Determination of Copper, Lead, and Nickel in Edible Oils by Plasma and Furnace Atomic Spectroscopies

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ABSTRACT: An atmospheric pressure microwave digestion procedure is described for the determination of copper, lead, and nickel in edible oils. The digestion procedure is compatible with either inductively coupled plasma atomic emission spectrometry (ICP-AES) or graphite furnace atomic absorption spectrometry (GFAAS). Instrument conditions are specified to allow the simultaneous determination of all three metals for each sample introduction. Method detection limits were element-specific but equal to or less than 50 ng/g with ICP-AES and 30 ng/g with GFAAS. Results from spike and recovery experiments at levels of 50, 100, and 200 ng/g are reported for corn and soybean oil. For soybean oil, recoveries at 100 ng/g were 90, 100, and 106%, respectively, for copper, lead, and nickel with ICP-AES, and 89, 106, and 96%, respectively, with GFAAS. Recoveries for corn oil spiked at 100 ng/g were 93, 95, and 103%, respectively, for copper, lead, and nickel with ICP-AES, and 90, 117, and 100%, respectively, with GFAAS. Day-to-day reproducibility was demonstrated by similar method recoveries and reproducibilities in independent analysis of two canola oil sample sets that were spiked at 100 ng/g.

JAOCS 75, 477–481 (1998).

KEY WORDS: Atomic absorption spectrometry, edible oils, inductively coupled plasma atomic emission spectrometry, microwave dissolution, trace metals.

The presence of metals in edible oils occurs through natural contamination and by introduction during the refining process. Specific to refining is the introduction of nickel, which is used as a hardening agent (1). Lead and copper are potentially present in oils because of environmental contamination. The presence of metals in the final, refined oil is undesirable because the metals can facilitate oxidative degradation of the oil and decrease shelf life. Additionally, the U.S. Food and Drug Administration has recently expressed interest in defining lead level exposure from edible oils because even trace levels in oils that have high consumption can result in significant exposure (2). The nonspecific target level for metal contamination is 100 ng/g. Recent reports regarding the determination of these metals in edible oils at sub-ppm levels have been based on voltammetry (3), atomic absorption (4–9), and atomic emission spectroscopies (10). Excluding graphite furnace atomic absorption spectroscopy (GFAAS), most techniques require some type of sample pretreatment prior to analysis. Sample pretreatment options include off-line ashing, direct ashing with GFAAS, solubilization, and extraction.

Basic alcoholic solubilization has been used with both voltammetry (3) and GFAAS (4). Required times for solubilization were not specified, but for voltammetry, the authors indicated that samples were left overnight prior to dilution and analysis. In both reports, quantitation required the method of standard addition. In combination with voltammetry, solubilization was demonstrated for cadmium, copper, and lead (3). However, deviations of 20% were reported for both copper and lead in control samples when solubilization was compared to high-pressure ashing (3). With GFAAS, solubilization was adequate for the determination of cadmium, chromium, cobalt, copper, iron, lead, manganese, nickel, and tin on an individual element basis (4). Recoveries varied from element to element but were generally 90% or better for spiked levels in excess of 100 ng/g, and the relative standard deviations for six replicates were under 10%. Method detection limits were element-specific but were under 30 ng/g.

Acid extraction was evaluated as a means of isolating copper and iron from the oil matrix with GFAAS detection (5). Single extractions produced efficiencies of 60% with nitric acid and 70% with a combination of nitric acid and hydrogen peroxide. The mixing step required 24 h before isolation of the aqueous fraction, and analysis of the extract produced levels for control samples that were 2 to 10 times greater than values obtained with oxygen ashing.

The only recent report pertaining to complexation involved ethylene diamine tetraacetic acid (EDTA) and was lead-specific (10). Recoveries ranged from 93 to 98% for spike levels of 200 ng/g to 10 mg/g, but no indication of measurement precision was reported. Lead levels were determined by direct current plasma atomic emission spectrometry (DCP-AES), which limited results to levels greater than 80 ng/g. Extension of this approach to other metals was not reported but should be possible although metal-specific. Because equilibration for lead required 2 h, extension to other metals would also be time-intensive.

