Report of the Instrumental Techniques Committee[1] AOCS, 1971-72

Introduction

The Instrumental Techniques Committee met in conjunction with both of the national meetings of the Society during the past year. The first meeting was held during the 45th Fall Meeting in the Chalfonte-Haddon Hall Hotel, Atlantic City, N.J.,on Monday, October 14, 1971, with 15 members attending. The second meeting was held in the Detroit Room of the Los Angeles Hotel, Los Angeles, Calif., on Sunday, April 23, just prior to the 1972 JOCS-AOCS Joint Meeting, with nine members in attendance. Following usual custom, these meetings consisted mainly of reports of progress and plans for the immediate future by each of the subcommittee chairmen.

Spectroscopy Subcommittee

Progress of the Spectroscopy Subcommittee has been delayed while chairman Robert R. Allen has been busy with tasks incidental to his election and inauguration as president of the Society. The subcommittee is now making active plans to investigate, in collaborative effort, two proposals designed to broaden the scope and, under specified conditions, permit more rapid analyses of isolated *trans* isomers in fatty acids, esters and triglycerides with AOCS Official Method Cd 14-61. Both proposals require collaborative study of published methods. The first proposal would permit the determination to be made without recourse to external standards by a considerably more rapid technique and is based on a collaborative study of a published method entitled, "A Rapid Method for the Determination of *trans* Unsaturation in Fats and Derivatives" (1). The second collaborative effort designed to modify Method Cd 14-61 has for its objective the inclusion of a technique to permit more accurate measurements of very low concentrations of isolated *trans* content, based on the published method "Determination of Low Level Isolated *trans* Isomers in Vegetable Oils and Derived Methyl Esters by Differential Infrared Spectrophotometry" (2).

NMR Spectroscopy Subcommittee

The NMR Spectroscopy Subcommittee is still without a chairman. While attempting to locate a qualified candidate for this position, some progress has been made with cooperation between task group chairmen and the committee chairman.

A.J. Haighton, from Unilever Research, Vlaardingen, The Netherlands, is chairman of a task group established to study a method for the determination of solid-fat index by nuclear magnetic resonance. Despite the handicap of distance, Haighton has made considerable progress with this task group. During 1971, three collaborative investigations have been completed. In the first collaborative test, four laboratories participated, one from Sweden, two from the U.S. and one from the Netherlands. The collaborators were furnished five liquid oils: sunflowerseed, soybean, safflower, rapeseed and olive, with request to record their signal temperature curves. Solid fat content is mainly influenced by two factors: (a) influences inherent in the method and apparatus used; and (b) temperature treatments, temperature cycles, holding times and holding temperatures. The first collaborative test was concerned primarily with the first aspect, as the tempering method will have to be formulated in cooperation with other bodies such as the IUPAC and our Uniform Methods Committee.

Comparable signals of the oils were tabulated, and from analysis it is obvious that the intensity of the signals, as expected, depends greatly on the hydrogen content of the individual oils; in no case did the curves follow the theoretical course, due to saturation effects. It was concluded from this investigation that for the calculation of the solid-liquid ratio in fats, empirically found factors or a reference oil must be used for the calculation of the liquidus line of the fat, because no use can be made of the 1/T law.

In the second collaborative test five laboratories participated, one each in Sweden, Holland, England and two in the U.S. The collaborators were furnished three refined fat samples: palm oil, hardened fish oil and a margarine fat composed mainly of hardened soybean and sunflowerseed oils. As the first collaborative test had demonstrated that, for the calculation of the amount of solid phase, the liquidus curve cannot be theoretically determined from the 1/T law owing to saturation effects, a tube of olive oil was also furnished as a reference oil which was to be measured at all temperatures given in a precisely worded procedure furnished each participant.

Tabulation and analysis of the results obtained revealed a disappointing lack of accuracy. Standard deviations of the percentage of solids in the three oils were 10, 4 and 15%, respectively. It is obvious that there are systematic differences between the laboratories arising from either the apparatus used or in the sample treatment.

To establish the real cause of discrepancies or lack of agreement among the laboratories, a third collaborative test was designed in which the collaborators were asked to measure mixtures with a known percentage of solids. These samples were prepared to meet the following requirements: (a) the samples must crystallize in a stable crystal modification when a specific cooling procedure is applied; (b) they must have a low solubility in oil, as a result of which temperature fluctuations (even of a few degrees) in the bath and in the magnet do not influence the percentage of solid; and (c) the samples must be easy to prepare. The collaborators were furnished two samples containing 20.0 and 50.0% fully hydrogenated palm oil in 80 and 50% peanut oil, respectively, which met these requirements as well as a tin with the same peanut oil as reference. The collaborators were furnished specific instructions as to the treatments of these mixtures and for measuring their NMR spectra.

