

# INTERNATIONAL UNION OF PURE AND APPLIED CHEMISTRY

APPLIED CHEMISTRY DIVISION  
COMMISSION ON OILS, FATS AND DERIVATIVES\*

## DETERMINATION OF COPPER, IRON AND NICKEL IN OILS AND FATS BY DIRECT GRAPHITE FURNACE ATOMIC ABSORPTION SPECTROMETRY

### Results of a collaborative study and the standardised method

*Prepared for publication by*

P. W. HENDRIKSE<sup>1</sup>, F. J. SLIKKERVEER<sup>1</sup>, J. ZAALBERG<sup>1</sup> and A. HAUTFENNE<sup>2</sup>

<sup>1</sup>Unilever Research Laboratory Vlaardingen, The Netherlands

<sup>2</sup>Université Catholique de Louvain, Unité des Procédés (PRCD), B-1348 Louvain-la-Neuve, Belgium

\* Membership of the Commission during the period of the collaborative study (1983–87) was as follows:

*Chairman:* 1983–85 M. Naudet (France); 1985–87 Ö. Levin (Sweden); *Vice-Chairman:* A. Hautfenne (Belgium); *Secretary:* W. D. Pocklington (UK); *Titular Members:* J. L. Beare-Rogers (Canada; 1985–87); É. Kurucz (Hungary; 1985–87); Ö. Levin (Sweden; 1983–85); J. Pokorný (Czechoslovakia; 1983–85); H. Wessels (FRG); *Associate Members:* J. L. Beare-Rogers (Canada; 1983–85); A. Dieffenbacher (Switzerland); E. Fedeli (Italy); J. Gracian-Tous (Spain; 1983–85); T. Hashimoto (Japan; 1985–87); G. Højlmer (Denmark; 1985–87); B. Jacobsberg (Belgium); A. Karleskind (France; 1985–87); F. H. Koelman (Netherlands); É. Kurucz (Hungary; 1983–85); R. G. Manning (USA); A. T. Møller (Denmark; 1983–85); R. Ohlson (Sweden; 1985–87); M. Teupel (FRG); A. Visapää (Finland); G. Zwerenz (Austria); *National Representatives:* A. M. Gad (Arab Republic of Egypt); N. G. Bazan (Argentina; 1983–85); R. R. Brenner (Argentina; 1985–87); A. R. Johnson (Australia); R. C. A. Lago (Brazil; 1983–85); D. G. Chobanov (Bulgaria); A. P. Tulloch<sup>†</sup> (Canada); J. Pokorný (Czechoslovakia; 1985–87); G. Højlmer (Denmark; 1983–85); J. V. Holm (Denmark; 1985–87); G. Ostermann (FRG); R. Linko (Finland); J. P. Wolff (France); V. M. Kapoulas (Greece; 1983–85); D. Boskou (Greece; 1985–87); M. Jeránek (Hungary; 1985–87); N. V. Bringi (India; 1983–85); A. G. Mathew (India; 1985–87); M. McCarthy (Ireland; 1983–85); A. M. Sharkey (Ireland; 1985–87); A. Dolev (Israel); E. Tiscornia (Italy); T. Hashimoto (Japan; 1983–85); S. Watanabe (Japan; 1985–87); A. S. H. Ong (Malaysia); P. W. Hendrikse (Netherlands); F. B. Shorland (New Zealand; 1983–85); L. Eyres (New Zealand; 1985–87); W. Zwierzykowski (Poland); G. D. Carr (Republic of South Africa); R. Stoica (Romania); M. Gassiot Matas (Spain); R. Ohlson (Sweden; 1983–85); H. Brüsweiler (Switzerland); R. Perin (Turkey; 1983–85); T. Tuğlular (Turkey; 1985–87); V. L. Roubajilo (USSR); A. E. Waltking (USA).