Dilution with methyl isobutyl ketone (MIBK), followed by direct analysis, has been demonstrated with GFAAS (5,6).

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Sensitivity differences between standards, prepared in oil and MIBK, were problematic for the determination of aluminum, chromium, iron, and nickel with wall atomization but were not a factor with platform atomization or for the determination of copper and lead (6). The dilution level tested was 1:10 (wt/vol), and for higher oil percentages or lower sample dilution, sensitivity differences may be more problematic. Furnace procedures were element-specific, but detection limits were less than 15 ng/g.

Methods recommended by the Association of Official Analytical Chemists, the American Oil Chemists' Society, and the International Union of Pure and Applied Chemistry are based on direct analysis with GFAAS and limited to copper, iron, nickel, and lead (7–9). For copper, iron, and nickel, samples are analyzed by direct deposition in the cuvette, whereas the determination of lead requires that samples be mixed with a lecithin-based matrix modifier prior to analysis. With the copper, iron, nickel procedure, differences in optimal pyrolysis and atomization temperatures only allow for simultaneous detection of iron and nickel, although the furnace cuvette must be pretreated with niobium prior to the determination of iron. For all procedures, matrix matching between standards and samples is required, which necessitates purification of an oil for standard preparation. The lowest levels evaluated by spike and recovery experiments were 35 ng/g for copper, 20 ng/g for lead, and 150 ng/g for both nickel and iron (7–9).

In this report, we describe an atmospheric pressure microwave dissolution procedure for edible oils that is compatible with both inductively coupled plasma atomic emission spectroscopy (ICP-AES) and GFAAS for the determination of copper, lead, and nickel by using external calibration with aqueous standards. Simultaneous or rapid sequential analysis is reported for both instrumental techniques, representing all three elements per sample introduction. The method is evaluated by spike and recovery experiments at 50, 100, and 200 ng/g in corn and soybean oil. Spike and recovery experiments at 100 ng/g are also included with canola oil, and day-to-day reproducibility is demonstrated by the independent analysis of two sets of canola oil samples. The extension of this method to other elements is also discussed.

EXPERIMENTAL PROCEDURES

Samples and spiking procedures. The oils used for the spike and recovery experiments were obtained from a local grocery and represent the oils after refining. A spiked stock standard was gravimetrically prepared for each oil by utilizing a multielement organometallic standard (Standard S-12, Conostan, Ponca City, OK). Spiked subsamples were then gravimetrically prepared in the oil of interest with the appropriate spiked stock standard. The oils and the standard were miscible, so both the stock standards and subsamples were homogeneous. Sample spikes were prepared in triplicate at 50, 100, and 200 ng/g for the corn and soybean oils, and two separate triplicate sets, spiked at 100 ng/g, were prepared for canola oil. Samples were stored in high-density polyethylene (HDPE) bottles at room temperature.

Reagents and glassware. All glassware was scrupulously cleaned and acid-soaked prior to use. National Institute for Standards and Technology (Washington, D.C.)-certified standards were used to prepare the aqueous stock solutions which were then utilized to prepare working standards. Trace-metal-grade nitric acid (HNO_3) and sulfuric acid (H_2SO_4) and reagent grade 30% (wt/vol) hydrogen peroxide (H_2O_2) were used for digestion. The ammonium phosphate used for matrix modification was also reagent grade. Unless otherwise indicated, all other reagent percentages reflect volume-to-volume dilutions.

Sample digestion. The sample size used for digestion was 5 mL, and this was volumetrically transferred to the digestion vessels. For the oils used in this work, the 5-mL aliquot was gravimetrically determined to represent 4.55 g of oil. Each sample was pre-reacted with 1.5 mL of 18 M H_2SO_4 for 15 min, followed by two 2-mL additions of 16 M HNO_3 at 10-min intervals. Samples were then allowed to stand for an additional 20 min prior to placing in the microwave cavity.