Results of this collaborative test are tabulated in Table I. These data show that the results obtained from the five laboratories with various types of apparatus are in surprisingly good agreement. This is especially evident from the difference in standard deviation of 1-2%, as compared to the 5-15% in the second collaborative effort. It is concluded that it now appears possible to achieve the same results with various types of apparatus in various laboratories. Hence differences in the first tests must be attributed mainly to the treatment of the samples, e.g., waiting times, temperature constancy of the baths, the presence or absence of a temperature air controller for the sample holder in the magnet, etc. The NMR Subcommittee has prepared a precise method to be submitted to the Instrumental Techniques Committee for approval for recommen-

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[1] Report of collaborative work from Government, Industrial and Academic Laboratories by members of ARS, USDA, Southern and Eastern Marketing and Nutrition Divisions; Anderson Clayton and Co., Food Division; Unilever Research; and A.E. Staley Mfg. Co.

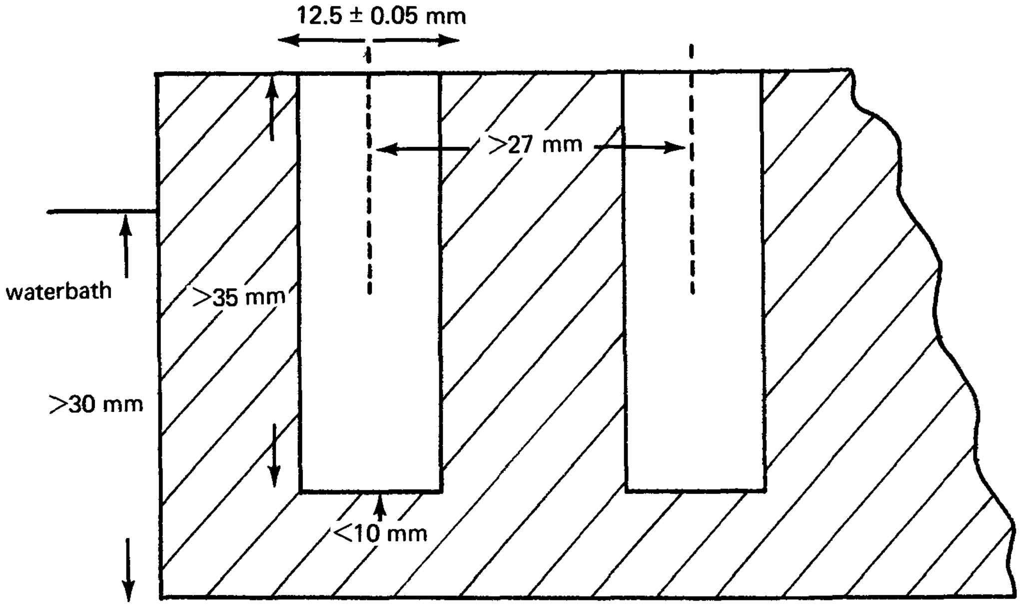

FIG. 1. Drawing of a part of the metal block.

dation to the Uniform Methods Committee for inclusion in *Offical and Tentative Methods of the AOCS* (Appendix II).

During the course of their investigations of procedures for the determination of solid-fat index by means of wide-line NMR spectroscopy, this task group of the NMR Subcommittee has tested, for comparison, procedures for determination of this index by means of pulsed-source NMR spectroscopy. Preliminary results indicate that this latter technique might be superior to the wide-line procedure. However decision has been made to continue with a method based on the wide-line technique for two reasons: (1) procedures based on wide-line NMR spectroscopy have been evaluated and found satisfactory, while pulsed-source techniques have just been introduced; and (2) even although found superior to the wide-line techniques, pulsed-source techniques will probably be available to only a relative few fatty acid research laboratories due to lack of appropriate equipment. It was therefore decided to continue with the recommendation of the wide-line NMR spectroscopy method for solid-fat index with the possible future modification to include the pulsed-source technique as an alternate procedure in the official method.

The NMR Subcommittee also has under consideration a proposal to investigate published methods for the determination of total oil content of oilseeds by means of NMR spectroscopy. Attempts are in progress to find a qualified task group leader and establish a task group to initiate collaborative testing with the objective of evaluating and recommending a specific procedure to be recommended as an AOCS official method.

