Evaluation of a Graphite Furnace Atomic Absorption Method Developed for the Determination of Lead in Sugars

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A graphite furnace atomic absorption spectrometry (GFAAS) method was collaboratively studied to see if it would provide reliable performance in laboratories with a wide range of GFAAS expertise. Reliable detection of lead at levels of 100 ng/g was essential, and the method exceeded this requirement and provided quantification at levels of 30–50 ng/g with good contamination control. The intralaboratory precision was better than 10% RSD, and the interlaboratory precision was better than 20% RSD for samples with Pb at levels of 100 ng/g or greater. Problems with contamination are highlighted, and the maturity of GFAAS as a routine analytical technique is discussed.

Keywords: *Graphite furnace atomic absorption; platform atomization; collaborative study; lead in sugars*

INTRODUCTION

Exposure to lead is a public health concern. Recently, there has been renewed concern about the possible negative health impact in individuals exposed to levels of lead that were previously thought to be safe (Baghurst et al., 1992; Centers for Disease Control, 1991). In the United States, the Centers for Disease Control recently lowered the concentration of lead in blood considered to be harmful to young children from 25 to 10 μg/dL (Centers for Disease Control, 1991). Because food is a potential source of lead, there is significant interest in lowering the acceptable lead limits of foods that are frequently consumed (Bigelow, 1992). One such class of foods comprise sugars and syrups which are used commercially for the preparation of many beverages and processed foods, as well as being used in the home for baking and sweetening of selected foods and beverages. The Food Chemicals Codex (FCC) Committee sets the limits for lead and other heavy metals. The limits for lead in sugars established in 1991 were 0.1 μg/g for dextrose and fructose and 0.5 μg/g for glucose syrup, sucrose, and high fructose corn syrup (*Food Chemicals Codex*, 1992). Those limits were set on the basis of the availability of suitable methodology to provide sufficiently reliable, sensitive results. The official recommended FCC method (*Food Chemicals Codex*, 1992) was a graphite furnace method that used antiquated technology and which did not provide reliable performance because it employed in situ tungsten coating of the graphite tube. That method was collaboratively studied in 1992 by the National Food Processors Association (NFPA) and the International Life Sciences Institute (ILSI) and provided poor detection capability (detection limit 0.1 μg/g), poor sensitivity (0.1–0.2 μg/g), low average recoveries (86%), and poor average intralaboratory precision (26%) and interlaboratory precision (42%) (International Life Sciences Institute, 1993). ILSI, through its Food, Nutrition and Safety Committee's Subcommittee on Trace Minerals in Foods, initiated efforts to evaluate the state of current methodology and explore alternatives to identify a method that would be sufficiently reliable, transportable, sensitive, accurate, and precise to allow the detection of lead at or below the specified limit levels in sugars and syrups. The Subcommittee was aware of a method developed in the U.S. Department of Agriculture's Food Composition Laboratory which is part of the Beltsville Human Nutrition Research Center located in Beltsville, MD (Miller-Ihli, 1994), and requested the author to be involved in validation of her method through a collaborative study.

The results of the collaborative study are contained herein. Data were analyzed using the guidelines outlined in the Statistical Manual of the Association of Official Analytical Chemists (AOAC) (Youden and Steiner, 1975) since this method will be submitted for consideration as an AOAC Official Method. Spiked (up to 0.5 μg/g of Pb) and unspiked sucrose and high fructose corn syrup (HFCS) samples were sent to 12 laboratories and 8 returned data. The intralaboratory precision was 9.6%, the interlaboratory precision was 21%, and the average recovery was 115% over the range of all samples. As a result of this collaborative study, the Subcommittee recommended that the FCC Committee replace the existing method (*Food Chemicals Codex*, 1992) with this method (Miller-Ihli, 1994) since it provides better detection capability and precision. This recommendation was recently adopted (*Food Chemicals Codex*, 1995).

