RESIDUES AND TRACE ELEMENTS

Determination of Magnesium and Calcium in Foods by Atomic Absorption Spectrometry after Microwave Digestion: NMKL[1] Collaborative Study

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On the basis of results of the performed collaborative study, the 49th Annual General Meeting of the Nordic Committee on Food Analysis (NMKL) in The Faroe Islands, August 1995, approved this method to be printed and included in NMKL's collection of methods of analysis of foods. Eleven laboratories participated in an interlaboratory methods-performance (collaborative) study of a method for determining magnesium and calcium in foodstuffs by atomic absorption spectrometry (AAS) after wet microwave digestion. The study was preceded by a practice round of familiarization samples. The method was tested on 7 materials: 5 foods (apple, milk powder, minced fish, wheat bran, and chocolate cake) and 2 composite diets ranging in Mg content from 240 to 3900 mg/kg and in Ca content from 290 to 9300 mg/kg. The materials were presented to study participants as blind duplicates, and participants were asked to perform single determinations on each sample. Repeatability relative standard deviations (RSD_r) ranged from 1.9 to 4.9% for Mg and from 2.2 to 8.1% for Ca. Reproducibility relative standard deviations (RSD_R) ranged from 4.0 to 13% for Mg and from 5.9 to 23% for Ca. For Ca, lowest RSD_R values were found for samples with high concentrations of Ca (>3800 mg/kg sample) and with nitrate ion residues of <1.3% (w/v).

Magnesium and calcium are essential nutrients involved in bone and tissue formation. Analytical food laboratories need validated methods of analysis for determining these elements in foods in general. Validated methods for determining Mg and Ca may be found in the *Official Methods of Analysis* of AOAC INTERNATIONAL (1), but none seem to have been validated for a wide range of matrixes. For example, among the more modern methods using atomic absorption spectrometry, validated methods are available for drugs, cheese, and infant formulae but none for foods in general.

Flame atomic absorption spectrometry (FAAS) is still a much used analytical technique for determining elements in biological samples. Determinations are performed in sample solutions after digestion of the food material in acids. Microwave oven, wet digestion offers an alternative to traditional closed- and open-tube sample dissolution technique. Since the first description of the use of microwave radiation as an energy source in acid digestion (2), the technique has attracted considerable attention, and several successful methods have been described (3). However, digestion conditions must be established by taking into account individual analytical problems, that is, sample type, elements to be determined, and microwave system used.

The present method has been in use for several years at the Institute of Nutrition (Bergen, Norway). The method was selected by the Nordic Committee on Food Analysis (NMKL) to be evaluated in a collaborative study (repeatability and reproducibility) using judiciously chosen foodstuffs.

Prior to the collaborative study, the method was tested and optimized with respect to factors that could affect the trueness and reproducibility of the method, such as fat or carbohydrate content of sample to be digested, digestion programs for microwave ovens, concentration of lanthanum in the sample solution, ratio of acetylene and air in the flame, and concentration of nitric acid in the sample solutions to be measured by FAA.

Collaborative Study

A description of the method and an invitation to participate in the study were sent to 13 laboratories in Denmark, Finland, Norway, and Sweden. Eleven laboratories accepted and agreed

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to follow the method procedure and the time schedule of the study. The design, conduct, and interpretation of the study followed guidelines recommended by AOAC (4) and NMKL (5). Participating laboratories were from 4 countries and represent the food industry, commercial laboratories, universities, and government laboratories.

Study Materials

Each participant received 7 test materials: wheat bran, milk powder, and minced fish (materials obtained from the National Food Agency, Copenhagen, Denmark), 2 composite diet materials (diet D and diet F; obtained from the Swedish National Food Administration, Uppsala, Sweden), dried apple, and chocolate cake (produced at the Institute of Nutrition, Bergen, Norway). The composite diets were mixtures of different proportions of a number of foodstuffs, for example, meat, liver, potatoes, milk, and flour. These 2 diets were originally produced as reference materials for determination of metals for use in an interlaboratory study of a method for determination of lead, cadmium, zinc, copper, iron, chromium, and nickel (6). The expected values of Mg and Ca in the materials used in the study were obtained by analyzing 10 replicates per material (Table 1).