Atomic Absorption Subcommittee

Following a modification of a published method (3), the Atomic Absorption Subcommittee has been working in collaborative effort to develop and evaluate a direct method for the determination of trace metals in vegetable oils and animal fats by atomic absorption spectrophotometry. Results of previous collaborative investigations have been published (4) and have led to a procedure that the subcommittee finds satisfactory. This method, reproduced in full in Appendix I, will be submitted to the entire Instrumental Techniques Committee for approval for submission to the Uniform Method Committee with recommendation for its inclusion in *Official and Tentative Methods of the AOCS.*

With the completion of this phase of their objective, the Atomic Absorption Subcommittee has been considering the extension of the method to the lower levels of trace metals commonly found in refined vegetable oils. Investigations have been made, in a preliminary manner, of proposed techniques for preconcentration or sample enrichment by such techniques as extraction of metals or ashing of the oil sample. Specifically, enrichment by an extraction procedure using ammonium 1-pyrrolidine-dithiocarbamate-hydrochloric acid mixtures at reflux temperatures as the extraction medium has shown, in preliminary experiments, quantitative recovery of copper and chromium added to soybean oil at the 0.2 ppm level and a 75% recovery of

TABLE I

The Average N-Values from Two Calibrants Containing 20.0 and 50.0% Solids

Laboratory		Instrument	Calibrant with 20.0%	Calibrant with 50.0% solids
Swift	U.S.	Varian	20.2	51.0
Karlshamn	Sweden A[a]	Newport	19.9	49.2
Karlshamn	Sweden B[a]	Newport	21.1	50.4
Unilever	Holland	Newport	20.5	50.2
University of Guelph	Canada	Newport	20.8	49.5
B.F.M.I.R.A.	England	Newport	20.6	49.0
Varian A.G.	Switzerland	Varian	20.3	49.3
Average			20.5	49.8
Standard deviation (% of N)			2.1	1.5

[a] A and B are two different instruments.

TABLE II

Proposed Methods and Their Present Status

Method	Status
1). The rapid analysis of isolated *trans* isomers in fatty acids, esters and triglycerides without recourse to external standards	Collaborative testing within the Spectroscopy Subcommittee
2). The determination of *trans* isomer content at low levels in fatty acids, esters and triglycerides	Collaborative study within the Spectroscopy Subcommittee
3). The determination of solid-fat ratio by wide-line NMR spectroscopy	Final draft of method in preparation for submission to entire ITC for approval
4). The determination of solid-fat ratio by means of pulse-source NMR spectroscopy	In planning stage for collaborative testing within the NMR Subcommittee
5). A method for the determination of total oil in oilseeds by wide-line NMR spectroscopy	Collaborative investigations of published methods pending formation of qualified task group within NMR Subcommittee
6). A direct method for the determinaton of trace metals in fats and oil by means of atomic absorption spectroscopy	Final draft to be submitted to entire ITC Committee for approval
7). A method for the determination of trace metals in refined vegetable oils at low levels by means of preconcentration techniques	Collaborative testing in planning stage within the Atomic Absorption Subcommittee
8). The determination of pesticides in fats, oils and other lipids by means of GLC	An AOAC method being evaluated within the GLC Subcommittee for adoption as an AOCS method
9). A method for the determination of fatty acid composition by means of programed GLC	Collaborative study within the GLC Subcommittee
10). A method for the quantitative determination of free sterols by GLC	Collaborative study within the GLC Subcommittee
11). A method for the determination of resin acids in rosin by means of GLC	Method recently adopted by ASTM being evaluated within the GLC Subcommittee for adoption as AOCS methods
12). Composition of turpentine by GLC	Ibid.

nickel. As an alternate approach to providing increased sensitivities, the subcommittee is investigating recent improvements in the graphite furnace and graphite rod atomizer systems. Detecting limits at the picogram level appear to be attainable by incorporation of these techniques. The subcommittee plans to continue investigation with the ultimate objective of devising, evaluating and recommending a satisfactory method for the determination of trace metals at very low levels.

Gas Chromatography Subcommittee

The Gas Chromatography Subcommittee has task groups involved in the investigation of four procedures based on gas liquid chromatographic techniques. Jairo E. Pena is chairman of a task group to investigate the determination of pesticides in fats, oils and other lipids by means of gas liquid chromatography (GLC). This group has been investigating AOAC methods for chlorinated and phosphated pesticides (5). The task group is considering the suitability of these methods for adoption as AOCS tentative methods.

John L. Iverson is chairman of a task group to investigate a procedure involving programed temperature analysis of fatty acid composition designed to modify or augment AOCS Tentative Method Ce 1-62, Rev. 1970. A third task group, under Francis E. Luddy as chairman, is initiating an investigation of the quantitative determination of free sterols in fats and oils. The ultimate object of this study is to select a specific method for recommendation for adoption as an official AOCS method to augment Ce 1-62 "Fatty Acid Composition by Gas Chromatography," particularly to provide a standard method for free sterol determination by GLC for the Joint FAO-WHO Codex Alimentarious Commission's proposal to use fatty acid composition coupled with free sterol composition, both as determined by GLC, to identify specific oils and fats (4).

