

Determination of total lead in lipstick: Development and validation of a microwave-assisted digestion, inductively coupled plasma–mass spectrometric method

NANCY M. HEPP, WILLIAM R. MINDAK, and JOHN CHENG,
*Office of Cosmetics and Colors (N.M.H) and Office of Regulatory Science
(W.R.M., J.C.), Center for Food Safety and Applied Nutrition,
U.S. Food and Drug Administration, College Park, MD 20740.*

Accepted for publication May 6, 2009.

Synopsis

Recent reports describing the presence of lead (Pb) in lipsticks have suggested that, under ordinary use, the potential amount of Pb exposure is harmful. To permit independent assessment of the Pb contamination, a method for determining total Pb in lipstick using microwave-assisted digestion and analysis employing inductively coupled plasma–mass spectrometry (ICP–MS) was developed and validated. Since lipsticks may contain fats, oils, pigments, dyes, and minerals, several reference materials (RM) were analyzed, including coal, wear metals in oil, organic Pb in oil, milk powder, and estuarine sediment. With the exception of the RM with mineral content (estuarine sediment), complete recovery of Pb from the RMs was obtained by simple nitric acid (HNO₃) digestion. Complete recovery of Pb from estuarine sediment was achieved only when hydrofluoric acid (HF) was added to the digestion mix, followed by treatment with excess boric acid (H₃BO₃) to neutralize the HF and to dissolve insoluble fluorides. Commercial lipsticks were tested for total Pb by the validated method. The detection limit was estimated to be 0.04 µg Pb/g. The average value obtained for the lipsticks was 1.07 µg/g. Undigested material was present in some lipstick digests when only HNO₃ was used, and generally lower Pb values were obtained. All of the Pb levels found by the U.S. Food and Drug Administration (FDA) were within the range the agency would expect to find in lipsticks formulated with permitted color additives and other ingredients prepared under good manufacturing practice (GMP) conditions. This method will be useful for the FDA and industry in helping to ensure the safety of cosmetic products.

INTRODUCTION

Although major sources of lead (Pb) contamination from leaded gasoline, Pb-based paints, Pb in public water systems, and Pb solder for sealing canned foods have been reduced through various regulatory actions, public concerns still exist over possible sources of Pb contamination. Pb from gasoline and paint can remain in soil and dust for many years, and imported foods and cosmetics may contain unsafe levels of Pb (1). The FDA issued warnings in 2003 for litargirio, a yellow- or peach-colored powder used in traditional remedies by people of Central America and the Caribbean region, particularly the Dominican Republic, because it contains up to 79% Pb. In 2003 and 2006 the FDA issued warnings for kohl, a traditional cosmetic eyeliner common in the Middle East, North Africa, Sub-Saharan Africa, and South Asia, because it frequently contains more than 50% Pb.

Recent media reports and e-mail hoaxes describing the presence of Pb in lipsticks have suggested that under conditions of ordinary use, the potential amount of Pb exposure is harmful (2–4). Pb contamination of lipsticks may originate from Pb solder or leaded paint in production equipment or from contaminated dust. Lipsticks also may be contaminated with Pb if they are manufactured with ingredients that naturally contain Pb or are produced under conditions that could introduce Pb into the ingredients. Dyes and pigments used as ingredients in lipsticks are regulated as color additives by the FDA and must undergo pre-market approval by the agency before they may be used in any cosmetics. The FDA controls potential Pb exposure from color additives by setting limiting specifications for Pb (5). Under current regulations, most color additives approved for cosmetic use are permitted to contain up to 20 µg Pb/g. In addition, certain color additives are required to be batch certified by the FDA, and analysis for Pb is part of the certification process.

Other than color additives, the FDA does not have the statutory authority under the Federal Food, Drug, and Cosmetic Act (FD&C Act) to require pre-market approval of cosmetic products such as lipsticks or their ingredients. It is the responsibility of the manufacturer or distributor to ensure that cosmetic products and their ingredients are in compliance with requirements of the FD&C Act and other applicable laws and regulations (6). With the exception of color additives, a manufacturer may use any ingredient in the formulation of a cosmetic that does not cause the cosmetic to be adulterated or misbranded under the FD&C Act.

