

Update on methodology topics

The following column on methodology topics was prepared by AOCS Technical Director Dave Berner. Questions on these or other topics concerning analytical methodology of fats and oils or related products may be directed to Dave Berner, AOCS, PO Box 3489, Champaign, IL 61821-0489, USA.

Tanker cargo contamination

The July 1987 issue of *JAOCS* (page 968) noted concern about tanker cargo contamination. Since that time, trade associations, government organizations and shipping companies have been involved in discussions to find suitable methodology for detecting possible contamination in tanker cargo shipments. U.S. Environmental Protection Agency (EPA) methods for detecting priority pollutants seemed to be a logical starting point for evaluating existing methodology because the EPA methods were well-studied and defined.

E. Wayne Emmons, general manager and lab director for Charles V. Bacon Inc. laboratories in Galena Park, Texas, reports that repeated interference has been experienced with GC/FID packed columns and GC/MS capillary columns for the detection of benzene and styrene in vegetable oils, particularly crude oils. This interference appears to be due to natural constituents in the oils.

In an attempt to reduce or eliminate these interferences, HPLC was investigated as a means of detecting benzene, xylenes and styrene monomers in vegetable oils. At this time, Emmons says he has confidence only in the HPLC methods for these items. These methods have been properly verified by high and low recoveries, with excellent results from 0-20 parts per million (ppm). The benzene and xylene analyses are reported to be easier to perform than the styrene method, with the latter requiring more rigid laboratory techniques.

AOAC General Referee report

David Firestone, chairman of the

AOCS Uniform Methods Committee, is also the Association of Analytical Chemists' (AOAC) General Referee for oils and fats. Firestone's current report on oils and fats recently appeared in "General Referee Reports," *JAOAC* 71(1):76 (1988).

Firestone's report included information on investigations into methodology concerning antioxidants, emulsifiers, fatty acids (shorter chain), marine oils, olive oil adulteration, oxidized fats, pork fat in other fats, and sterols and tocopherols.

In his report, he recommended that (a) IUPAC method 2.310, Determination of Butyric Acid, be adopted by AOAC as an official first action; and (b) AOAC adopt as official final action the following first action methods: beta-sitosterol in butter oil (AOAC method 28.104-28.109), cyclopropene fatty acids in oils (AOAC method 28.120-28.123), foreign fats containing tristearin in lard (AOAC method 28.130-28.131) and chick edema factor (dioxins) in oils and fats (AOAC method 28.139-28.141). He also recommended that studies continue on all other topics.

Insoluble impurities method

Arthur Carnrick of Marshall Durbin Companies, Jackson, Mississippi, has been actively seeking an updated version of the AOCS insoluble impurities method Ca 3-46. Filtration problems were experienced with the current method when feed-grade fats with relatively high levels were analyzed.

In December 1987, individuals met in Memphis to suggest improved methodology for the analy-



sis of samples with high levels of impurities. On the basis of these recommendations, Carnrick developed four variations of the current method for study using shredded filtration medium, a two-gram sample, a "Gelman" filtration apparatus and petroleum ether.

Five laboratories participated in a study to compare these modifications to method Ca 3-46 with no modifications. A detailed report recently submitted by Carnrick concludes that a collaborative study should be organized to develop a new method using the "Gelman" filtration apparatus, petroleum ether and a five-gram sample. Alternately, shredded glass fiber filter paper may be studied vs. the current version of Ca 3-46.

The five laboratories participating in this preliminary study have indicated they are willing to participate in a collaborative study. Any other laboratory interested in participating should contact the AOCS technical director.

AOCS thanks Carnrick and the five laboratories for an excellent job in validating the proposed changes to the insoluble impurities method and for reporting the results in a detailed manner.

Dave Berner
AOCS Technical Director