B.D. Thomas has taken over activities to investigate and evaluate methods for the determination of resin acid in rosin and for the determination of turpentine by means of GLC. This task group is exploring gas chromatographic techniques for these determinations recently adopted by ASTM with the purpose of proposing them for adoption as AOCS methods.

Summary

As described in this report, the Instrumental Techniques Committee has in various stages of progress several methods which will be proposed for adoption as official methods of the AOCS. For convenience, these proposed methods and their present status are summarized in Table II.

R.T. O'CONNOR, CHAIRMAN
R.R. ALLEN, SUBCOMMITTEE CHAIRMAN
K.M. BROBST, SUBCOMMITTEE CHAIRMAN
S.F. HERB, SUBCOMMITTEE CHAIRMAN

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5. Association of Official Analytical Chemists "Methods of Analysis," 11th Edition, 29 "Pesticide Residues" p. 475 Methods 29.001, 29.002, 29.005, 29.008 and 29.011 through 29.017, 1971.

APPENDIX I

Analyses for Chromium, Copper, Iron and Nickel in Vegetable Oils by Atomic Absorption Spectrophotometry

Definition

Vegetable oils dissolved in methyl isobutyl ketone

(MIBK) are analyzed for metals by direct aspiration. Calibration is based on MIBK solutions of organometallic reference compounds solubilized in purified vegetable oil.

Scope

This method is intended for the analysis of crude or partially refined vegetable oils which contain at least 0.3 ppm of each measured metal in the form of oil soluble adducts. Hardened oils and animal fats have limited solubilities in MIBK, and even at reduced solute concentrations they readily foul the burner. For these reasons this method is not recommended for such samples (Note 1).

A. Apparatus

1. Atomic absorption spectrophotometer, air-acetylene laminar-flow burner head, and appropriate hollow cathode lamps
2. Desiccator containing fresh phosphorus pentoxide
3. Weighing bottles, 10-15 ml
4. Hot plate with rheostat temperature control
5. Oven
6. Beakers, 25 ml and 100 ml
7. Volumetric flasks, 25 ml, 50 ml and 250 ml
8. Polyethylene bottles, 2 oz and 4 oz
9. Pipettes, graduated, T.D., 5 ml and 50 ml
10. Balance, top loading, 500 g capacity with ± 0.01 g sensitivity
11. Balance, analytical, 200 g capacity with 0.0001 g sensitivity

B. Reagents

1. Organometallic standards, NBS or equivalent
   a. tris (1-phenyl-1,3-butanediono) chromium III, NBS #1078a (9.7% Cr)
   b. bis (1-phenyl-1,3-butanediono) copper II, NBS #1080 (16.5% Cu)
   c. tris (1-phenyl-1,3-butanediono) iron III, NBS #1079a (10.30% Fe)
   d. nickel cyclohexanebutyrate, NBS #1065b (13.89% Ni)
2. 4-Methyl-2-pentanone (methyl isobutyl ketone), reagent grade, Eastman #416, or equivalent
3. Xylene, reagent grade, Eastman #13040, or equivalent
4. 2-Ethylhexanoic acid, Eastman #P4442
5. 2-Ethylhexylamine, Eastman #P6759
6. Purified vegetable oil (base oil): corn or soybean oil containing not more than 0.02 ppm Cr, Cu, Ni or Fe can be prepared by distilling a low-ash refined oil on a rotating disc molecular still, rejecting the first and last 15% fractions
7. Phosphorus pentoxide, reagent grade, Fisher Scientific Co. #A-245, or equivalent

C. Preparation of standards

1. Chromium stock standard, 100 μg/g: Dry ca. 0.1 g tris (1-phenyl-1,3-butanediono) chromium III (9.7% Cr) 1 hr at 110 C. Weight 51.6 mg into a 25 ml beaker, add 3 ml xylene, 3 ml 2-ethylhexanoic acid, and warm on hot plate without refluxing, swirling occasionally until salt dissolves, then let cool to ambient. Transfer solution to a tared 2 oz polyethylene bottle, rinsing beaker with five 5 ml portions of base oil, then bringing the contents to 50 ± 0.05 g with additional base oil. Purge head-space with nitrogen, cap, mix and store in a cool location. Standard solution should be stable for at least 6 months.