Several methods have been reported for the analysis of Pb in lipstick and other cosmetics. Okamoto *et al.* (7) used a 1-gram portion of lipstick ignited at 500°C. The resulting ash was extracted with 20 ml and then 10 ml of 2N hydrochloric acid (HCl) and made up to 50 ml with 0.5% HCl. Pb was determined by atomic absorption spectrometry (AAS) using the standard addition method. A simple microwave-assisted acid extraction technique for determining Pb by inductively coupled plasma–optical emission spectrometry (ICP–OES) was reported by Besecker *et al.* (8). In their method, 0.15-g portions of several types of cosmetics were treated with 3.0 ml of HNO₃ and heated in quartz vessels for a total time of 50 minutes at a maximum pressure of 74 bar. Accuracy was verified by spike recoveries and by recoveries from an estuarine sediment reference material (RM) (National Institute of Standards and Technology, NIST, Estuarine Sediment SRM 1646). Besecker *et al.* mentioned that their quartz vessels were cleaned with a mixture containing hydrofluoric acid (HF). Others have noted an increased recovery of Pb when using vessels previously exposed to HF (9). Satisfactory recovery of Pb from the RM may not have occurred with vessels that had no prior HF exposure. The Lead Analysis Task Force of the Cosmetic, Toiletry, and Fragrance Association (CTFA, now the Personal Care Products Council) (10), developed a method for determining Pb in cosmetics using HNO₃ and HF, microwave heating in sealed vessels, and for those cosmetics containing refractory materials, a subsequent treatment with boric acid (in smaller quantities than used in this study). This report presents a validated method for determination of total Pb in lipstick, the Pb content of several lipstick products analyzed by the validated method, and a comparison with Pb content determined by other techniques.

The FDA has developed and validated a method for determining Pb in lipstick in order to independently assess possible Pb contamination of lipstick products on the market. Analysis by x-ray fluorescence (XRF) was initially investigated in order to avoid lengthy sample preparation. However, the technique is not sensitive enough with the available

equipment and is subject to matrix absorption errors. AAS with electrothermal atomization analysis was also considered. However, since sample digestion would be necessary, inductively coupled plasma–mass spectrometry (ICP–MS) was chosen because of its potential for better sensitivity and speed. Thus, the effort focused on developing a method using microwave-assisted acid digestion for sample preparation and determination by ICP–MS.

EXPERIMENTAL

CHEMICALS, REAGENTS, AND REFERENCE MATERIALS

Twenty shades of lipstick sold in the United States under ten brand names were purchased from retail stores or provided by manufacturers. Multiple samples with the same lot number were obtained for several shades in order to compare analytical techniques. Six RMs were used for comparing different preparation techniques and for quality assurance: wear-metals in lubricating oil, SRM 1084a (NIST, Gaithersburg, MD); whole milk powder, SRM 8435 (NIST); estuarine sediment, SRM 1646a (NIST); lead in base oil 20 standard, ORG-PB8-2Y/Z (SPEX CertiPrep, Inc., Metuchen, NJ); base oil 20 standard (SPEX CertiPrep, Inc.); and trace elements in coal, SRM 1632c (NIST).

American Society for Testing and Materials (ASTM) type 1 grade water was used to prepare reagents, standards, and analytical solutions. Pb standards (0, 0.1, 0.5, 1.0, and 10 ng Pb/ml), Pb stock (0.1 and 1.0 μg Pb/ml), and thallium internal standard (0.1 μg Tl/ml) solutions were prepared from commercial ICP–MS grade single-element analyte solutions (High-Purity Standards, Charleston, SC). Trace metals grade (TMG) HNO_3 (Fisher Scientific, Pittsburgh, PA) was used for cleaning laboratory ware and digestion vessel liners. Optima grade HNO_3 and HF (Fisher Scientific) were used for sample, stock, and standard solutions. Boric acid (Puratronic grade, Alfa Aesar, Ward Hill, MA) was used to prepare 4% boric acid solution, which was conveniently dispensed with a bottle-top dispenser. A 0.100- μg Pb/g in base oil 20 stock solution was prepared from 1000 μg Pb/g (SPEX ORG-PB8-2Y/Z organo-metallic standard solution) serially diluted to 10.00 and then to 0.100 μg Pb/g with SPEX base oil 20.