EXPERIMENTAL PROCEDURES

Study Protocol. The study was done in two phases. During the first phase, participants were asked to review the method, make comments, complete a worksheet detailing the equipment available to them, run a calibration curve, report several analytical figures of merit, and analyze an unknown water sample. The second phase involved the digestion and analysis of several sucrose and high fructose corn syrup (HCFS) samples. The sample study design was done by Jonathan DeVries of General Mills, who prepared the test samples. The design was based on a Youden matched pair design (Youden and Steiner, 1994) for collaborative evaluation of overall precision, single analyst precision, and mean recovery of the analytical method. Participants received a total of 16 samples (see Table 1). The spiked sucrose and HFCS samples were prepared by Jonathan DeVries, and the homogeneity and spiking accuracy were verified in the author's laboratory (Miller-Ihli, 1994). Unspiked "as is" blank samples

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Table 1. Spike Concentrations of Sugar Samples Analyzed

material	added spike (ng/g of sucrose)	material	added spike (ng/g of HFCS)
sucrose E	unspiked	fructose I	unspiked
sucrose F	32.2	fructose J	31.8
sucrose C	53.7	fructose H	44.8
sucrose A1–A3	105	fructose K1–K3	93.3
sucrose D	214	fructose M	151
sucrose B	508	fructose G	454

of sucrose (E) and HFCS (I) were included but, because of the low levels, many laboratories could not accurately quantify the lead levels. As a result, well-characterized values obtained by FCL were used for blank correction before spike recoveries were calculated.

Sample Digestion. The detailed description of the wet ashing procedure has been previously reported (Miller-Ihli, 1994). Briefly, the method recommends the triplicate digestion of samples using a wet ash nitric acid and hydrogen peroxide procedure. To ensure that a representative sample of crystalline sucrose is used for analysis, the method recommends the preparation of a sucrose solution using equal masses of sucrose and deionized distilled (18 MΩ) water. Approximately 1.5 g of sample (3.0 g of sucrose solution) is heated with 0.75 mL of sub-boiling distilled nitric acid and treated with two 0.5 mL aliquots of hydrogen peroxide added dropwise. Samples are diluted to a final volume of 10 mL.

GFAAS Method. The detailed description of the method has been given previously (Miller-Ihli, 1994). Briefly, the method was designed for use with a wide range of modern graphite furnace atomic absorption spectrometry (GFAAS) instruments. The method calls for platform atomization, magnesium nitrate as a modifier (0.06 mg), oxygen ashing, peak area measurements, and calibration against aqueous standards. The method requires a spectrometer equipped with adequate background correction, an autosampler, a lead hollow cathode lamp or electrodeless discharge lamp, two gas flows to facilitate air ashing, and a well-defined calibration function. Analyses are performed at the 283.3 nm line using a 0.7 nm slit. The method was developed on a Perkin-Elmer Model Z5100 equipped with Zeeman background correction, an HGA-600 graphite furnace, and an AS-60 autosampler (Perkin-Elmer Corp., Norwalk, CT). The instrumental detection limit (based on 3.29σ) was 10 pg, or 0.5 μg/L for a 20 μL injection, corresponding to a method detection limit of 3.3 ng/g of sugar.

The method requires that the sensitivity be checked prior to running a calibration curve and recommends the use of 20 μL of a 25 μg/L standard which should provide an integrated absorbance of approximately $0.18s$ for a standard Massman style furnace (28 mm × 6 mm), resulting in a characteristic mass of approximately 12 pg. The characterisitc mass is the amount of material needed to generate an integrated absorbance of $0.0044s$. Calibration requires the use of four standards (10, 25, 50, 100 μg/L; 20 μL aliquots). The method cautions analysts that a nonlinear calibration algorithm is required since the calibration curve for lead is typically nonlinear above 0.5 ng (20 μL of 25 μg/L). To ensure analytical accuracy, the method recommends that National Institute of Standards and Technology (NIST) Standard Reference Material (SRM) 1643c or equivalent be analyzed prior to the analysis of unknown samples. This acidified water sample has a certified Pb content of 35.3 ± 0.9 μg/L. The method requires that the determined concentration be within ±10% of the mean reference value (31.8–38.8 μg/L) prior to analysis of the unknown samples. The preparation of an in-house sucrose control solution is suggested. The in-house control solution should be spiked to produce a concentration of 100 ng/g of Pb in sucrose, recoveries should be 100 ± 20%, and the precision for replicate digestions should be less than 5% RSD.