Microwave digestion was accomplished with the Star System 6 Atmospheric Pressure Microwave System (CEM Corporation, Matthews, NC), equipped with air-cooled condensers, automated reagent addition, and a vapor removal system. No more than two samples were digested at one time, which allowed the program to proceed in a continuous fashion and avoided overloading of the vapor removal system.

The microwave program is summarized in Table 1. In brief, the program used for digestion consisted of five temperature stages. The first two stages were reached slowly to allow solubilization and preliminary digestion to occur without excessive foaming. The remaining stages employed shorter ramp times but longer hold times for reagent addition. Throughout this program, automated addition was performed such that the total reagent volume for any stage was added at equal time intervals by using the specified aliquot volume, vs. complete addition at the start of the stage. For example, a total volume of 5.0 mL with a specified aliquot volume of 1.0 mL and a hold time of 5.0 min would cause the system to dispense 1.0 mL of the reagent every min, so that at the end of 5.0 min, a total volume of 5.0 mL would have been dispensed.

After digestion, the condenser and sides of the digestion flask were rinsed with 1% HNO_3. The digests were then transferred to 50-mL volumetric flasks and diluted to volume with 1% HNO_3. Reagent blanks were prepared similarly to samples except that all of the H_2O_2 was added at 130°C, vs. sequential additions at 135 and 145°C, in order to reduce temperature cycling. Within 24 h, samples were transferred to HDPE bottles for storage prior to analysis. Samples were directly analyzed by ICP-AES, but aliquots were further diluted with 1.1% (wt/v) $(NH_4)_2HPO_4$ to make a modified sample matrix of 0.1% $(NH_4)_2HPO_4$ prior to determination by GFAAS.

ICP-AES. Analyses were conducted with a Thermo Jarrell Ash Polyscan 61E (Franklin, MA), equipped with an ultrasonic nebulizer (5000AT, CETAC, Omaha, NE). The spray chamber and condenser temperatures for the nebulizer were

TABLE 1
Microwave Digestion Program

Initial addition: 16 mL HNO_3, in 8-mL aliquots					
Ramp time (min)	Temperature (°C)	Hold time (min)	Reagent	Aliquot volume (mL)	Total volume (mL)
10	105	3.0			
5	125	5.0	HNO_3	1.0	5.0
1	130	5.0	HNO_3	1.0	5.0
1	135	7.5	H_2O_2	1.0	15.0
1	145	10.0	H_2O_2	1.0	20.0

140° and 5°C, respectively. The plasma, auxiliary, and carrier gas flow rates used were 14, 1.0, and 0.9 L/min, respectively. The applied power was 1.15 kW, and the monitored plasma zone was 14 mm above the upper load coil. The liquid flow rate to the nebulizer ranged from 1.4 to 1.6 mL/min and was controlled by a Gilson (Middleton, WI) Minipuls 2 peristaltic pump. Analysis timing and switching were controlled by an autosampler (TJA 300; Thermo Jarrell Ash). The analyte lines used were 324.7 nm for copper, 220.3 nm for lead, and 231.6 nm for nickel. All lines were on the polychromator, and background correction was employed by using a point located + 0.14 nm from the main line for reference. The integration time used was 5 s.