Lipsticks were digested using XP-1500 Plus vessels in a MARS microwave digestion oven (CEM Corp., Matthews, NC). Pb determinations were performed on an Agilent 7500c ICP–MS (Agilent Technologies, Inc., Santa Clara, CA) equipped with a Peltier cooled Scott double-pass spray chamber and a MicroMist nebulizer (Glass Expansion, West Melbourne, Victoria, Australia). The built-in peristaltic pump was used to deliver the analytical and thallium internal standard solutions to the nebulizer at 0.17 ml/min and at 0.01 ml/min, respectively. The analytical and internal standard solutions were merged with a Tee fitting.

METHOD DEVELOPMENT

Lipstick is a challenging matrix of many ingredients including waxes, oils, dyes, and pigments (11). The pigments may include refractory minerals such as alumina, silica, titanium dioxide, and mica. Preliminary experiments with one lot of lipstick revealed an easily detectable amount of Pb, but quantitative results varied depending on preparation technique. In order to evaluate preparation techniques, a composite was prepared by

melting and mixing together eleven tubes of lipstick (same brand and shade, but several lot numbers). These lipsticks were placed in a beaker submerged in a water bath at 85°C and stirred with a propeller-type mixer.

Initial attempts to completely dissolve a lipstick sample using typical microwave-assisted HNO₃ digestion were unsuccessful. A cloudy, white suspension remained after the treatment. Also, replicate results for a single lot of lipstick showed variations in Pb recovery for different lipstick portion sizes and maximum digestion temperatures (see Table I).

The portion size and temperature effects on Pb recovery suggested that microwave-assisted digestion with HNO₃ was incomplete for Pb and that levels of Pb recovered would vary depending on digestion parameters. Therefore, other preparation procedures were investigated that might achieve total recovery of Pb. A dry ash procedure was tried in which a lipstick sample was heated gradually to 540°C and held at that temperature for an hour. The resulting residue was treated with HNO₃ and HCl but would not completely dissolve. The acid leachate was diluted with water and analyzed by ICP–MS. Results were variable and lower compared to values obtained with microwave-assisted acid digestion. Similarly, sodium carbonate fusion at 1075°C resulted in a residue that would not completely dissolve in HNO₃ or HCl and produced variable values for Pb. The results indicated that some Pb must be associated with the undissolved refractory mineral matter in the lipsticks and suggested that HF would be necessary to break down the minerals.

Based on work of other investigators (10), the initial HF dissolution procedure used 0.3-g portions of lipstick, 7 ml HNO₃ + 2 ml HF, and sealed Teflon microwave digestion vessels. A two-step procedure was used for digestion. The vessels were heated to 130°C over 15 minutes and held at that temperature for three minutes before ramping to 200°C over 15 minutes and holding for an additional 30 minutes. The vessels were allowed to cool to <50°C and then were vented. Initially 6 ml of 4% boric acid was then added to each vessel and the solutions were heated to 170°C over 15 minutes and held for ten minutes to complex the HF. Boron forms a strong complex with fluoride according to the reaction shown in equation 1. After cooling and venting, the solutions were diluted to a final volume of 200 ml for ICP–MS analysis. However, the digests were still cloudy, with a gelatinous precipitate appearing upon centrifugation. Also, results varied depending on the analytical portion. Equation 1 is as follows:



Therefore, an excess of boric acid was used to dissolve any insoluble fluorides. The revised HF digestion procedure used 30 ml of 4% boric acid and resulted in clear solutions for all

Table I
Analytical Portion and Temperature Effects on Pb Determined After Digestion with Nitric Acid

Portion size (g)	Maximum digestion temperature (°C)	µg Pb/g
0.3	240	1.4
0.1	240	2.1
0.3	200	0.5

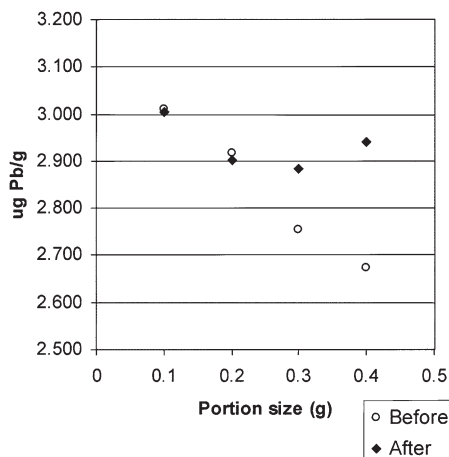


Figure 1. Portion size effect before and after adding excess boric acid.