<sup>†</sup> deceased 1987

---

*Republication of this report is permitted without the need for formal IUPAC permission on condition that an acknowledgement, with full reference together with IUPAC copyright symbol (© 1988 IUPAC), is printed. Publication of a translation into another language is subject to the additional condition of prior approval from the relevant IUPAC National Adhering Organization.*

# Determination of copper, iron and nickel in oils and fats by direct graphite furnace atomic absorption spectrometry: results of a collaborative study and the standardised method

**Abstract-** A description is given of the development by collaborative study of a standardized method for the determination of copper, iron and nickel in edible oils and fats by direct graphite furnace atomic absorption spectrometry. The procedure is both rapid and sensitive allowing determination at levels of 0.005-0.20 mg/kg for copper and 0.01-1.00 mg/kg for iron and nickel.

## INTRODUCTION

The pro-oxidant effect of small amounts of metals in edible oils and fats has been known for many years (1). In particular copper, iron and nickel have a catalytic effect on the mechanism of autoxidation (2-8). The metals present in oils and fats may be of natural origin or due to processing actions such as bleaching (Fe), hardening (Ni, Cu) and corrosion of processing equipment (Fe, Ni). In the oils and fat industry a rapid, accurate and standardized method for the determination of these metals is very important for quality control.

Atomic absorption spectrometry (AAS) has been generally accepted for the determination of Cu, Fe and Ni in oils and fats, and a variety of procedures based on AAS are currently used.

## COLLABORATIVE STUDY

The method subjected to collaborative study was based on a thoroughly tested procedure. In order to check the validity of the method as an international standard method for both IUPAC and ISO, the method has been subjected to an extensive international collaborative study by the IUPAC Commission VI3, members of ISO/TC34/SC11 and other laboratories worldwide. The method studied took into account that various types of equipment for graphite furnace atomic absorption spectrometry can be used. To this end the possibility of dilution of the oil or fat prior to injection into the graphite cell was given as an alternative procedure.

Materials provided for the study were oils (soya bean and groundnut) and fats (cocoa butters) containing copper, iron and nickel at three concentration levels (high, medium and low). Nickel was not required to be determined in the cocoa butters since this metal is not normally found in cocoa butter.

Each concentration level was represented by two batches. In the case of oils by soya bean and groundnut oil, and in the case of fats by two cocoa butters. Each sample was provided in duplicate (blind coded) so that participants received in all 24 samples. Participants were asked to analyse each sample in duplicate and to report both values obtained. A statistical evaluation of the data was made for each level and for each type of sample separately.

## RESULTS

**Data screening.** From 32 laboratories data have been received for evaluation. Of these laboratories three did not report results for Ni and two did not report duplicate results. At a first survey four laboratories have been excluded from the test because of deviations from the method. Moreover, the Cu data from one laboratory were judged unsuitable for inclusion in the test. The data from the remaining laboratories (27 for Cu, 28 for Fe, 27 for Ni) have been subjected to tests for outliers according to Cochran and Dixon.

**Cochran tests.** As the 24 samples analysed were in fact 12 pairs of corresponding samples, the differences between these blind (hidden) duplicates have been tested for stragglers and outliers according to Cochran's procedure.

**Dixon tests.** After elimination of the significant outliers (i.e. significant at the 1% level) according to Cochran's test, the twelve batch averages per laboratory have been tested for stragglers and outliers according to Dixon's procedure.

Most of the deviations concerned the copper data in which two laboratories displayed so many deviations that it has been decided to omit the data for these laboratories from further evaluation. All other data have been maintained in the evaluation after discarding the outliers. Hence, for the final calculations of repeatability and reproducibility, there remains a total of 25, 28 and 27 laboratories for copper, iron and nickel data respectively.

## Precision

In Table 1 the batch averages for Cu, Fe and Ni at each concentration level in liquid oil or in solid fat have been listed. The results of two batches at one concentration level are close enough to each other to be considered representative of the same concentration, except for iron at the low concentration level in solid fat: batch CB1 is closer to the medium than to the low level. Therefore, averages per concentration level have been calculated in all cases except for the low iron level in solid fat; in the latter case, the calculation of the precision parameters, too, has been carried out per batch instead of per concentration level.