GFAAS. The furnace system used was the AA Scan 4 (Thermo Jarrell Ash). All elements were determined in a simultaneous fashion during each furnace cycle, which refers to a rapid sequential scanning of the wavelengths. Hollow cathode lamp line sources were employed to operate at conditions that facilitated Smith-Hieftje background correction. Lamp currents and photomultiplier tube voltages were optimized according to manufacturer's directions. The wavelengths used during the analyses were 324.7 nm for copper, 283.3 nm for lead, and 232.0 nm for nickel. The spectral bandpass was set to 0.40 nm. Peak area was used for integration at a time interval of 6 s during the atomization cycle. Samples were introduced by aerosol deposition with a 15-s deposit time at an uptake rate of 1.3 to 1.5 mL/min. The cuvettes used were of the delay atomization design and were pyrolytically coated. Sample drying occurred at a temperature of 150°C, which was obtained by a 2-s ramp, held for 3 s and purged with argon at a low setting (1–2 L/min). The first pyrolysis temperature was 500°C, which was reached in 10 s, held for 5 s, and purged with air at a medium rate of 3–4 L/min. The second pyrolysis temperature was also 500°C and was maintained for 20 s with a medium purge of argon. The furnace was then stepped to 1900°C for atomization. The hold for atomization was 4 s, and the purge gases were off. The furnace was then cleaned by stepping to 2200°C, holding for 1 s, and purging at medium flow with argon.

Calibration and numerical protocol. For both ICP-AES and GFAAS, quantitation was accomplished by external calibration with aqueous standards, prepared in a 3% H_2SO_4 and 1% HNO_3 matrix. Four exposures were used to define the average total signal, and background noise was determined from 20 replicates of the appropriate blank. All calibration curves were initially based on five standards, including the blank, and the concentrations used ranged from 4 ng/mL to 50 ng/mL. Calibration curves were modified as necessary to provide optimal performance through the range of interest. Instrument detection limits were calculated based on three times the standard deviation of the blank standard, and quantitation limits were based on 10 times the standard deviation of the blank standard. Method detection and quantitation limits were

TABLE 2
Percentage Recoveries and Standard Deviations for Spiked Soybean

A. Analysis by ICP-AES			
	Spike concentration[a]		
Element	50 ng/g	100 ng/g	200 ng/g
Copper	84 (12)	90(*)[b]	95 (2.6)
Lead	107 (9.8)	100 (3.4)	101 (8.1)
Nickel	104 (11)	106 (9.3)	107 (3.9)
B. Analysis by GFAAS			
	Spike concentration		
Element	50 ng/g	100 ng/g	200 ng/g
Copper	92 (5.5)	89 (*)	94 (4.5)
Lead	86 (3.4)	106 (16)	106 (12)
Nickel	116 (8.7)	96 (8.1)	102 (4.5)

[a]The number in parentheses represents the percentage relative standard deviation for triplicate samples.
[b]An asterisk (*) denotes results based on duplicate samples. ICP-AES, inductively coupled plasma atomic emission spectrometry; GFAAS, graphite furnace atomic absorption spectrometry.

TABLE 3
Percentage Recoveries and Standard Deviations for Spiked Corn Oil

A. Analysis by ICP-AES			
	Spike concentration[a]		
Element	50 ng/g	100 ng/g	200 ng/g
Copper	89 (15)	93 (11)	97 (3.5)
Lead	75 (7.3)	95 (7.5)	95 (3.6)
Nickel	108 (15)	103 (7.4)	101 (5.1)
B. Analysis by GFAAS			
	Spike concentration		
Element	50 ng/g	100 ng/g	200 ng/g
Copper	78 (9.5)	90 (7.9)	95 (9.2)
Lead	98 (21)	117 (*)[b]	88 (6.3)
Nickel	95 (10)	100 (9.1)	107 (6.2)

[a]The number in parentheses represents the percentage relative standard deviation for triplicate samples.
[b]An asterisk (*) denotes results based on duplicate samples. For abbreviations see Table 2.

calculated relative to the initial sample size and corrected for any dilutions.

RESULTS AND DISCUSSION

Results from spike and recovery experiments with the soybean and corn oils are summarized in Tables 2 and 3, respectively. In both tables, part A represents ICP-AES detection and part B represents GFAAS. The first value reported represents the average recovery for triplicate samples, and the number in parentheses is the percentage relative standard deviation.