Table II
Final Method Parameters

<ul style="list-style-type: none"> ● 0.3-g lipstick portion ● 2 ml HF + 7 ml HNO₃ ● CEM XP-1500+ vessels, MARS Microwave Digestion System ● Heat in two steps to 200°C, hold for 30 min: 					
Stage	Power (watts)	Ramp (min)	Pressure (psi)	Temperature (°C)	Hold (min)
1	1200	15:00	0800	130	3:00
2	1200	15:00	0800	200	30:00
<ul style="list-style-type: none"> ● Cool to <50°C, vent, add 30 ml 4% boric acid ● Heat to 180°C, hold for 10 min ● Dilute to 200 ml 					

lipstick samples and no variation in ICP–MS values for lipstick portion sizes ranging from 0.1 to 0.4 g (see Figure 1). The final method parameters are outlined in Table II.

For each lipstick sample, duplicate portions and portions fortified at 0.5 and 1.0 µg Pb/g were digested. A Pb solution in 1% HNO₃ was used for fortification. Each digestion batch of 12 vessels also included a blank, a blank + 0.02 µg Pb/g, an organic Pb RM (0.100 µg Pb/g in base oil 20), and an RM with mineral content (estuarine sediment, SRM 1646a).

RESULTS

METHOD VALIDATION

Accuracy of the method was demonstrated by measuring recoveries of Pb from RMs and from fortified lipstick samples. Since no lipstick-type RM was available, several RMs were analyzed representing varying matrix types. NIST 1635 trace elements in coal (complex matrix, organics); NIST 8435 whole milk powder (high fat matrix); NIST 1084a wear metals in oil and SPEX ORG-PB8-2Y/Z lead in base oil 20 (oily matrices

containing organically complexed Pb); and NIST 1646a estuarine sediment (refractory mineral matrix). Pb recoveries from RMs with and without HF are shown in Table III. Complete recovery of Pb from the RMs was obtained by simple HNO₃ digestion, with the exception of estuarine sediment, for which complete recovery was obtained only when HF was used in the digestion. Each lipstick sample was fortified at two levels and analyzed following HNO₃/HF digestion, with recoveries averaging 98.1%. Absence of matrix influence was shown by sequentially diluting several analytical solutions, with no significant differences.

Analytical solution stability was demonstrated by analysis of three representative analytical solutions over time. Two analytical solutions containing approximately 0.25 and 1.0 µg Pb/l were analyzed on days 1, 3, 7, and 14 using freshly prepared standard solutions on each day. There was <5% variation over the time period. A 10-µg Pb/l standard solution prepared on day 1 and analyzed with the analytical solutions on subsequent days behaved similarly.

Method precision was demonstrated by between-day and within-day repeatability experiments. A 3% relative percent difference (RPD) was observed from analyzing 22 portions of lipstick composite over three days, and 2% RPD was obtained from analyzing 12 portions of one lipstick brand over three days. The precision of the instrument was tested by analyzing an analytical solution seven times on one day, yielding 2% RPD.

The ruggedness of the method was demonstrated by varying the analytical parameters. There were no significant differences in Pb results with portion size variations of 0.1 to 0.4 g. The volume of HF was varied from 0 to 4 ml (0, 0.5, 1.0, 2.0, 3.0, and 4.0 ml). Pb recovery became constant when ≥1 ml was used. The 4% boric acid solution amount was varied from 6 ml to 60 ml (6, 20, 30, 40, and 60 ml), with Pb recovery becoming constant when ≥20 ml was used. Solutions also became clear, eliminating the need for filtration or centrifugation before ICP-MS analysis. There was no significant change in Pb recovery when the maximum digestion temperature was lowered from 200°C to 180°C.

As a measure of quality control, each digestion batch included two RMs: lead in base oil 20 (representing organic Pb in an oily matrix) and estuarine sediment (representing a mineral matrix). Recovery of Pb from the RMs is shown in Figure 2.

