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Toxic Elements

Interlaboratory Trial: Graphite Furnace Atomic Absorption Spectrometric Determination of Lead and Cadmium Extracted from Ceramic Foodware

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INTRODUCTION

CFSAN recently developed a graphite furnace atomic absorption spectroscopy (GFAAS) procedure to replace the use of flame AAS in the official method for determination of lead and cadmium extracted from ceramic foodware[1,2]. An interlaboratory trial was also conducted to assess the GFAAS procedure in other laboratories. In the official method, lead and cadmium are determined in solutions obtained by leaching the ware 24 h with 4% acetic acid at room temperature[3,4]. Since the purpose of the trial was to assess the GFAAS measurement procedure, not the 24-hour leaching procedure, leach solutions (not ceramic foodware articles) were sent to participants for analysis. Results of the interlaboratory trial of the GFAAS procedure are presented in this LIB.

MATERIALS AND METHODS

Solutions used in the trial were obtained by leaching 4 ceramic foodware articles (one unit of each article) 24 h at room temperature with 4% acetic acid. The leach solution from article A was diluted with 4% acetic acid to obtain a concentration appropriate for evaluation of GFAAS near FDA's lowest action level for lead (0.5 μg/mL). Ceramic foodware articles were chosen to represent various types of ceramicware that FDA generally encounters in its regulatory program and are described in Table 1. Samples A, B, and C were obtained by FDA field laboratories. Sample D was purchased in the Washington, DC metropolitan area. Leach solutions from each article were analyzed twice in a time period ≤15 days: once by the reference laboratory (CFSAN) and once by a participating trial laboratory. Solution identifications were coded so that sample identity was unknown during the second analysis. Three different laboratories participated in the trial. Each laboratory was given (1) a draft copy of the method[1], (2) ERB Standard Operating Procedure 108 for the method[2], (3) fill-in-the-box forms for reporting results, and (4) the trial solution(s).

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Table 1. Results of interlaboratory trial

ID	Sample Description	Trial Lab	LEAD RESULTS Reference µg/mL	Trial µg/mL	Trial/Ref %	CADMIUM RESULTS Reference µg/mL	Trial µg/mL	Trial/Ref %
A	multicolor decorated ceramic plate	1	0.431	0.429	100	0.0067	0.0066	99
B	traditional Chinese ceramic plate	1	597	604	101	0.0025	0.0025	100
C	traditional Mexican earthenware bowl	2	1.47	1.40	95	<0.0005[a]	<0.0006[a]	100
D	uniform red color glazed bowl	3	0.019	0.017	89	0.0846	0.0864	102

[a] Less than X (<X) indicates not found above the lower reporting limit of X.

Table 2. Quality control measurements of interlaboratory trial

Figure of merit	Lab ID	Lead	Cadmium
Instrument sensitivity	Reference	9.0	0.41
Characteristic mass, pg equivalent to	1	34	1.5
peak area of 0.0044 A-s	2	10	0.50
	3	12	0.64
Sample concentration limits	Reference	0.005	0.0005
µg/mL equivalent to	1	0.023	0.0009
peak area of 0.050 A-s	2	0.006	0.0006
	3	0.014	0.0005
Analysis of 0 µg/mL solutions	Reference	≤0.008	≤0.003
peak area, A-s	1	≤0.004	≤0.003
	2	≤0.004	≤0.015
	3	≤0.002	≤0.004
Recovery	Reference	99-107	91-106
%	1	81-104	91-100
	2	98	not analyzed
	3	92	101

RESULTS AND DISCUSSION

Trial results are presented in Table 1. The data show that for lead concentrations greater than approximately 0.5 μg/mL, trial results agreed 95-101% with reference values. Lead concentrations approximately equal to 0.020 μg/mL agreed 89% with reference values. Trial results for all cadmium concentrations agreed 99-102% with reference values. Lead and cadmium concentrations in the trial leach solutions ranged 0.019-597 and <0.0005-0.0846 μg/mL, respectively.

The quality of analytical work in the interlaboratory trial was excellent. Results of quality control measurements required by the method are presented in Table 2. Instrument sensitivity achieved by the participants for lead was within ±20% of manufacturer specifications as required in the method. Sensitivity achieved for cadmium in labs 2 and 3 was within 50% of specifications and most likely will improve as the labs become more familiar with the method. Manufacturer specifications for the instrument used in lab 1 are approximately 3 times less sensitive than those for the instruments used in the reference and labs 2 and 3.

Sample concentration limits achieved by the participants were excellent and were well below those required to enforce FDA's lowest lead and cadmium regulatory guidelines (0.5 and 0.25 μg/mL, respectively).

Analysis of 4% acetic acid with no analyte intentionally added was required by the method to check for contamination and carry-over. In general, participants had better control over carryover than the reference lab. Lab 2 found cadmium contamination in a method blank. The level of contamination did not meet requirements of the method but was less than the sample limit. The contamination was confirmed by analysis of the solution in the reference lab.

Analysis of fortified portions of leach solutions as required by the method also gave excellent results. Recovery ranged 81-107%. Lead and cadmium fortification levels were approximately 0.020–600 μg/mL and 0.003-0.085 μg/mL, respectively.

CONCLUSION

The interlaboratory trial of the GFAAS method for determination of lead and cadmium in ceramicware leach solutions was successfully completed. Results of the trial demonstrated that the method is rugged, gives excellent results, and is appropriate for use in laboratories outside CFSAN.

REFERENCES

[1] Hight, S. C., *FDA Laboratory Information Bulletin No. 4123*, 1998, FDA, Rockville, MD

[2] Hight, S. C., *Standard Operating Procedure No. 108*, 1997, Elemental Research Branch, CFSAN, FDA

[3] American Society for Testing and Materials, *Annual Book of ASTM Standards, Volume 15.02, Glass; Ceramic Whitewares*, designation C738-94, ASTM, West Conshohocken, PA, 1997

[4] *Official Methods of Analysis*, 1997, 16th Ed., 3rd Revision, Method 973.32, AOAC International, Arlington, VA