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The Determination of Small Amounts of Trichloroethylene

LIONEL K. ARNOLD AND E. GRAHAM HOLLOWELL

In research studies on the extraction of soybean oil by trichloroethylene it became necessary to determine small amounts of the solvent in the oil. For amounts above 10 percent trichloroethylene the simplest and best method for control purposes is by means of a specific gravity determination. Measamer (3) has shown that the trichloroethylene may be steam-distilled from the oil and weighed or measured. Three percent trichloroethylene in soybean oil may be determined with a maximum error of 10 percent. Greater accuracy should be possible in solutions containing more of the solvent. For amounts above 10 percent the method is more time consuming and no more accurate than the specific gravity method.

Amounts of trichloroethylene between 0.1 and 1.0 percent in soybean oil may be determined spectrographically with an error of 7 percent according to Yates (4). A copper wire is dipped in the oil and held in the flame, the amount of solvent being indicated by the duration of the copper chloride bands between wave lengths of 4259 and 4518 angstrom units. This method has the disadvantage that, because of differences in color perception, each operator must calibrate his own time-percent trichloroethylene relation.

A method for determining very small amounts of chloroform is described by Fujiwara (1). He added pyridine to a 10 percent solution of either sodium hydroxide or potassium hydroxide and heated them to boiling. The two liquids remained as separate layers after the heating. When a solution containing chloroform was added to the mixture the pyridine layer turned a blue-red.

Fujiwara found that this test is more sensitive than other tests used for this purpose such as insonitrile, resorcin, and naphthol tests. He was able to detect one part of chloroform in 1,000,000 parts of water, in 500,000 parts of ether, or in 300,000 parts of alcohol. He reports that the test may be used for other halogen compounds but gives no data on other than chloroform. Gilchrist (2) in this laboratory repeated Fujiwara procedure with trichloroethylene. The pyridine layer turned a deep cherry red. He found that the intensity of the color varied with the amount of trichloroethylene present thus making it possible to make it a quantitative as well as a qualitative test. Work by Gilchrist and later by the authors developed a method now used for routine determinations of trichloroethylene in soybean oil, soybean meal, and water.

The method for soybean oil is as follows:

To $1\frac{1}{2}$ ml. of 10 percent sodium hydroxide in a test tube add 1ml of pyridine and heat to boiling. Add 2 drops of the oil. Allow to stand one minute for the color to develop and compare depth of color with color standards. Originally the color standards were made by adding

oil containing known amounts of trichloroethylene to pyridine-sodium hydroxide solutions prepared as in the test. The color of these standards faded rapidly so permanent standards were devised. These were made by mixing a water solution of sodium dichromate and cobaltous chloride made acids with hydrochloric acid. A series of these standards from 0.02 to 2.0 percent are used.

While this test is the most sensitive test found for the detection of traces of trichloroethylene in soybean oil, it is not as sensitive as is the test for chloroform in water. The smallest amount of trichloroethylene in oil that may be detected is less than 0.02 percent, the exact amount being dependent upon the color of the oil, the lighting condition, and the color accuracy of the operators eyes. Tests with both chloroform and trichloroethylene in oil indicate that it requires only one-eighth as much of the former as the latter to give a certain intensity of coloration. In methanol where there is no color to interfere the ratio is one to five. Amounts of trichloroethylene in methanol as low as 5 parts in 1,000,000 have been detected using only 3 drops of the methanol solution. When testing a sample of soybean oil it is helpful to increase the amount of oil added to 3 or 4 drops if there is question as to the presence of any trichloroethylene. Any additional oil is of doubtful value as the color of the oil tends to interfere more when larger amounts are present. For amounts of the solvent above two percent the sample should either be diluted with solvent-free oil or only one drop used in the test. In the latter case for accurate results new standards should be prepared based on one drop of oil added.

The determination of traces of trichloroethylene in soybean meal is made by the following modification of the procedure for the solvent in oil. Ten grams of the meal are weighed rapidly and added to 125 ml. flask containing 40 ml. of hexane ("Skellysolve B") and the flask stoppered and allowed to stand for a few minutes. Four drops of the hexane are added to the hot pyridine-sodium hydroxide mixture, and the depth of color compared with standards prepared for this test.

The method has also been applied to the determination of trichloroethylene in water, using 2 drops of the water and comparing with suitable standards.

The three tests have been used successfully in routine control work by the authors in checking operations in a soybean oil extraction plant using trichloroethylene as a solvent. The test for trichloroethylene in the oil is carried out in a routine manner by the plant workmen as a regular check on the oil quality.

Several precautions should be observed in using these tests. Pyridine is sufficiently toxic that is advisable to carry out the operations in a hood. Only a pure grade of pyridine can be depended upon not to give the red coloration before adding the solution of trichloroethylene. Some bottles of the so-called "practical" pyridine have been found satisfactory; some have not. Care should be taken not to con-

taminate the pyridine. This may occur if the bottle is left unstoppered in a room containing small amounts of trichloroethylene vapor in the air.

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