Trichloroethylene is gaining widespread use as a solvent for the extraction of vegetable oils because of elimination of the fire hazard always present when using inflammable solvents. It has the disadvantage of interfering with hydrogenation by reducing the catalyst activity. Amounts as low as 0.005% have been shown to be definitely harmful, and concentrations above 0.03% make the oil very difficult to hydrogenate. A practical maximum limit is felt to be 0.01%.¹

Only two published methods for trichloroethylene in vegetable oil have been found. Arnold and Hollowell (1) report a sensitivity of 0.02% when using a modification of Fujiwara's method for chloroform. Two drops of the oil sample are added to a hot mixture of sodium hydroxide and pyridine. A red color shows the presence of halides and may be compared to standards. This test is neither as rapid nor as sensitive as the proposed flame test. Eisdorfer and Mehlenbacher (2) collect the solvent by distillation and measure the red color with a spectrophotometer to get quantitative results. The determination requires two hours.

The classical qualitative test for halogen is the Beilstein test which involves heating over a burner a copper wire dipped in the sample. A green flame shows the presence of halogen. Two modifications of the test have been suggested for the determination of volatile organic halides. Ruigh (3) tested for volatile halides by adding a sample dropwise to a warmed bottle, through which the gas supply to the burner passed, and observed the green color near a copper screen in the flame. Stenger, Shrader, and Beshgetoor (4) used a copper strip in the flame to detect methyl bromide vapors in the air.

¹After this note was submitted for publication, Norris, F. A., Mattil, K. F., and Lehmann, W. J., *J. Amer. Oil Chem. Soc.*, 29, 28-32 (1952), published results showing that concentrations of trichloroethylene greater than 0.003% retard hydrogenation and that refining and bleaching processes greatly reduce the trichloroethylene content.

Neither of these procedures appeared applicable to crude vegetable oils, and a number of means of uniting the sample and hot copper in a flame were tried. The method adopted as most sensitive employs a 40-mesh copper gauze² cut in a strip one-fourth inch by two inches. The oil is applied with a glass stirring rod by spreading along the center of the length of the strip, keeping the edges free of oil. The strip is grasped by crucible tongs at one end and held horizontally in the colorless flame of a burner. A green flame shows the presence of halide.

To determine the sensitivity of the test, known mixtures of trichloroethylene and "expeller crude" soybean oil were made and tested. Results are summarized in the table below:

Trichloroethylene in Oil, Wt. %	Result of flame test
0.1	Bright green flame—persists during burning of oil.
0.05	Green flame—usually disappears before the oil ignites.
0.01	Faint green flash as gauze is put in flame.
0.005	Green can rarely be detected.

The test must, of course, be carried out in air free of halides. Hydrochloric acid fumes, in concentrations which cannot be detected by odor, give a bright green flame with the bare copper.

If the copper strip is dipped in the oil sample, sensitivity is reduced and a 0.05% sample shows the same flame as a 0.01% mixture tested in the suggested manner. The solvent present in unknown samples may best be estimated by dilution with expeller or hydraulic crude soybean oil until the test is comparable to a 0.01% known mixture.

Each analyst using the test should prepare his own calibration by testing known mixtures. Independent tests in different laboratories have verified that 0.01% trichloroethylene is a practical limit to set for the test.

²Central Scientific Company, No. 19956-D is satisfactory.

REFERENCES

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