The homogeneity of the test materials was investigated by estimating within-container and between-container variations of the 7 study materials. The homogeneity test was performed by taking 5 subsamples, of 5 g, from each food sample. Two replicates of 0.25 g (according to the method) from each subsample were digested. In total, 10 replicates of each food sample were analyzed for Mg and Ca. Results were analyzed by 2-way analysis of variance (ANOVA) at the 5% level. Values of $p < 0.05$ were obtained for all test materials, indicating that test samples were homogeneous.

Protocol

Before the full collaborative study, participants were given the opportunity to become familiar with the method in a pretrial test. Test materials included 2 certified reference materials: wheat flour and oyster tissue, both purchased from the U.S. National Institute of Standards and Technology (Gaithersburg, MD). The oyster tissue material contained certified concentrations of Mg and Ca in the range 1000–2000 mg/kg. The wheat flour material contained between 100 and 500 mg/kg of the 2 elements. Nine laboratories reported results for the certified reference materials. All results for Mg concentration were acceptable and fell within the certified range. For Ca, only the results for the oyster tissue material having a high Ca concentration were acceptable. Several laboratories reported very low Ca results for the wheat flour material with a lower Ca concentration. The reason for these low results was most probably due to interference from nitrate ions in less diluted sample solutions. This fact was taken into account when elaborating the final text for the full collaborative study.

The 7 test materials of the collaborative study were all dry and packed in small plastic containers. They were presented to participants as blind duplicates, that is, as 14 randomly coded materials, but the fact that blind duplicates were included in the set of materials was not disclosed to the participants. Participants were asked to perform single determinations of the Mg and Ca concentrations of the materials according to the method described below and to report results in mg/kg on a dry weight basis. Participants were also asked to give information on the microwave oven used and the temperature program used, as well as to report the absorbance values obtained for the working standard solutions. All test samples were dried and had a moisture of 2 to 10%. The participants were asked to perform dry matter determinations on the test materials and report their values on dry weight basis.

Table 1. Types of materials included in the study and their expected magnesium and calcium concentrations (average of 10 independent decompsitions per sample)

Material	Expected values, mg/kg (dry weight)	
	Mg	Ca
Wheat bran	3900 ± 156	900 ± 30
Simulated diet (D)	636 ± 12	520 ± 10
Simulated diet (F)	600 ± 6	290 ± 15
Milk powder, freeze dried	820 ± 11	9300 ± 200
Minced fish, freeze dried	739 ± 13	4000 ± 200
Apple, dried	240 ± 7	300 ± 25
Chocolate cake, dried	270 ± 5	850 ± 40

METHOD

Field of Application

The method is applicable to quantitative determination of Mg and Ca in various types of foodstuffs, with the exception of oils, fats, and extremely fatty products. The method has been tested primarily on dry products but may, under certain conditions, be used for fresh samples. High residual concentrations of nitric acid in solutions to be measured by AAS using a flame of air and acetylene interfere with the determination of Ca.

Principle

Concentrated nitric acid and hydrogen peroxide are added to the weighed sample. The sample is digested in a microwave oven. Any commercially available microwave oven for laboratory use may be used. Lanthanum(III) oxide is added to standards and sample solutions to prevent interference from phosphate ions. The concentrations of Mg and Ca are determined by FAAS. The concentrations of the elements are calculated from standard curves.

Chemicals and Reagents

(**a**) *Concentrated nitric acid (HNO_3).*—65% Suprapur.

(**b**) *Nitric acid, 0.65% (w/v).*—Dilute 10 mL concentrated nitric acid (**a**) to 1000 mL with water.

(**c**) *Hydrogen peroxide (H_2O_2).*—30%, analytical quality.

(**d**) *Deionized and possibly filtered water.*—Specific resistance, >18 MΩ/cm.

(**e**) *Lanthanum(III) oxide (La_2O_3).*—For AAS.

(**f**) *Lanthanum solution, 5% (w/v).*—Weigh 14.66 g lanthanum(III) oxide (**e**) into 250 mL beaker, moisten with 10 mL

water, and cautiously add 62.5 mL concentrated HCl (**g**). Transfer to 250 mL volumetric flask and dilute to the mark with water. The solution will keep for 1 month.

