

The Determination of Fish Oil in Vegetable Oils

The following test is a modification and extension to quantitative results of the official qualitative test No. 26,084 of the Association of Official Analytic Chemists (AOAC) for detection of fish and marine animal oils in the presence of vegetable oils and in the absence of metallic salts.

It originated in the course of examining hundreds of samples, often 25 to 30 at a time, toward the end of a long period of bulk shipments of oils for foreign relief when these came under suspicion of adulteration.

Normally soyabean oil would not be thought of as being admixed with fish oil since it is the cheapest of the edible oils and because the presence of fish oil should be readily detected by taste and odor on heating. The fish oil however was well refined, bleached and deodorized and passed the contractual specifications of conventional analysis. The first clear-cut clue arose in the case of sperm oil addition to soya oil from the abnormal amount of unsaponifiable and its waxiness, whereupon the AOAC test was applied. From then on all exports were tested but handling so many tests continuously, with strong bromine, often proved too hazardous even under the best of precautions. Later the contents of over a hundred tanks at Bayonne had to be identified for the benefit of creditors of a bankrupt organization. The bromine had to be diluted considerably and further improvements introduced to make the test safe, quick and easy.

The test is carried out as follows: Dilute bromine with 3 to 4 vol of glacial acetic acid, roughly estimated by the eye. Pour some into a burette. Dissolve 30 drops of the oil to be tested in 8 ml chloroform; add 10 ml Wijs iodine solution. Let stand for 10 min; then add dropwise the diluted bromine solution from the burette at a fairly steady rate with constant stirring using a 50 ml beaker rather than a test tube, passing a point in the titration where the solution appears to bleach out, whereupon a slight excess of

bromine is added. Stir and immediately pour the solution into a flat-bottomed test tube such as the kind used for Lovibond color reading. Let stand 1 hr. Read the height of the precipitant by measuring with a millimeter rule and compare this with known mixtures of fish oil. Since the amount of the insoluble bromides is directly proportional to the content of fish oil, only one known blank needs to be run and measured, and after that none needs to be made for comparison. In this way a great number of samples can be tested with a fair degree of accuracy. The test is sensitive to at least 1% of fish oil content, with the appearance of a cloud by a slight precipitate and after standing overnight.

The Wijs iodine is added to absorb some of the oil but mainly to permit observation of the endpoint approach and to permit the addition of a slight excess of bromine without obscuring the sedimentation. In this test, hexabromides will not precipitate even in the case of sunflowerseed oil. If in doubt, as much as possible of the supernatant liquid may be decanted away and chloroform added, or the test tube may be placed in boiling water as in the AOAC test. In either case, the octabromides of fish oil will remain insoluble.

A more precise method may be made of this test by weighing the sample oil, transferring the precipitate to a Gooch filter and washing it with cold ether, drying and weighing, and comparing with known fish oil mixtures. For most purposes the volumetric test suffices.

The test was checked by UV absorption of tetraenic and pentaenic fatty acids on the same deep sea tanks of four shipments.

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Suspension Stability of Solid Particles in the Presence of Various Types of Electrolytes

Sir: With reference to the article entitled "Suspension Stability of Solid Particles in the Presence of Various Types of Electrolytes" by Tokiwa and Imamura that appeared in the November 1969 issue of JAOCS, may I call the attention of the aforementioned authors and your readers to an article on the same subject that I published in the May 1952 issue of Industrial and Engineering Chemistry (44, 1151-1159, 1952).

My treatment of the subject covered the factors of the polarity of the suspended particles (I used

both MnO_2 and carbon powder), type of surfactant (anionic, nonionic and cationic), type of builder, cellulose gum additives, pH and temperature.

I also feel that no research on suspension stability should omit references to the basic work by Vold and his co-workers (J. Phys. Colloid Chem. 53, 67 and 1262, 1949).

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