2. Copper stock standard, 100 μg/g: Dry ca. 0.05 g bis (1-phenyl-1,3-butanediono) copper II (16.5% Cu) 30 min at 110 C. Weigh 30.3 mg into 25 ml beaker, add 2 ml xylene, 2 ml 2-ethylhexylamine and proceed as above beginning with "and warm on hot plate . . . ."
3. Iron stock standard, 100 μg/g: Dry ca. 0.1 g tris (1-phenyl-1,3-butanediono) iron III (10.30% Fe) for 1 hr at 100 C. Weigh 48.6 mg into a 25 ml beaker and proceed as in C.1. beginning with "add 3 ml xylene . . . ."
4. Nickel stock standard, 100 μg/g: Desiccate ca 0.05 g nickel cyclohexane butyrate (13.89% Ni) for 48 hr over phosphorus pentoxide. Quickly weigh 36.0 mg into a 25 ml beaker and proceed as in C.1. beginning with "add 3 ml xylene . . . ."
5. Mixed stock standard, 20 μg/g Cr, Cu, Fe and Ni: Weigh carefully by additions into a tared 4 oz bottle 20 ± 0.02 g each of the 100 μg/g Cr, Cu, Fe and Ni stock standards, and 20 ± 0.02 g base oil. Purge with nitrogen, cap and mix.
6. Mixed working standards: Weigh 50 ± 0.1 g base oil into 100 ml beaker, transfer to 250 ml volume flask with MIBK, dilute to volume, and mix (20 wt/volume % base oil in MIBK). Weigh accurately (± 5 mg) 5.00 g, 3.75 g, 2.50 g, 1.25 g and 0.625 g mixed stock standard into 50 ml volume flasks. In order, add by pipette 25.0 ml, 31.3 ml, 37.5 ml, 43.8 ml and 46.9 ml of the 20% base oil in MIBK solution. Dilute to volume with MIBK and mix. These solutions contain 2.0, 1.5, 1.0, 0.5 and 0.25 μg/ml Cr, Cu, Ni and Fe. Retain the remaining 20% base oil solution for zeroing the spectrophotometer and, if necessary, diluting the sample oil.

D. Procedure

1. Weigh accurately 5.00 ± 0.0005 g of sample oil into a 25 ml volume flask, dilute with MIBK and mix.
2. Set up the instrument for operation with an air-acetylene-organic solvent flame according to the manufacturer's recommendations (Note 2).
3. Install the approiate hollow cathode lamp, turn on the instrument, adjust to the recommended operating lamp current, set the monochromator to the recommended wave length (see following table) and allow the instrument to stabilize.
4. Light the burner and allow to warm to equilibrium (ca. 3 min).
5. While aspirating the 20% base oil, solution, reduce the acetylene flow until the yellow incandescence of the flame just disappears. Next, optimize the fuel-air ratio for the determination (Note 3) by varying the acetylene flow while aspirating one of the mixed standards until a maximum absorption is obtained. Flush the burner by aspirating MIBK for 15 sec (Note 4) and then zero the instrument while aspirating the 20% base oil solution.
6. Record absorptions for the five mixed working standards in order of increasing concentration. Record the absorption of the sample solution and then aspirate the 20% base oil solution to check the zero point (Note 5). If the sample absorption exceeds the highest standard, prepare a new sample solution by weighing a suitable ratio of sample oil to base oil to produce a 20% oil solution which absorbs within the calibration range and repeat the absorption measurements.
7. From the standard absorptions construct a calibra-

tion curve to be used to determined the concentration (μg/ml) of metal in the sample solution.

8. Repeat steps 3-7 for each analysis using the appropriate source lamp and conditions.

Recommended Wave Lengths

Element	Wave length, A
Cr	3579
Cu	3248
Fe	2483
Ni	2320

E. Calculations

$$\text{Metal, } \mu g/g = C \times \frac{25}{W} = \text{ppm}$$

where:

C = metal in μg/ml from calibration graph.

W = grams sample oil in 25 ml of sample solution.

F. Precision

The following tabulation gives the standard deviations for each metal analysis. Eight collaborators analyzed two samples of soybean oil. Test solutions were retained and absorption measurements were repeated on the following day.

Element	Level, μg/g	Standard deviations		
		Day 1	Day 2	Average
Cr	2.5	0.0535	0.1155	0.0845
	8.5	0.2654	0.3007	0.283
Cu	3.5	0.1137	0.1344	0.124
	9.5	0.1574	0.1940	0.176
Fe	2.0	0.0627	0.1640	0.113
	8.0	0.4990	0.2875	0.393
Ni	3.0	0.3599	0.3239	0.342
	9.0	0.3159	0.2797	0.298

Notes

1) Mixed solvent systems, i.e., chloroform-MIBK), may reduce fat solubility problem and lessen burner fouling, but it will be necessary to prepare the standards in the same solvent system.