RESULTS AND DISCUSSION

Phase 1. As previously described, the study was conducted in two phases. It was clear from a review of the information returned that some laboratories had difficulty with calibration, instrument performance, or contamination, and some had not followed the method. Those collaborators were contacted, and any significant problems were identified and resolved. Collaborators using the new Perkin-Elmer 5100ZL and 4100ZL generally found that the furnace program had to be modified to decrease the temperature settings since they found that the characteristic red glow seen at 800 °C was apparent at set temperatures as low as 550–600 °C on these systems. Some of the collaborators had never used air ashing previously but noted that it was easy to implement. During this phase, collaborators provided assurance that the exact method was being followed and that reasonable performance was obtained based on review of the sensitivity data and the accuracy data for the analysis of SRM 1643c. Pertinent data returned by 13 collaborators for phase 1 appear in Table 2.

Interestingly, most of the participants used Perkin-Elmer spectrometers and nonlinear calibration. Characteristic mass data cannot be compared without taking into account the furnace geometry, but it is interesting to note that for laboratories 5, 8, 9, and 11 which use the same furnace design, the characteristic mass data agreed well, ranging from 11.4 to 15.2 pg and were in good agreement with the manufacturer's recommended value of 12 pg. Likewise, laboratories 6, 7, and 11, which all used the smaller transversely heated furnace tubes, had characteristic mass values ranging from 37.7 to 45.3 pg, which is higher than the manufacturer's reference value of 30 pg but experience has repeatedly shown that values typically range from 38 to 48 pg (G. R. Carnrick, Perkin-Elmer Corp., personal communication, 1994). The characteristic mass data for laboratory 13 suggest that perhaps the collaborator used a 10 μL sample volume rather than the 20 μL sample volume reported, since the sensitivity is more than twice as poor as expected.

Data for SRM 1643c are in excellent agreement with the certified reference concentration of 35.3 ± 0.9 μg/L Pb. The overall mean value for the 13 laboratories is 37.5 ± 4.0 μg/L Pb. Laboratories 9 and 13 had values which were significantly high, but no specific source of contamination could be identified. Since many of the collaborating laboratories have analysts who do not have a great deal of GFAAS experience, these accuracy data are very encouraging. The unknown sucrose and HFCS solutions for phase 2 were sent to each of these laboratories.

Phase 2 SRM 1643c Data. Of the 13 laboratories that received samples, 9 returned data for the sugar samples. Laboratory 4 had difficulty with their calibration curve and, as a result, their phase 2 data could not be used. SRM 1643c Pb data for the other eight laboratories are summarized in Figure 1. It is clear that all of the laboratories reported values which fell between 31.8 and 38.8 μg/L Pb, which is within ±10% of the mean reference value. The water sample was labeled SRM 1643c and was not sent as an unknown sample, as was done during phase 1.

Phase 2 Data for Unknown Sugar Samples. All eight laboratories were able to report data for both the sucrose samples and the HFCS samples. The blank corrected data for the spiked sucrose and HFCS samples appear in Table 3. Collaborators did, in fact, receive three samples of sucrose A (A1–A3) and HFCS K (K1–K3), and data for those samples are reported separately. Triplicate samples were sent so that the within-labora-