Table 1 Concentration levels and average recoveries (mg/kg) of Cu, Fe and Ni in oils and fats

Substrate	Concentration level	Batch	Copper (k = 25)		Iron (k = 28)		Nickel (k = 27)	
			Batch averages	Level averages	Batch averages	Level averages	Batch averages	Level averages
Oil	High	SB	0.1515		0.6764		0.8037	
		GN	0.1344	0.1429	0.8202	0.7483	0.7291	0.7664
	Medium	SB	0.0896		0.4312		0.5067	
		GN	0.0803	0.0850	0.5058	0.4685	0.4751	0.4909
	Low	SB	0.0324		0.1524		0.1354	
		GN	0.0387	0.0355	0.1429	0.1477	0.1715	0.1535
Fat	High	CB1	0.1499		0.8349	0.9007		
		CB2	0.1554	0.1526	0.9665			
	Medium	CB1	0.1003		0.5842			
		CB2	0.1042	0.1023	0.5574	0.5758	b	b
	Low	CB1	0.0363		0.4137			
		CB2	0.0446	0.0404	0.1334	a		

Notes: <sup>a</sup> Batch averages are so far apart that they cannot reasonably be regarded as batches from the same concentration level. Key: SB = soya bean  
<sup>b</sup> Nickel usually does not occur in cocoa butters; measuring was therefore considered irrelevant. GN = groundnut  
CB = cocoa butter

Table 2 Precision parameters (mg/kg) of the collaborative AAS study 1984

Metal	Substrate	Concentration	Parameters							
			m	s <sub>A</sub>	s <sub>r</sub>	r	CV <sub>r</sub> <sup>a</sup>	s <sub>R</sub>	R	CV <sub>r</sub> <sup>a</sup>
Cu (k = 25)	Oil	High	0.143	0.0055	0.0107	0.030	7.5	0.0214	0.061	15.0
		Medium	0.085	0.0041	0.0078	0.022	9.2	0.0135	0.038	15.8
		Low	0.036	0.0026	0.0054	0.015	15.1	0.0076	0.021	21.4
	Fat	High	0.153	0.0073	0.0097	0.027	6.4	0.0270	0.076	17.7
		Medium	0.102	0.0039	0.0055	0.016	5.4	0.0192	0.054	18.8
		Low	0.040	0.0030	0.0045	0.013	11.2	0.0079	0.022	19.5
Fe (k = 28)	Oil	High	0.748	0.0276	0.0465	0.132	6.2	0.1471	0.416	19.7
		Medium	0.469	0.0253	0.0435	0.123	9.3	0.0887	0.251	18.9
		Low	0.148	0.0174	0.0307	0.087	20.8	0.0397	0.112	26.9
	Fat	High	0.901	0.0377	0.0669	0.189	7.4	0.1834	0.519	20.4
		Medium	0.576	0.0283	0.0507	0.143	8.8	0.1184	0.355	20.6
		Low <sup>b</sup>	0.414	0.0275	0.0395	0.112	9.5	0.0935	0.264	22.6
Ni (k = 27)	Oil	High	0.133	0.0095	0.0179	0.051	13.4	0.0363	0.103	27.2
		Medium	0.766	0.0315	0.0493	0.139	6.4	0.1299	0.367	16.9
		Low	0.491	0.0202	0.0475	0.134	9.7	0.0853	0.244	17.6
	Fat	High	0.153	0.0134	0.0258	0.073	16.8	0.0337	0.095	22.0
		Medium								
		Low								

Notes: <sup>a</sup> expressed in %<sup>b</sup> as the m-values of both batches were too far apart, precision parameters have been given for each batch separately.

Key: m = mean value of the relevant characteristics at the specified concentration level.  
s<sub>A</sub> = standard deviation from known duplicates (analytical error).  
s<sub>r</sub> = repeatability standard deviation.  
r = repeatability.  
CV<sub>r</sub> = repeatability coefficient of variation.  
s<sub>R</sub> = reproducibility standard deviation.  
R = reproducibility.  
CV<sub>R</sub> = reproducibility coefficient of variation.