2) If a Boling burner head is used, be sure that it is designed to withstand the highly oxidizing conditions required for the nickel determination. Boling burners of an early design could be permanently warped under these conditions.

3) Chromium requires a rich (yellow) flame, copper and iron a lean (blue) flame and nickel a very lean flame.

4) The MIBK flush removes contaminated oill and minimizes fouling of the burner head by oil buildup.

5) With correct fuel adjustment a linear calibration curve which intercepts the origin can usually be obtained.

APPENDIX II

Draft for a Standard Procedure

Determination of the per cent solids in fats by wide-line NMR

The percentage solids of a partly crystallized oil and fat mixture is measured after solidification of the mixture under carefully prescribed conditions.

Principle

Wide-line NMR gives only a signal for the liquid part in a mixture of solid and liquid material. For the determination of the percentage solids, therefore, two signals at the same temperature are required: (a) the signal of the sample (oil + crystals); (b) the signal of the same sample in totally liquid form. The second signal can be found by extrapolation of the oil signal at 60 C (by multiplication of this signal with the slope of the oil line determined with a reference oil).

Apparatus

A wide-line NMR apparatus capable of taking sample tubes of 12 mm diameter (Note 1). A digital voltmeter and, if available, a printer. A temperature controller blowing dry air of constant temperature through the sample holder (Note 2). Metal blocks (aluminum) (Note 3) with holes for the sample tubes dimensions holes: depth ⩾35 mm, i.e., at least twice the height of the fat column; internal diameter 12.5 ± 0.05 mm, i.e., to ensure a good contact between glass and metal (good heat transfer); distance between the holes >20 mm (center to center); thickness metal under the holes ⩽10 mm; emersion depth in waterbath >30 mm (Fig. 1). Constant temperature waterbaths (minimum 2). Waterbaths must be fitted with thermostatic controls, capable of holding the temperature of the water at any set temperature between 10 and 60 C with a precision of ±0.1 C. Equipped with a vigorous stirrer and a thermometer accurate to 0.05 C. The heating elements of the baths must have sufficient capacity to raise the temperature of the water by 10 degrees in a maximum of 8 min if two water baths are used (Note 4). The sample tubes are transferred in a constant sequence from the metal block in water bath (a) via the magnet to a block in water bath (b) with a higher temperature.

0 C bath: If crushed ice is used as the 0 C bath it must be avoided that water at a temperature above 0 C collects at the bottom of the vessel. The ice should be repeatedly stirred and repacked. A cooling unit maintained at 0 C is recommended.

Sample tubes: dimensions: external diameter 12.0 ± 0.3 mm; height ⩾45 mm; thickness wall ca. 0.6 mm.

Samples

Sample weight: between 1.5-1.8 g exact weighing is not necessary (filling to a volume mark is sufficient).

Reference oil: a fused tube with olive oil (Note 5).

Procedure

Three procedures for tempering (Note 6) may be used:

(a) short range; temperatures of measurement 20, 25, 30, 35 and every 5 C up to 60 C as required; (b) long range; temperatures of measurement 10, 15, 20, 25 and every 5 C up to 60 C as required; (c) rapid method for factory control; temperatures of measurement 20 and 30 C and any higher temperature required. On reporting, the designation "short range," "long range" or "rapid" must be added, since the number of temperature steps applied influences the solid content values.

Filter the melted fat if it is not clear. Fill the sample tubes with fat (1.5-1.8 g) and place them in the metal block in a bath of 60 C. After 30 min at 60 C, the first tube (reference oil) is placed in the magnet, through which air of 60 C is blown. The average of two readings at the digital voltmeter is used for the calculation. At 60 C the reference oil is measured twice at the beginning and the end of the series. When two values deviate less than 1.5% the average is taken for the calculation. At a higher value all the measurements at 60 C must be repeated (instrumental instability). After the first measurement of the reference oil the other samples (including again the reference oil) are measured in a fixed sequence and placed in a bath of 0 C every minute and a half (Note 7). After 30 min the signals are measured at 10 C (air through the magnet also 10 C) and the samples are then placed in a bath at the next measuring temperature. Repeat the procedure for all the measuring temperatures. If by mistake the temperature of the bath exceeds the required temperature, it must not be cooled because the melted part of the fat will not crystallize again. Maintain the wrong temperature and report the NMR values at that temperature.