Method blanks and method blanks spiked near the detection level were also included in each digestion batch. The average and standard deviation for blanks from fifteen batches

Table III
Lead Recoveries from Reference Materials and Lead Values from a Compositing Lipstick
With and Without HF*

	Certified value (µg Pb/g)	±95% C.I.	HNO ₃ only (µg Pb/g)	HF + HNO ₃ (µg Pb/g)
NIST 1635 (trace elements in coal)	1.9	0.2	1.8 (95%)	1.8 (95%)
NIST 1084a (wear metals in oil)	101.1	1.3	103.7 (103%)	
SPEX ORG-PB8-2Y/Z (lead in base oil 20)	1000			1000 (100%)
NIST 8435 (whole milk powder)	0.11	0.05	0.10 (91%)	0.11 (100%)
NIST 1646a (estuarine sediment)	11.7	1.2	8.2 (70%)	10.8 (93%)
Compositing lipstick	—	—	0.29	2.91

*Values are the average of 3 to 15 samples. Recoveries are indicated in parentheses.

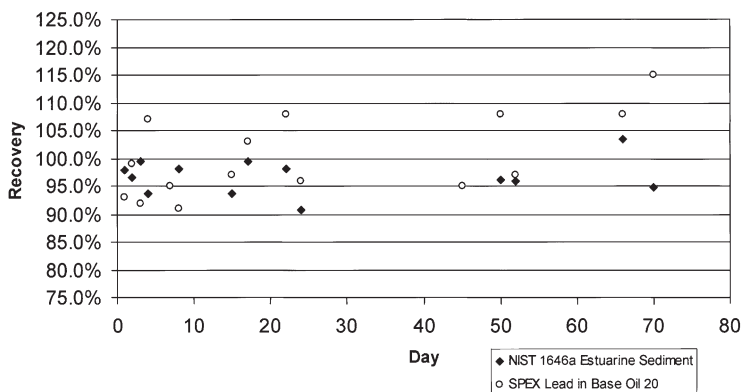


Figure 2. Lead recoveries from reference materials.

are shown in Table IV, along with the aggregate RM results. Method blank results were used to estimate the detection limit of 0.04 $\mu\text{g Pb/g}$ using equation 2 (12):

$$\text{Detection limit} = (2 \cdot t \cdot \sigma \cdot \sqrt{(1 + 1/N)}) \quad (2)$$

LIPSTICK SURVEY AND COMPARISON WITH VALUES BY OTHER METHODS

Twenty-two lipstick samples (not including the composite), identified by brand, shade, and lot number, were analyzed for Pb by the validated method. The results are summarized in Table V. All of the lipsticks contained detectable amounts of Pb, with values ranging from 0.09 to 3.06 $\mu\text{g/g}$ and an average amount of 1.07 $\mu\text{g/g}$. Despite the limited size and color range of the survey samples (all were red shades), samples from a few manufacturers (A – C) appeared to contain the highest levels of lead.

As stated above, recoveries from lipsticks fortified with lead nitrate, $\text{Pb}(\text{NO}_3)_2$, and analyzed by the validated method (using HNO_3 and HF) averaged 98.1%. However, recoveries from some lipsticks were equally good using digestion with HNO_3 alone (see Table III). Good recoveries were observed for all but one RM (the mineral-containing estuarine sediment) using either technique. This suggests that Pb-containing minerals were present in some but not all lipsticks.

Table IV
Mean Values and Standard Deviations for Reference Materials, Blank, and Composited Lipsticks Analyzed Repeatedly

	NIST 1646a [estuarine sediment ($\mu\text{g/g}$)]		SPEX ORG-PB8-2Y/Z [lead in base oil 20 ($\mu\text{g/g}$)]		Method blank ($\mu\text{g/g}$)	Blank + 0.02 $\mu\text{g Pb/g}$ ($\mu\text{g/g}$)	Composite lipstick
	Value (n = 13)	Ref. value	Value (n = 15)	Ref. value		Value (n = 15)	Value (n = 22)
Average	10.84	11.7 ± 1.2	0.100	0.100	0.019	0.037	2.91
S.D.	0.36	—	0.007	—	0.008	0.009	0.09

Table V
Lead Content in Lipsticks by Validated Method and by Two Other Techniques