(**g**) *Concentrated HCl.*—37%, analytical quality.

(**h**) *Magnesium standard.*—1000 mg/L in for example 2.5% (v/v) nitric acid (commercial standard solution).

(**i**) *Magnesium standard solution, 10 mg/L.*—Dilute 1 mL Mg standard (**h**) to 100 mL in a volumetric flask with 0.65% nitric acid (**b**). The solution will keep for 1 month.

(**j**) *Calcium standard.*—1000 mg/L in for example 2.5% (v/v) nitric acid (commercial standard solution).

(**k**) *Calcium standard solution, 100 mg/L.*—Dilute 10 mL Ca standard (**j**) to 100 mL in a volumetric flask with 0.65% nitric acid (**b**). The solution will keep for 1 month.

Apparatus, Equipment, and Gases

(**a**) *Analytical balance.*

(**b**) *Microwave oven.*—CEM microwave sample preparation system MDS-81D (maximum initial effect, 750 W) with capping station, rotational table, and 120 mL digestion containers capable of withstanding a pressure of 830 kPa.

(**c**) *Dispenser.*

(**d**) *Glassware.*—25, 50, 100, and 250 mL volumetric flasks; 250 mL beakers; 10 and 100 mL measuring cylinders.

(**e**) *Polyethylene tubes.*—10 mL.

(**f**) *Polyethylene tubes with screw caps.*—15 and 50 mL.

(**g**) *Automatic pipets with tips.*

(**h**) *Atomic absorption spectrometer.*

(**i**) *Computer calculation program.*—AA WinLab, Instrument Control Software (Perkin-Elmer Corporation).

(**j**) *Acetylene.*—Welding quality.

(**k**) *Air.*

Procedure

(**a**) *Preparation of test sample.*—The procedure for determining dry matter content involves freeze-drying and thermal drying at 105°C for 12 h until constant weight.

Weigh into the digestion container an amount of homogeneous sample corresponding to 0.2–0.25 g dry material. Each digestion series must contain 2 reagent blanks, that is, acids only without sample materials. If possible, include certified reference materials containing Mg and Ca in amounts corresponding to those found in samples to reveal systematic or random errors.

Note: The following 2 sections should be regarded as examples. Digestion programs and amounts of acids will vary with different digestion systems.

(**b**) *Digestion.*—Add 4 mL concentrated nitric acid (**a**) to each container. Seal the containers in the capping station. Place the carousel with the digestion containers in the microwave oven and start the program: S1, 250 W, 1.00 min; S2, 0 W, 1.00 min; S3, 250 W, 5.00 min; S4, 400 W, 5.00 min; and S5, 650 W, 5.00 min.

Open the cooled containers and add 0.5 mL H_2O_2 (**c**). Recap the containers, return them to the oven and start the next program: S1, 650 W, 1.00 min, and S2, 0 W, 20.00 min.

Note: In this example, H_2O_2 is added in a separate step, but the peroxide may also be added together with the nitric acid without affecting the accuracy of the determination.

Open the cooled containers and rinse down with water any condensed water in the cap and on the walls. Quantitatively transfer the sample solution to a 25 mL volumetric flask and dilute to the mark with water. Then transfer the sample solution to a polyethylene tube (**f**).

(**c**) *Dilution.*—Take out a suitable volume, add 5% La solution (**f**), and dilute this volume with 0.65% (w/v) nitric acid (**b**) so that the final concentration of Mg and/or Ca will be within the linear range of measurement of the metals. In this example, the following ranges are selected for the standard curves: for Mg, 0.05–0.4 mg/L, and for Ca, 0.5–4.0 mg/L. The lowest point may be lower if required by the concentrations of the sample solutions. Add 5% La solution (**f**) to a final concentration of La of 1% (e.g., 2 mL 5% La solution is diluted to 10 mL).