Calculation

$$\text{Per cent solids at } t \text{ C } (N_t) = 100 - \left(\frac{\text{sample reading at } t \text{ C}}{\text{sample reading at 60 C}} \times \frac{\text{reference reading at 60 C}}{\text{reference reading at } t \text{ C}}\right) \times 100.$$

Instrument

At the moment the Newport Quantity Analyser (delivered by Newport Instruments, U.K.) is the most used instrument. Therefore the required settings for this instrument are given below.

Newport Quantity Analyser with supplementary modulation unit and 2 ml sample assembly. Settings: RF-scale: 100 μA; RP-level: 60 μA (An improved Quantity Analyser has been developed, the MK II; with this instrument it is possible to measure at a 25 μA-RF level with the same results and accuracy as with the MK I at 60 μA.); AF gain: setting such that the signal at 0 C for the reference oil is ca. 10% below the maximum of the digital voltmeter; Supplementary modulation unit: on; Automatic loss control: high; Integration time: 2 times x 4 (2 x44 sec) after placing the tube in the magnet push the reset knob, wait until two readings are given and use the average value for the calculation. The maximum number of samples 12 + 1 reference oil depends on the measuring time in the magnet and the tempering time at each measuring temperature; Temperature controller: on; Oscilloscope: two peaks must lie totally within the filter gates.

ANNOUNCEMENT

The AOCS Governing Board has approved a revision to the price of AOCS Official and Tentative Methods. Effective immediately, a complete set will still cost $50, but a complete set will include all revisions through 1969. The 1970 and 1971 sets will continue to sell for $6 each.

Notes

1) 2 ml sample tubes are most widely used and are efficient regarding waiting times. Solid phase values depend on the cooling time and cooling profile. Hence it is essential to have strict constraints regarding the tube dimensions. The tubes mentioned in the procedure can easily be obtained from, e.g., Newport Instruments.

2) When use is made of a constant, well dried airstream with a flow of minimally 25 liters/min it is not necessary to have an accurate control system for the air temperature. For temperatures between 0 and 40 C the variation can be plus or minus 2 C; above 40 C, the air temperature should not be lower than the sample temperature, so that a variation of only plus 2 C is permitted (e.g., at a sample temperature of 60 C the air temperature may be between 60 and 62 C). The air temperature must be measured as closely as possible to the magnet (= sample holder).

3) Provided the block has a uniform temperature, the size and form are not important. The size limits given in the drawing must be strictly adhered to because otherwise differences in heat transfer between sample tube and metal block can be expected.

4) The number of samples (full capacity 12 + 1 reference oil), the tempering time of 30 min at each measuring temperature and the time necessary for changing the air temperature through the magnet determine the time necessary to reach a temperature which is 10 degrees higher.

5) Olive oil is chosen as reference oil because of its stable character and because of its average number of H protons in comparison with hydrogenated fats and fat compositions. The reference (olive) oil is measured at each temperature together with the samples for correcting instrumental instabilities. Oils with a saponification value between 191-193 and an iodine value between 83-89, e.g., peanut oil, can also be used provided they remain clear at 10 C.

6) This procedure is chosen because it allows the possibility to measure solid phase values of different products in a reasonable time, e.g., the short range for hardened fats and tropical margarines; the long range for soft margarines (refrigerator margarines); the rapid method for factory control, e.g., hardening. It is proposed here to carry out the cooling step at 0 C because (a) this temperature can be reached conveniently in all laboratories; (b) less probability of formation of metastable polymorfic forms melting at too low temperatures; and (c) mixed crystals are formed under conditions more closely resembling those obtained in the factory cooling process.

7) The samples should be transferred every minute and a half in the same sequence to the next temperature bath, as all the samples should be allowed to stand at the same temperature for exactly 30 min. Some fats are not yet in equilibrium after a holding time of 30 min; hence it is important to keep this time at exactly 30 min.

Appendix I

For checking the performance of the instrument from time to time, fat samples with known percentage solids can be used. (a) As solid phase can be used: palm oil hydrogenated to an iodine value of 5. The liquid part of the hardened palm oil should be removed by crystallizing twice from acetone (1:5) at 20 C. Dissolve 1 part of molten palm oil in 5 parts acetone at 50 C. Cool at 1 C/min to room temperature and filter the crystals. Repeat this procedure with the crystals. After fractionating the crystals must be

(Continued on page 437A)

Mahadevan joins Supelco as Senior Biochemist

Vaidyanath Mahadevan has joined Supelco, Inc., Bellefonte, Pa., as Senior Biochemist. Mahadevan has many years experience in biochemistry, having spent 6 years as a Research Fellow, Government of India; 1 year as Research Fellow, Banting and Best Department of Medical Research, University of Toronto, Canada; 9 years as Research Fellow and Assistant Professor of Biochemistry at Hormel Institute, University of Minnesota; and 5 years as a Biochemist with the Veterans Administration Hospital in Minneapolis, Minnesota.