Product	Shade	Lot	FDA: HF + HNO ₃ (µg Pb/g)	FDA: HNO ₃ (µg Pb/g)	CSC (µg Pb/g) (lot #s unknown)
A	1	a		1.40	
		b		1.20, 1.22	0.12, 0.56
		c	3.06	1.90	
		d	3.05		
B	1	Composite	2.91	0.29	<0.02
		a	2.38		
A	2	a	2.24		0.03, 0.03
C	1	a	1.79		<0.02, <0.02, 0.06
A	3	a	1.76		0.28
C	2	a		1.53	
		b		0.62, 0.68	0.50, 0.65
		c	1.47	1.20	
B	2	a	1.37	0.91	<0.02
		b		0.83, 0.81	
C	3	a	1.21		0.19
B	3	a	1.04		<0.02, 0.03
C	4	a	0.67	0.60	0.58
		b	0.79	0.74, 0.80	
D	1	a	0.55		<0.02
D	3	a	0.48		0.03
D	2	a	0.43		<0.02
E	1	a	0.33		0.09
C	5	a	0.23		0.12
F	1	a	0.17		0.12
G	1	a	0.15		<0.02, 0.04
H	1	a	0.12		0.21
I	1	a	0.10		<0.02, 0.03
J	1	a	0.09		<0.02

The method developed here is for total Pb. Our studies showed that variable amounts of Pb can be extracted depending upon experimental conditions such as analytical portion, acids used, temperature, decomposition procedure, etc. Consistent results could usually be obtained only by including HF in the digestion procedure. To illustrate this, five lipstick lots and the composite were analyzed by digestion with HNO₃ alone or with HNO₃ + HF. Substantially higher values were obtained for three of the lots and the composite using HNO₃ + HF compared to HNO₃ alone. Pb values for two different lots of the same lipstick were equivalent by digestion with HNO₃ alone or with HNO₃ + HF (see C-4, lots a and b). Results reported by the Campaign for Safe Cosmetics (CSC), in which several of the same brands and shades of lipsticks were analyzed (2), are also listed in Table V. The CSC method used a 0.5-g portion, extraction with sulfuric and nitric acids, and ICP–MS analysis. Lot numbers were not reported by the CSC.

Differences among values obtained by the three digestion techniques can be explained by the presence of mineral content in some formulations, as well as by the fact that Pb is easily precipitated as a sulfate after sulfuric acid is used in the CSC extraction. Mica, a mineral permitted as a color additive, which frequently contains small amounts of Pb (13), would require HF for complete dissolution. The use of sulfuric acid in the CSC ex-

traction method may have reduced the soluble Pb available for ICP–MS analysis, thus resulting in the lower values reported by the CSC.

The FDA has not set specifications for Pb in cosmetics, except that color additives permitted as ingredients are usually limited to 20 µg Pb/g (20 ppm) (5). The Pb levels found in these lipsticks, determined by the validated method, are within the range that might be expected from lipsticks formulated with permitted color additives and other ingredients prepared under good manufacturing practice (GMP) conditions.

CONCLUSIONS

Typical microwave-assisted HNO₃ digestion produced low-biased, inaccurate values for some lipstick samples and one of the RMs. All of the lipsticks analyzed in this work contained detectable amounts of Pb when digested with HNO₃ and HF. Some of the Pb in certain lipstick samples appears to be incorporated in the refractory mineral pigments, which require HF for complete digestion. Pb levels found by the FDA are within the range that might be expected from lipsticks formulated with permitted color additives and other ingredients prepared under GMP conditions.

ACKNOWLEDGMENTS

The authors thank Richard Jacobs of the FDA's San Francisco District Lab for analyzing several lipsticks after a HNO₃ extraction treatment. Nancy Hepp would like to thank Stephen Capar in the FDA's Office of Regulatory Science and his group for allowing her to use his laboratories, equipment, and instrumentation to do the studies, and for providing helpful guidance. She would also like to thank Fred Hurley for advice and for sharing knowledge of cosmetics and lipsticks. Finally, we are very grateful to representatives from several cosmetic companies and the industry trade group, the Personal Care Product Council, for cooperation in providing samples and information about testing, and to Stanley Milstein in the FDA's Office of Cosmetics and Colors for coordinating all involved parties.

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