When the values for  $r$  (repeatability) and  $R$  (reproducibility), as given in Table 2, are expressed as functions of their corresponding mean concentration values ( $m$ ), the following equations are obtained:

Cu in oil	$r = 0.0102 + 0.140 m$	$R = 0.0085 + 0.358 m$	[Eq. 1a]
in fat	$r = 0.0076 + 0.109 m$	$R = 0.0028 + 0.492 m$	[Eq. 1b]
Fe in oil	$r = 0.077 + 0.081 m$	$R = 0.040 + 0.480 m$	[Eq. 2a]
in fat	$r = 0.026 + 0.196 m$	$R = 0.031 + 0.543 m$	[Eq. 2b]
Ni in oil	$r = 0.056 + 0.127 m$	$R = 0.027 + 0.442 m$	[Eq. 3]

## DISCUSSION

### Duplicates

Two types of duplicates have to be distinguished:

- duplicate determinations in the same sample, so the operator knows the concentrations to be found should be equal;
- duplicate samples of which the identity has been hidden by randomization and coding.

Two types of standard deviations have therefore been derived:

- $s_A$ , the standard deviation from the known duplicates, also indicated as analytical error;
- $s_r$ , the repeatability standard deviation, which is one of the parameters asked for in collaborative studies.

From Table 2 it is clear that  $s_r$  derived from blind duplicates is generally considerably larger than  $s_A$ ; this means that the repeatability, estimated from "uniform level" collaborative studies as indicated by ISO (9) will often be an underestimate of the true repeatability. The incorporation of blind duplicates (double split level) is therefore preferred.

### Relative precision

From the equations describing the precision as linear functions of the metal concentration (Eqs. 1-3) it can be concluded that the straight lines corresponding to these equations do not pass through the origin, but intersect the ordinate invariably at positive values. This implies that the relative precision (the coefficients of variation) will tend to be inversely proportional to the metal concentration measured (Table 2). When these coefficients of variation are plotted against the metal concentrations on a log-scaled abscissa (Fig. 1), it can be seen that both for repeatability and reproducibility the coefficients of variation increase approximately linearly with decreasing logarithms of metal concentration. This finding is in agreement with that published recently by Horwitz (10). According to the latter paper, for an analytical method to be acceptable, the relative reproducibility should be about 16% at the 1 mg/kg level, 23% at the 0.1 mg/kg level, and 32% at the 0.01 mg/kg level. For nickel this criterion is complied with, for copper the precision is even (much) better, whereas for iron it is not fully met, the precision being about 20% at the 1 mg/kg level and between 35 and 40% at the 0.1 mg/kg level.

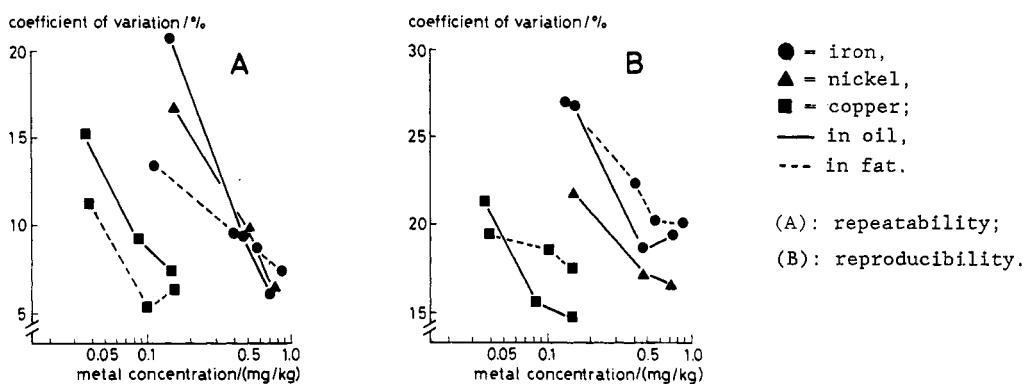


Fig. 1 Coefficient of variation as a function of the metal concentration in oil and fat.