(**d**) *Preparation of working standard solutions.*—Prepare working standard solutions for Mg of 0.05, 0.1, 0.2, and 0.4 mg/L from the Mg standard solution (**i**): Add 0.25, 0.5, 1.0, and 2.0 mL to separate 50 mL volumetric flasks, add 10 mL 5% La solution (**f**), and dilute to the mark with 0.65% (w/v) nitric acid (**b**). Prepare fresh solutions daily.

Prepare working standard solutions for Ca of 0.5, 1.0, 2.0, and 4.0 mg/L from the Ca standard solution (**k**): Add 0.25, 0.5, 1.0, and 2.0 mL to separate 50 mL volumetric flasks, add 10 mL 5% La solution (**f**), and dilute to the mark with 0.65% (w/v) nitric acid (**b**). Prepare fresh solutions daily.

Note: Check that the residual concentration of nitric acid in the sample solution has no effect on the determination of Ca. Any interference can be eliminated in the following alternative ways: (*1*) Prepare a standard curve containing the same residual concentration of nitric acid as the sample solution, or (*2*) use the standard addition method. Prepare a zero solution for the standard curves by measuring 2 mL 5% La solution (**f**) into a 10 mL volumetric flask and dilute to the mark with 0.65% (w/v) nitric acid (**b**).

(**e**) *Determination.*—Connect the Mg and/or the Ca lamp and switch on the AAS instrument. Retrieve the stored program for Mg and/or Ca and adjust to the correct wavelength and slit. Allow the instrument to warm up for ca 30 min. Readjust the position of the lamp and the wavelength by using setup to maximum energy. Light the flame and adjust the instrument to zero against water. Measure the sample series (including the reagent blank), the working standard solutions, and the zero solution. Measure the standard and blanks first, last, and after every 15 sample solutions.

(**f**) *Calculations.*—Calculate standard curves for Mg and Ca by simple linear regression (method of least squares) on the basis of the data obtained from AAS measurements. Use the standard curves to calculate the amounts of the elements in reagent blanks and sample solutions as follows:

$$m_{bl} = A_{bl} \times k \times V_0 \quad (1)$$

where m_{bl} = amount of element in the reagent blank solution V_0 (μg), A_{bl} = absorbance of the reagent blank solution (milliabsorbance units), k = slope of the standard curve (μg/mL per

Table 3. Results of the collaborative study for determination of Ca in foods

Sample	Ca, mg/kg, determined by indicated laboratory										
	1	2	3	4	5	6	7	8	9	10	11
Wheat bran	909	941	532	764	951	625	909	658	952	832	969
	913	970	476	741	1023	727	918	686	973	839	813
Simulated diet (D)	513	508	312	482	532	310	512	307	491	474	621
	522	539	270	451	511	351	524	303	528	452	572
Simulated diet (F)	254	275	207	291	278	176[a]	275	196	258	214	250[a]
	276	270	176	305	280	261[a]	262	210	249	212	423[a]
Milk powder, freeze dried	9460	9610	9450	8580	9340	9500	10100	10500	9460	9270	10500
	9280	9140	8560	8250	9090	9490	9070	10100	9440	9310	10300
Minced fish, freeze dried	4050	4250	3590	3440	3710	3850	3760	4010	3790	3880	4140
	3750	4200	3500	3660	3620	3800	3640	3970	3770	3890	4080
Apple, dried	334	256	154	273	260	152	229	201	254	274	244
	271	263	140	263	311	141	231	210	255	259	256
Chocolate cake, dried	983	1020	660	700	887	777	817	610	848	829	981
	881	1190	532	665	933	773	936	617	921	839	1050

[a] Outliers indicated by the Cochran test at $p < 0.01$.

milliabsorbance units), and V_0 = total volume of the reagent blank solution (mL).

$$m_{pr} = A_{pr} \times k \times V_1 \times f \quad (2)$$

where m_{pr} = amount of element in sample solution V_1 (μg), A_{pr} = absorbance of the sample solution (milliabsorbance units), k = slope of the standard curve (μg/mL per milliabsorbance units), V_1 = total volume of sample solution (mL), and f = dilution factor (further dilution of the sample).

Calculate the concentration of Mg and Ca in the sample from

$$c = (m_{pr} - m_{bl})/w \quad (3)$$

where c = concentration in sample (μg/g) and w = weighed amount of sample (g).