In general, recoveries for copper at 100 and 200 ng/g were adequate, at or above 90%. At 50 ng/g, the recoveries were lower and ranged from 78 to 92%. There was no clear distinction in the recoveries with respect to the detection technique. Method detection limits for copper were lower with GFAAS, 5 ng/g, than with ICP-AES, 20 ng/g. The relative standard deviations increased to 15% at 50 ng/g but were generally lower at the higher spike concentrations. Similar to the recoveries, there was no clear difference in the deviations between GFAAS and ICP-AES. The behavior of the relative standard deviations in addition to the quantitation limits suggests that the variability in recoveries at 50 ng/g may reflect detectability vs. imprecision in the digestion procedure. Also, the detected outlier at 100 ng/g for the soybean oil was the same for both ICP-AES and GFAAS. Copper was detected in the unspiked corn oil at an average 8 ng/g but was not detected in the soybean oil or reagent blanks.

Lead recoveries were also acceptable at the higher spike levels, 100 and 200 ng/g. Specifically, recoveries at 100 and 200 ng/g ranged from 95 to 101% with ICP-AES and from 88 to 117% with GFAAS. At 50 ng/g, recoveries for ICP-AES extended outside the range of the higher spikes, at 75 and 107%, whereas the recoveries for GFAAS at 50 ng/g were not significantly different. Method detection limits were near 50 ng/g for ICP-AES and ranged between 25 and 50 ng/g for GFAAS. The higher method detection limits may account for the poor performance at 50 ng/g. Relative standard deviations were generally greater for lead than for copper, which also reflects the higher method limits. Standard deviations were generally lower with ICP-AES than with GFAAS, which may be attributed to the increased compromise in using a multielement procedure with GFAAS vs. ICP-AES. Lead was detected in both oils at 30 ng/g by GFAAS but was not detected in the reagent blanks. Further improvement in method performance, specifically quantitating the blank levels, could be achieved by increasing the initial sample aliquot. Nonetheless, the current procedure adequately surveys for lead levels of 100 ng/g or higher.

Recoveries for nickel ranged from 96 to 116%, and again, variability at 50 ng/g was encountered. There was no significant difference in method performance with respect to detection. Method detection limits were lower with GFAAS, 8 ng/g, than with ICP-AES, 30 ng/g. Relative standard deviations ranged from 15% at 50 ng/g to 5% at 200 ng/g. Reagent blanks averaged 2 ng/mL by GFAAS, but neither the corn oil or the soybean oil had a measurable blank level after reagent subtraction.

TABLE 4
Day-to-Day Reproducibility Based on Canola Oil Spiked at 100 ng/g

	ICP-AES[a]		GFAAS[a]	
Element	Set 1	Set 2	Set 1	Set 2
Copper	96 (8.0)	92 (2.4)	89 (14)	86 (8.3)
Lead	100 (12)	91 (6.0)	103 (15)	78 (8.0)
Nickel	89 (4.3)	91 (3.7)	98 (6.2)	95 (2.2)

[a]The number in parentheses represents the percentage relative standard deviation for triplicate samples. For abbreviations see Table 2.

Canola oil was also spiked at 100 ng/g, and recoveries were determined on two separate sample sets as a means of defining day-to-day reproducibility. Sample sets were prepared approximately 60 d apart, and the results are summarized in Table 4. With ICP-AES detection, recoveries for both sets were at or above 90% with relative standard deviations at or below 10%. With GFAAS, similar recoveries were obtained for copper and nickel; however, the recoveries for lead varied dramatically, from 78 to 103%. It is likely that the lower lead recovery for set 2 with GFAAS is related to aging of the furnace cuvette. This is proposed because, during analysis of the second sample set, the standard deviation for a 10 ng/mL external standard increased from 5 to 20%. Nonetheless, one cuvette adequately analyzed both the corn and soybean oil samples, representing over 250 furnace cycles. The standard deviations for set 2 were consistently lower than for set 1, which is attributed to improved reproducibility in the digestion procedure after the reagent valve in the addition unit failed and was replaced shortly after completing set 1.