## CONCLUSION

Direct graphite furnace atomic absorption spectrometry is a rapid and sensitive method which allows reliable determination of the total concentration of copper, iron and nickel in edible oils and fats.

After an extensive collaborative study, participants from the Commission, ISO/TC34/SC11 and many others found the method acceptable as an analytical method for the determination of trace concentrations of these metals in edible oils and fats.

On the basis of the results the Commission has decided to adopt the method. The text of the standardised procedure is given on the following pages.

## **2.631 DETERMINATION OF COPPER, IRON AND NICKEL BY DIRECT GRAPHITE FURNACE ATOMIC ABSORPTION SPECTROMETRY**

### **1. SCOPE AND FIELD OF APPLICATION**

This Standard describes a method for the determination of trace amounts ( $\mu\text{g}/\text{kg}$ ) of copper, iron and nickel in all types of crude or refined oils and fats, including contaminants from bleaching earth and/or catalysts.

### **2. PRINCIPLE**

Vaporisation of the oil/fat in a suitable graphite furnace connected to an atomic absorption spectrometer, previously calibrated using standard solutions of organocompounds of copper, iron and nickel. Measurement of the metal content from the observed absorptions at selected wavelengths. The elements are analysed sequentially.

### **3. APPARATUS**

- 3.1 Polyethylene or polypropylene-capped bottles, 20 and 50 ml, metal-free (Note 1).
- 3.2 Micropipettors - 20 and 50  $\mu\text{l}$
- 3.3 Pipettor tips.
- 3.4 Atomic absorption spectrometer. - Equipped with either "peak height" mode and printer, or "continuous" mode and pen recorder (full scale response in 0.2 s) together with appropriate hollow cathode tubes and deuterium background corrector. The spectrometer should be located in a dust-free atmosphere.
- 3.5 Graphite furnace atomiser (equipped with facilities).
- 3.6 Graphite tubes for use in a furnace atomiser (3.5).
- 3.7 Chromatographic columns (diameter/height ratio 1/10) (Note 2).
- 3.8 Electric oven - Regulated at  $60 \pm 2^\circ\text{C}$  and suitable for heating at  $150^\circ\text{C}$ .
- 3.9 Water bath.

### **4. REAGENTS**

- 4.1 n-Heptane, analytical grade.
- 4.2 Light petroleum, b.p.  $40\text{-}60^\circ\text{C}$ , analytical grade.
- 4.3 Sunflower oil, refined, or a similar, stable liquid oil with a low metal content (Note 2).
- 4.4 Aluminium oxide, chromatographic grade (Note 3).
- 4.5 Nitric acid, 2M, free from traces of iron, nickel and copper.
- 4.6 Niobium nitrate ( $\text{Nb}(\text{NO}_3)_5$ ), aqueous solution 1000 mg/l (Note 4).
- 4.7 Standard stock solutions - Stock solutions of Fe 10 mg/kg, Ni 10 mg/kg and Cu 2 mg/kg, prepared by appropriate dilution of organometallic standards with sunflower oil (4.3) (Note 5).
- 4.8 Standard working solutions. - Prepare daily the following working solutions by diluting the stock solutions (4.7) with low metal content (4.3):

Copper	0.05,	0.1,	0.2 mg/kg
Iron	0.25,	0.5,	1.0 mg/kg
Nickel	0.25,	0.5,	1.0 mg/kg
- 4.9 Argon, purity 99.99% minimum (Note 6).

### **5. PROCEDURE**

#### **5.1 Treatment of samples and solutions**

Place all samples and working solutions in the oven (3.8) during the period of determination.

Shake samples vigorously before analysis.

If the metal content of a crude oil is known to be outside the range specified for the standards in (4.8), dilute the sample with low-metal content oil (4.3) to bring the metal content within that range.