Table 2. Performance Data—Phase 1

lab no.	instrument model/ manufacturer	bkgd cor	calibration	20 μL, 25 μg/L (abs-s)	characteristic mass (pg)	SRM 1643c Pb detd[a] (ng/mL)
1	GF2000/GBC	D_2	point to point	0.214	10.3	34.4 ± 0.3
2	Spec AA-20/Varian	D_2	rational	0.119	17.6	37.6 ± 0.9
3	5000/Perkin-Elmer	D_2	linear lst sq	0.149	14.8	36.3 ± 1.2
4	5000/Perkin-Elmer	D_2	linear lst sq	0.182	12.1	35.9 ± 0.5
5	Z5100/Perkin-Elmer	Zeeman	linear lst sq	0.173	12.7	34.0 ± 0.7
6	5100ZL/Perkin-Elmer	Zeeman	nonlinear	0.051	45.3	37.8 ± 1.8
7	5100ZL/Perkin-Elmer	Zeeman	nonlinear	0.060	37.7	35.8 ± 3.4
8	Z5100/Perkin-Elmer	Zeeman	nonlinear	0.161	15.2	35.6 ± 0.4
9	Z5100/Perkin-Elmer	Zeeman	nonlinear	0.193	11.4	42.6 ± 0.5
10	1100B/Perkin-Elmer	D_2	nonlinear	0.133	16.0	37.2 ± 1.6
11	Z5100/Perkin-Elmer	Zeeman	nonlinear	0.170	12.9	35.4 ± 0.6
12	4100ZL/Perkin-Elmer	Zeeman	nonlinear	0.050	44.0	36.3 ± 0.6
13	1100/Perkin-Elmer	D_2	linear lst sq	0.055	35.9	48.9 ± 0.5

[a] Uncertainty represents ±1 standard deviation (n = 5)

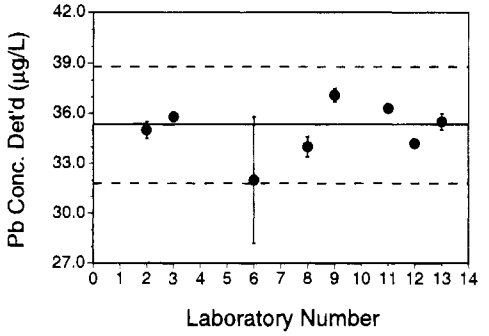

Figure 1. Results from the analysis of SRM 1643c (NIST acidified water). Certified Pb content: 35.3 ± 0.9 ng/mL. The solid line represents the mean certified value. The dotted lines represent ±10% of the mean certified value.

Table 3. Analytical Data for Sugars—Phase 2[a]

Sucrose Data—Spike Concentration Determined (ng/g)

lab no.	A1	A2	A3	B	C	D	F
2	122	122	123	516	81.6	278	61.0
3	105	111	110	486	57.4	232	37.7
6	121	123	121	526	57.4	285	47.2
8	107	109	111	512	58.2	222	37.7
9	79.8	75.5	70.0	365	38.7	164	20.5
11	106	114	106	518	68.8	228	36.3
12	118	123	119	560	72.6	230	46.2
13	114	114	114	447	72.6	222	47.6

Fructose Data—Spike Concentration Determined (ng/g)

lab no.	K1	K2	K3	G	H	M	J
2	133	142	147	485	68.1	190	49.5
3	116	118	119	499	55.9	187	39.5
6	130	118	122	540	65.7	191	50.4
8	100	103	106	458	48.6	166	33.6
9	58.4	62.7	62.7	320	25.6	105	13.9
11	121	100	104	505	56.0	157	46.6
12	130	150	124	567	95.0	179	62.0
13	103	102	108	368	57.1	175	46.5

[a] All data are blank corrected.

tory precision could be evaluated at the level of primary interest (100 ng/g). Sucrose E and fructose I were sent to each of the collaborators, but most of the laboratories could not reliably detect the Pb in the unspiked blank samples. FCL values (International Life Sciences Institute, 1993) for sucrose E using a variety of methods all resulted in a Pb value of less than 1 ng/g, which was low enough to be considered insignificant. As a result, sucrose data were not blank corrected. Fructose I, the unspiked HFCS sample, was higher but most laboratories reported "less than" values or "not detected". The data reported ranged from 4 to 34 ng/g. As a result, a well-characterized blank value of 19 ng/g established by the author was used to correct all of the fructose data (Miller-Ihli, 1993, 1994).