Results and Discussion

One of the critical factors studied was the effect of residual nitric acid on the Ca signal in FAAS. Standard curves of Ca constructed with 8% (v/v) or 5.2% (w/v) nitric acid showed an approximately 30% decrease in the slope of the curve compared with the slope of a curve constructed with 1% (v/v) nitric acid. A similar effect of nitric acid could be seen when a standard addition procedure was used. With our instrument settings, the suppressive effect of nitric acid started at a concentration of 2% (v/v). Users of the method are advised to apply a standard addition procedure for each dilution of the sample solution.

Results from the pretrial test for Mg and Ca in oyster tissue from 9 laboratories were 1180 ± 79 mg Mg/kg [1 SD (standard deviation)]; certified value, 1180 ± 170 mg Mg/kg (95% uncertainty); and 1964 ± 220 mg Ca/kg (1 SD); certified value, 1960 ± 190 mg Ca/kg (95% uncertainty). Results for Mg and Ca in wheat flour from 9 laboratories were 390 ± 20 mg Mg/kg (1 SD); certified value, 400 ± 20 mg Mg/kg (95% uncertainty); and 152 ± 28 mg Ca/kg (1 SD); certified value, 191 ± 4 mg Ca/kg (95% uncertainty).

Eleven laboratories reported results in the collaborative study (Tables 2 and 3). Results were statistically evaluated according to the IUPAC 1987 Protocol for the design, conduct, and interpretation of collaborative analytical studies (7). Results were tested for presence of outliers by using the Cochran and the Grubb's tests. Outliers were not included in the final estimation of method performance parameters. The following extreme (outlying) results were indicated by the Cochran test (extreme replicate results) at the $p < 0.01$ level (Tables 2 and 3): laboratory 11 results for Mg in apple and for Ca in diet material F, and laboratory 6 results for Ca in diet F.

The entire set of data indicated only one single extreme value as a result of the Grubb's test: laboratory 7 results for Mg in milk powder. Thus the set of data obtained in this study satisfies the requirement of the IUPAC 1987 protocol: The number of outliers in a collaborative study must not exceed a maximum of 2 out of 9 laboratories.

Tables 4 and 5 present estimated performance characteristics of the method.

Table 2. Results of the collaborative study for determination of Mg in foods

Sample	Mg, mg/kg, determined by indicated laboratory										
	1	2	3	4	5	6	7	8	9	10	11
Wheat bran	4230	3820	4050	4230	3610	4410	5290	4530	3750	3960	3630
	3780	3770	4120	3810	3800	4060	5570	4560	4030	4200	3370
Simulated diet (D)	660	746	642	679	624	626	686	623	642	715	649
	633	723	605	665	644	664	781	622	644	711	641
Simulated diet (F)	623	678	625	563	580	626	710	568	595	674	614
	584	681	585	635	602	605	635	574	563	662	603
Milk powder, freeze dried	822	908	881	851	848	845	1030[a]	839	808	953	862
	865	899	881	843	852	807	860	1020[a]	849	822	932
Minced fish, freeze dried	729	776	693	733	716	763	855	734	709	802	719
	738	837	710	722	675	740	857	740	727	789	721
Apple, dried	245	258	243	268	235	246	255	253	247	262	277[b]
	234	262	241	259	235	251	255	266	235	262	231[b]
Chocolate cake, dried	267	291	272	294	268	284	295	289	270	292	236
	286	296	264	288	277	290	300	290	274	292	283

[a] Outliers indicated by the Grubb's test at $p < 0.01$.
[b] Outliers indicated by the Cochran test at $p < 0.01$.

Table 4. Statistical analysis of collaborative study data for Mg

Sample	n^a	Mean	s_r	RSD_r, %	r^b	s_R	RSD_R, %	R^c	HORRAT
Wheat bran	11	4120	190	4.7	540	530	13	1490	2.8
Simulated diet (D)	11	665	25	3.8	70	46	6.9	129	1.2
Simulated diet (F)	11	618	27	4.4	76	43	6.9	120	1.2
Milk powder, freeze dried	10	859	18	2.1	51	39	4.6	111	0.8
Minced fish, freeze dried	11	749	18	2.4	50	51	6.8	144	1.2
Apple, dried	10	251	5.3	2.1	14.8	11.3	4.5	31.6	0.73
Chocolate cake, dried	10	284	5.7	2.0	15.9	11.3	4.0	31.8	0.58

[a] Number of laboratories remaining after elimination of outliers.
[b] $r = 2.8 \times s_r$.
[c] $R = 2.8 \times s_R$.