Mahadevan received his Ph.D. in biochemistry from the University of Minnesota in 1956. He has been author and coauthor of 50 publications in scientific journals which included reviews and chapters in books regarding lipids. In addition to AOCS, his professional memberships include Phi Lambda Upsilon, Sigma Xi, American Chemical Society, AAAS and the New York Academy of Sciences.

Mahadevan's research interests include dilatometric investigations on glycerides, synthesis of neutral lipids and phospholipids, lipids in relation to blood coagulation and the chemistry of blood lipids. ■

• Instrumental Techniques. . .

(Continued from page 436A)

dried at 40 C under vacuum for 1 day. (b) As liquid phase a winterized oil can be used. Winterization of the oil: remove solids deposited after standing 4 days at 0 C.

In order to obtain a stable standard, after mixing the two fat phases *a* and *b* (in a chosen proportion) the mixture must be treated in the following manner: (a) melt the standard mix at 70 C; keep at this temperature for ca. 30 min; (b) fill the NMR tube with fat sample and weigh, in this case, accurate to within 0.001 g; (c) cool *slowly* from 70 to 20 C in at least 8 hr (ca. 6 C/hr); (d) leave overnight at room temperature (ca. 20 C); (e) measure at room temperature with the pure winterized oil as reference calculated as volts per gram; (f) calculation of the per cent solid phase:

$$= 100 - \left(\frac{\text{sample reading V/g}}{\text{reference reading V/g}} \times 100\right)$$

sample and reference must have exactly the same temperature (ca. 20 C). Because of the low solubility of the hard fat phase this temperature may lie between 10 and 25 C.

Note: In general, for a standard with 20% hardened palm oil, a value between 19.5 and 20.5 was found on different instruments in different laboratories.

Appendix II

For fats such as cocoa butter, confectionery fats, tallow, oleo oil and tallow stearin, special stabilization procedures have to be used.

L.F. VERMAAS
Unilever Research Laboratory
Olivier van Noortlaan 120
Vlaardingen, The Netherlands
July 1972 ■

Carl Blumenstein joins staff of Nolan Co.

The Nolan Co. announces the addition of Carl R. Blumenstein to its staff as Regional Associate for the Southeastern States of North and South Carolina, Georgia, Alabama and Northern Florida. The Nolan Co. assists buyers and sellers in purchases and sales of manufacturing companies in the scientific fields throughout the country, as well as searching for firms desirous of merging their interests with other companies.

An organic chemistry graduate of the University of Maryland, Blumenstein has had 30 years experience in textile chemistry, surfactants, resins and polymers, and sanitary and maintenance chemicals. His experience includes research and development management, marketing studies, and plant layout and equipment planning. Prior to joining The Nolan Co. he was Technical Director for Seydel-Woolley & Co. of Atlanta, and Vice-President for Research at Texize Chemicals of Greenville, S.C. For the past 4 years has has been in his own consulting business. He will be working under the guidance of Paul M. Goodloe, President, from whose East Orange, N.J. office the national activities of The Nolan Co. are coordinated.

An AOCS member since 1962, Blumenstein belongs to several other professional societies, including the American Chemical Society and the American Association of Textile Chemists and Colorists. ■

Obituary

Word has been received of the death of T.R. Wannamaker on August 19, 1972. Wannamaker joined AOCS in 1935 and was an emeritus member at the time of his death.

Chemetron's Votator Division names E.T. Beck to top position

AOCS member E.T. Beck has been appointed Executive Vice-President of the Chemetron Corporation's Louisville-based Votator Division.

Announcement of the appointment was made by N. Tift Joyner, President of the Votator Division, which serves the food processing and chemical processing industries with such products as heat exchange equipment, fillers for containers, and deodorizer plants for treatment of fats and oils. The division also manufactures and markets Thermex high frequency industrial heating equipment.

Beck directs marketing and sales programs and also assists in the general management of the division. He joined the Votator operation in 1948 and served as Manager for the eastern Sales District in New York from 1951 to 1957. For four years starting in 1957, Beck was the division's Manager of Overseas Operations, and in 1961 he was named Sales Manager for the division. He became General Sales Manager in 1967 and Vice-President for Sales and Marketing a year later.

Beck is a graduate of the University of Kentucky with a metallurgical engineering degree. He is a member and former director of the Food Processing Machinery and Supply Association, the American Society of Mechanical Engineers and the American Institute of Metallurgical Engineers. ■