#### **5.2 Preparation of apparatus**

Switch on the atomic absorption spectrometer (3.4) and the  $\text{D}_2$  corrector. In accordance with the instructions of the manufacturer, adjust: lamp current, slit, wavelength and amplification. The required wavelengths are (nm):

Copper	324.7
Iron	302.1
Nickel	232.0

Optimize the position of the graphite furnace atomiser (3.5) in the atomic absorption spectrometer (3.4) and set the required programme for the furnace as follows:

	Step	Temp. (°C)	Ramptime (s)	Holdtime (s)	Int. gas flow (ml/min)
For Cu	1	900	50	30	300
	2	2700	1	5	50
For Fe, Ni	1	1200	50	30	300
	2	2700	1	5	50

If it is not possible to programme the graphite furnace exactly as above, use a comparative programme suitable for the equipment (Note 7).

Use a normal graphite tube (Note 8).

Pretreat the pipettor tip (3.3) by pipetting (using a micropipettor, 3.2) and then discarding 20  $\mu$ l heptane. The film of heptane remaining on the wall of the tip facilitates a reproducible transfer of the oil sample. The tip has to be pretreated before each injection of an oil sample.

### 5.3 Determination

#### 5.3.1 Graphite tube blank.

Record the absorption, if any, of the graphite tube (3.6) as such and autozero this absorption.

#### 5.3.2 Liquid oil blank.

By means of a micropipettor (3.2), inject 20  $\mu$ l low-metal oil (4.3) into the graphite furnace (3.5), initiate the temperature programme and record the absorption.

#### 5.3.3 Standardisation of apparatus.

By means of a micropipettor (3.3), inject 20  $\mu$ l of the three standard working solutions of the metal under investigation (4.8) into the graphite furnace (3.5) and record the absorption.

#### 5.3.4 Sample for analysis.

##### 5.3.4.1 Oil (liquid) samples.

By means of a micropipettor (3.2), inject 20  $\mu$ l of the oil sample into the graphite furnace (3.5), initiate the temperature programme and record the absorption.

##### 5.3.4.2 Fat samples (m.p. 40°C and higher).

By means of a micropipettor (3.2), introduce an extra temperature programming step: holdtime 20 s - temperature 60°C - internal gas flow 0 ml/min. Initiate the temperature programme. Within the first programme step introduce by means of a micropipettor (3.2), 20  $\mu$ l of the melted fat into the graphite furnace (3.5), allow the tip to remain in the injection opening to liquefy the fat and then inject. Record the absorption (Note 9).

#### 5.3.5 Number of determinations.

Carry out two determinations in rapid succession.

## 6. EXPRESSION OF RESULTS

### 6.1 Calculation

Measure the peak height on the recorder chart or read from the display or printer.

Draw a calibration curve by plotting the absorption of the three standards (5.3.3), corrected for the blank (5.3.2), against their respective metal contents (Note 10).

Read the metal content of the sample from the relevant calibration curve.

Report as the final result the mean of the results of the two determinations, provided the requirements for repeatability (6.2) are met. If the requirements for repeatability are not met, discard the results and carry out a further two determinations on the test sample.

### 6.2 Repeatability value

The differences between the values obtained from two single determinations, carried out in rapid succession by the same operator, using the same apparatus for the analysis of the same test sample, should not be greater than the repeatability value (r) as calculated from the formulae in Table 1, which express the precision in relation to the determined mean value.

Table 1 Repeatability (r) and reproducibility values (R)

Metal	Substrate	r	R
Cu	Oil	0.0102 + 0.140 m	0.0085 + 0.358 m
	Fat	0.0076 + 0.109 m	0.0028 + 0.492 m
Fe	Oil	0.077 + 0.081 m	0.040 + 0.480 m
	Fat	0.026 + 0.196 m	0.031 + 0.543 m
Ni	Oil	0.056 + 0.127 m	0.027 + 0.442 m

Key: m = corresponding mean concentration value.

### 6.3 Reproducibility value

The difference between the values for the final result, obtained by two (or more) laboratories using this standard method for the analysis of the same laboratory sample, should not be greater than the reproducibility value (R) as calculated from the formulae in Table 1 which express the precision in relation to the determined mean value.