Table 5. Statistical analysis of collaborative study data for Ca

Sample	n^a	Mean	s_r	RSD_r, %	r^b	s_R	RSD_R, %	R^c	HORRAT
Wheat bran	11	824	47.8	5.5	128	157	19	439	3.3
Simulated diet (D)	11	458	22.5	4.7	61	104	23	290	3.6
Simulated diet (F)	9	254	11.5	4.3	30	38	15	107	2.1
Milk powder, freeze dried	11	9450	245	3.6	940	600	6.3	1670	1.5
Minced fish, freeze dried	11	3830	86.2	2.4	253	227	5.9	635	1.3
Apple, dried	11	238	19.2	7.8	52	53	22	149	3.1
Chocolate cake, dried	11	839	62.8	7.3	172	166	20	465	3.4

[a] Number of laboratories remaining after elimination of outliers.
[b] $r = 2.8 \times s_r$.
[c] $R = 2.8 \times s_R$.

For determination of Mg, the relative standard deviation for repeatability (RSD_r) of the method was estimated to be between 2.0 and 4.7%. The relative standard deviation for reproducibility (RSD_R) was estimated to be between 4.0 and 13%. For determination of Ca, RSD_r was estimated to be between 2.4 and 7.8%, and RSD_R was estimated to be between 5.9 and 22%.

RSD_R values were compared with those obtained from a large number of interlaboratory method performance studies involving a wide range of analytes, matrixes, and measurement techniques. Horwitz et al. (8) found that the RSD_R value generally can be predicted from a general equation, the so-called Horwitz equation:

$$RSD_R = 2^{(1-0.5 \log C)}$$

where C is the concentration as a decimal fraction. According to Horwitz, the ratio between observed RSD_R values and the RSD_R values predicted by this equation, designated HORRAT, can be regarded as an indication of the acceptability of a method with respect to its precision. The HORRAT values of this method are presented in the last columns of Tables 4 and 5. According to Horwitz et al. (8), a series of ratios close to or consistently smaller than 1.0 indicates acceptable precision of methods. Correspondingly, HORRAT values consistently near or greater than 2 probably indicate an unacceptable method with respect to precision. The same approach has been adopted by IUPAC (9).

Table 4 shows that the present method has an acceptable precision for Mg determination. HORRAT values are below 2.0 for all test materials except wheat bran. This material had a high concentration of Mg, 4 g/kg. The required large dilution may have given rise to the poor agreement in results between laboratories.

Table 5 shows that for Ca determination, acceptable HORRAT values were obtained only for materials with the highest Ca concentrations: milk powder and minced fish. HORRAT values were above 2.0 for other test materials. The most probable reason for this is failure to correct for interference from the concentration of nitric acid in less diluted sample solutions.

Collaborators' Comments

Laboratory 1 reported that the sample solutions were calibrated against both 1% nitric acid and solutions having a nitric acid concentration corresponding to that in the sample solutions. The Ca concentrations of the sample solution were analyzed by both FAAS and ICP (inductively coupled plasma). Results from FAAS and ICP agreed in the case of milk powder and fish. In the case of other test materials, results from FAAS and ICP agreed if the FAAS system was calibrated against a standard curve constructed from standards containing the same nitric acid level as the sample solutions.

Laboratory 5 indicated that results from the determination of Ca varied on a day-to-day basis.

Conclusions

The results of the collaborative study of this AAS method for determining Mg and Ca indicate that the method is suitable for determinations of Mg in foods in the concentration range 250–1000 mg/kg dry matter and Ca in foods containing concentrations of Ca above about 4000 mg/kg dry matter. In the case of Ca, method precision could be improved by correcting for the suspected interference of nitrate ions when measurements are made with less diluted sample solutions.

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