In this report, element-to-element deviations were minimal, suggesting that digestion was complete. Differences in performance between GFAAS and ICP-AES were also minimal and further supported the concept that sample digestion was complete. Method detection limits were consistently lower with GFAAS than with ICP-AES, which would be anticipated on a per-element basis but is somewhat surprising for the multielement approach. With this method, lower limits should be possible by increasing the size of the sample aliquot, with corresponding adjustments to the reagent volumes used for digestion. The increased reagent consumption could possibly be counteracted by application of water-cooled vs. air-cooled condensers. With GFAAS detection, even lower limits could be realized by developing element-specific furnace programs.

By assuming complete digestion, the direct application of this procedure to other metals should also be possible. The procedure would be limited to nonvolatile metals that would not be lost during digestion, and detection protocols would need to be checked for optimal performance. Iron was also of interest in this report, but efforts were abandoned because of excessive environmental contamination from the fume hood used for sample transfer. Iron contamination was also prevalent in the hydrogen peroxide, so that the extension of this

approach to iron may require purification of commercially available hydrogen peroxide or selection of another oxidizing agent.

With respect to the digestion procedure, attempts at digestion in absence of sulfuric acid proved to be reagent-intensive and caused excessive temperature cycling. Smaller volumes of sulfuric acid (<1.5 mL) did not provide sufficient initial charring to allow for easy dissolution during the early stages or provide a sufficient volume for temperature control during the latter stages. Larger volumes (5.0 mL) would have facilitated digestion but would have required reduction/evaporation prior to analysis. Additionally, larger volumes of sulfuric acid could have been problematic, owing to the formation of lead sulfate. Pre-reaction outside of the microwave cavity minimized residue buildup along the sides of the vessel and minimized foaming during dissolution. Similarly, the initial ramp of 10 min to 105°C was determined to be necessary, as shorter times resulted in excessive foaming and sample loss through the vapor removal system. Procedurally, the addition of hydrogen peroxide occurred every 30 s, and the unit required approximately 10 s per addition so that only three samples could be digested at one time. However, after digestion of two samples it was necessary to replenish the sodium hydroxide in the acid vapor removal system. These limitations could be addressed simply by the addition of a second reagent pump and by increasing the neutralizing capacity of the vapor removal system.

As noted in the experimental procedure, spiked samples were prepared by using an organometallic standard that was miscible in the oil matrix. The introduction of the spike in an aqueous solvent inhibited pre-reaction or charring by the sulfuric acid and favored the formation of separate layers (organic and aqueous). Similarly, simultaneous introduction of sulfuric and nitric acid caused the formation of two layers vs. reducing the sample matrix.

Samples were volumetrically transferred to the digestion flasks. However, gravimetric transfer could also be employed. Volumetric transfers were highly reproducible and relatively rapid. Volumetric transfer was chosen over gravimetric transfer solely because of the weight tolerances of the available analytical balances.

In comparison to the other methods of sample preparation, atmospheric microwave dissolution vs. solubilization and extraction was more rapid and less labor-intensive in this instance because of automated reagent addition. Because the digests were clear, other means of detection, inductively coupled plasma mass spectrometry and flame atomic absorption spectrometry, should be acceptable, and the method should be directly extendable to other nonvolatile metals. With respect to direct analysis or dilution with MIBK, followed by GFAAS, digestion allows simultaneous determination of multiple metals and avoids limitations due to the introduction of a carbonaceous matrix. As a result, this method is a viable alternative to other procedures and offers the advantage of direct extension to other nonvolatile metals.

ACKNOWLEDGMENTS

This research was supported by an appointment to the Postgraduate Research Program at the National Center for Toxicological Research administered by the Oak Ridge Institute for Science and Education through an interagency agreement between the U.S. Department of Energy and the U.S. Food and Drug Administration.

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[Received May 5, 1997; accepted October 28, 1997]