## 7. NOTES

- The polyethylene or polypropylene-capped bottles are made metal-free in the following way:  
Clean the bottles thoroughly with warm nitric acid (4.5), rinse with distilled water and dry the bottles in a drying oven (3.8) at about 80°C.
- A sample of a low-metal content oil is obtained by the following procedure:  
Dissolve 1 part of oil (4.3) (m) in 3 parts (V) of light petroleum (4.2). Prepare an aluminium oxide column (3.7) using twice the mass of aluminium oxide (4.4), activated by heating in a oven (3.8) at 150°C for 14 h, as the mass of the oil to be purified. Add the oil solution to the column and elute with 5 parts (V) of light petroleum. Evaporate the light petroleum of the eluate on a heated water bath (3.9) using a gentle stream of nitrogen (2-5 l/min) (4.9). Remove final traces of light petroleum under vacuum.
- Merck product n<sup>o</sup> 1077 is suitable.
- Solution (code 88083) is available from Alfa Division, 152 Andover Street, Danvers, MA 01923, USA.
- Suitable standards are available from e.g. Continental Oil Company, Ponca City, Oklahoma, USA (Conostan, 5000 mg/kg) or Merck, D - 6100 Darmstadt, Federal Republic of Germany (metal in standard oil, 1000 mg/kg).
- If argon is not available, nitrogen may be used as purge gas. At temperatures above 2300°C nitrogen forms toxic cyanogen gas; therefore continuous ventilation in the furnace area should be provided.
- If in this case the background correction fails, dilute blank, standards and samples with an organic fat solvent, e.g. heptane (4.1) to a maximum of 1:2 (m/m) and work at ambient temperature.
- The graphite tube for the determination of iron has to be coated with niobium to ensure that the total amount of iron is determined. With an uncoated tube the result will vary according to the type of iron compound present in the oil. A suitable coating procedure is:  
  
By means of a micropipettor (3.2), inject 100 µl niobium nitrate solution (4.6) into the furnace. Start the temperature programme to dry at 100°C for 60 s and then atomise at 2700°C for 5 s. Repeat this procedure until 300 µl of niobium nitrate solution has been injected. Atomise at 2700°C till constant absorbance (to remove any iron contamination).

9. The normal, minimum limit of detection can be improved by either a greater scale expansion or by repeated injections of the sample at the end of the ashing operation then allowing the programme to proceed to completion. If the metal content is too high (i.e. exceeds the calibration curve) measure the absorption after a further dilution of the sample with sunflower oil.
10. With the use of sophisticated equipment auto-calibration can be applied.

### Acknowledgement

The commission is thankful to the collaborators in Australia, Austria, Belgium, Bulgaria, Denmark, Finland, France, Federal Republic of Germany, Hungary, The Netherlands, Republic of South Africa, Sweden, Turkey, United Kingdom and the United States of America for their participation and valuable cooperation.

### REFERENCES

1. A.E. King, H.L. Roschen and W.M. Irwin, Oil Soap (Chicago), 1933 10, 204.
2. R. Marcuse and P.O. Fredrikson, J. Am. Oil Chem. Soc., 1971, 48 448.
3. O. Notevarp and M.H. Chanine, J. Am. Oil Chem. Soc., 1972, 49 247.
4. W.G. Mertens, C.F. Swindells and B.F. Teasdale, J. Am. Oil Chem. Soc., 1971, 48 544.
5. G.R. List, C.D. Evans and W.F. Kwolek, J. Am. Oil Chem. Soc., 1971, 48 438
6. G.R. List, C.D. Evans, L.T. Black and T.L. Mounts, J. Am. Oil Chem. Soc., 1978, 55 275.
7. G.R. List, T.L. Mounts and A.J. Heakin, J. Am. Oil Chem. Soc., 1978, 55 280.
8. G.R. List, C.D. Evans and H.A. Moser, J. Am. Oil Chem. Soc., 1977, 49 287.
9. International Standardization Organisation, ISO 5725-1981.
10. W. Horwitz, Anal. Chem., 1982, 54 67A.