Table 4. Summary of Lead Data—Phase 2[a]

material	nominal spike level (ng/g)	% recovery	RSD_R[b] (%)	Horwitz value[c] (%)	RSD_r[d] (%)
sucrose					
B	500	96.7	12.3	17.8	
D	200	109	16.1	20.4	
A	100	105	14.0	22.6	2.67
C	50	118	21.0	25.1	
F	30	130	28.2	27.1	
sucrose av		112 ± 13	18.3		
fructose					
G	500	103	19.9	17.8	
M	200	112	19.6	20.4	
K	100	120	19.0	22.6	6.53
H	50	132	23.1	25.1	
J	30	134	30.2	27.1	
fructose av		120 ± 13	22.4		
overall av		116 ± 13	20.3		

[a] n = 8 laboratories, no outliers. [b] RSD_R = interlaboratory precision (% RSD). [c] Horwitz expected RSD_R ($RSD_R = 2^{(1-0.5\log C)}$ where C is the concentration expressed as a decimal). [d] RSD_r = intralaboratory precision (% RSD).

Data were analyzed using conventional statistical methods as well as AOAC statistical analysis macros developed by John Phillips which run in LOTUS and which calculate performance parameters according to the Harmonization Guidelines of AOAC (AOAC, 1989). A qualitative review of the data indicates that all of the values from laboratory 9 were 25–35% low, suggesting a systematic error. Application of the Dixon outlier test did not identify this laboratory as an outlier. No laboratory data were statistically rejected as outliers.

Data analyses using the data from all eight laboratories produced the results summarized in Table 4. Recoveries ranged from 96.7% for Pb in sucrose at the 500 ng/g level to 134% for Pb in HFCS at the 30 ng/g level. It is clear that recoveries increased with decreasing spike concentration, suggesting that contamination control is a real problem. This is highlighted in Figure 2. Although the method specifies the use of high-purity reagents, it is likely that airborne contamination is a source of Pb in some of the laboratories. Contamination no doubt occurs during the wet ash digestion since measurable contamination was not seen when collaborators analyzed SRM 1643c. More pronounced contamination below the 50 ng/g spike level is not unexpected since the digest concentrations are 7.5 and 4.5 ng/mL for the 50 and 30 ng/g spiked samples,

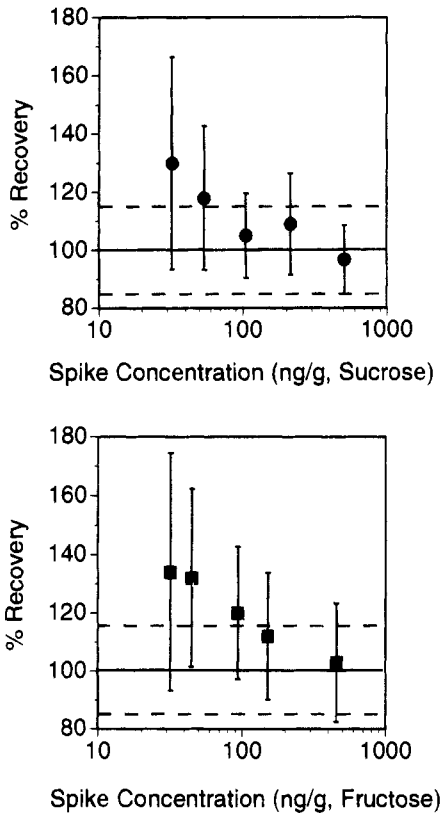

Figure 2. Average recovery data from the analysis of (A, top) sucrose samples and (B, bottom) fructose samples. Results are the mean of values from eight laboratories. Uncertainties represent ±1 standard deviation. The solid line marks 100% recovery. The dotted lines represent ±15%.

respectively. Review of the data in Table 3 highlights the fact that the mean values for the low-level spikes are significantly biased by extreme values. As an example, laboratory 2 reported recoveries of 152% and 189% for Pb in sucrose at the 50 and 30 ng/g spike levels, respectively, and laboratory 12 reported recoveries of 212% and 195% for the 50 and 30 ng/g spike levels for fructose, respectively.

From an accuracy point of view, the 100 ng/g level appears to be a safe minimum recommended level for quantification which should be achievable for all laboratories. It is also interesting to note that, in general, recoveries were higher for the HFCS samples. This may have been the result of inadequate homogenization of these more viscous samples, although data from a colleague in the author's laboratory (laboratory 8) showed an average 106% recovery for sucrose and 108% recovery for HFCS, suggesting that method performance is almost identical for the two matrices and that there is no systematic difference.

The interlaboratory precision (RSD_R) values increased with decreasing concentration as expected and agree very well with the Horwitz value, which is the expected interlaboratory precision based on the analyte concentration as defined by William Horwitz (Horwitz et al., 1990). With the exception of the 30 ng/g spike level, the measured interlaboratory precision was less than the predicted Horwitz value, suggesting that the precision for this Pb method is better than is typically found for analytes at these levels. The interlaboratory relative standard deviation was better than 20% at Pb levels of 100 ng/g or greater. The intralaboratory relative standard deviation was less than 10% at the 100 ng/g level.

Since the goal of the study was to have a method capable of determining Pb at the 100 ng/g level or greater, a review of the data for sucrose A1–A3 and fructose K1–K3 is very interesting. These samples, which were spiked nominally at the 100 ng/g level, highlight the fact that the accuracy (105% recovery sucrose; 120% recovery fructose), the interlaboratory relative standard deviation (14.0% sucrose; 19.0% fructose), and the intralaboratory relative standard deviation (2.67% sucrose; 6.53% fructose) are slightly better when the method is used to analyze sucrose samples as opposed to HFCS samples. It is clear from the data in Table 4 that the method performs well at Pb levels of 100 ng/g or higher, with regard to both accuracy and precision.

Table 5 contains the results of the analysis of the data based on the seven Youden matched sample pairs. The overall average accuracy was 100 ± 15% based on the average percent recovery of 115%. The average interlaboratory precision was 21.1%, and the average intralaboratory precision was 9.63%. As expected, the method provides better performance at higher Pb levels and provides much better performance than the old FCC method (International Life Sciences Institute, 1993), which provided an average interlaboratory precision of 42.4%, an intralaboratory precision of 26.4%, and an average percent recovery of 86%.

Conclusions. It is clear from these data that graphite furnace AAS has become a sufficiently mature technique that rugged methods may be transported fairly easily into laboratories that are not involved in GFAAS methods development or full-time analyses. The air ashing step makes the complete digestion of the sample during the wet ashing far less critical and has no significant deleterious effect on tube lifetime as long as high temperatures are not used. The detailed procedure verifying performance through sensitivity checks and accuracy verification is a critical part of the procedure which positively impacts the quality of data for unknown samples. It is clear from this study that sample contamination continues to be a problem, highlighting the need to develop methods which avoid the sample decomposition step. More recently, a method has been reported (Miller-Ihli, 1993) that allows the direct analysis of both sucrose and HFCS after dilution with an equal mass of water. This direct method, which saves time and minimizes the likelihood of sample contamination, is currently being peer validated. The direct method provides a method detection limit of 0.9 ng/g compared with 3.3 ng/g provided by this method.

With care to avoid contamination, the method studied herein provides excellent results. The method provides an intralaboratory relative standard deviation of better

Table 5. Youden Matched Pairs Data Analyses[a]

parameter	sucrose/fructose matched sample pairs							overall av
	B/G	D/M	A1/K1	A2/K2	A3/K3	C/H	F/J	
nominal spike concn (ng/g)	500	200	100	100	100	50	30	
% recovery	99.8	110	111	113	112	124	110	115
RSD_R (%)	15.3	16.5	18.7	19.8	18.9	27.3	31.0	21.1
RSD_r (%)	5.08	6.57	8.67	9.98	6.67	15.5	14.9	9.63

than 10% and an interlaboratory relative standard deviation of less than 20% for samples with Pb at levels of 100 ng/g or greater. As a result of this collaborative study the Subcommittee recommended to the FCC that the method studied be considered as the new FCC method for the quantification of Pb at levels of 100 ng/g or greater (International Life Sciences Institute, 1993). The Subcommittee noted that laboratories with competent analysts should be able to utilize the method to assure compliance at an acceptable cost and noted that routine use may be limited in a manufacturing setting, where the potential for sample contamination is great and where the expense, expertise, and equipment required may be a limitation.

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