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App Note

Analysis of Lipstick for Toxic Elements Using ICP-MS

Abstract

Evidence of the use of cosmetics, including lipstick, has been found in civilizations as early as ancient Mesopotamia. Many of the cosmetics used throughout history have contained potentially toxic elements and other contaminants. Ancient Egyptians used cosmetics containing large amounts of lead and mercury. Modern cosmetics are perceived to be free of dangerous toxins due to widespread regulation of many consumer products. The FDA, which oversees the regulation of cosmetic products, does not have regulations governing the level of toxic or dangerous contaminants in finished products such as lipsticks. The FDA regulates limits on compound concentrations of additives and colorants but no overall regulation is in place for the finished product's level of potential contamination.

Studies prior to 2012 have found levels of lead up to 3 ppm in lipstick. The purpose of this study was to re-examine the potential for lead contamination in lipstick and determine if any other potentially toxic metals were present in these lipsticks. Forty-eight lip products including lipsticks, lip glosses, moisturizing sticks, and lip stains were tested for the presence of toxic elements by ICP-MS.

Methods & Materials

Standards

Spex CertiPrep standards were used to calculate the levels of metals in the lipstick samples. The standards used were SPEX CertiPrepClaritas PPT® Grade Multi-Element Solutions: CLMS-1, CLMS-2, CLMS-3, and CLMS-4.

Reagents

High purity nitric acid and hydrofluoric acid were used to digest the lipstick samples. A 4% boric acid solution was added after digestion to neutralize the hydrofluoric acid.

Table 1. Masses examined.

Element	Gas Mode	Line	Element	Gas Mode	Line	Element	Gas Mode	Line
Ag	Air	107,109	K	He	39	Se	He	77
Al	Air & He	27	La	Air	139	Si	He	30
As	Air & He	75	Li	Air	7	Sm	Air	147
Au	Air	197	Lu	Air	175	Sn	He & Air	117-120
Ba	Air	135-137-138	Mg	Air	24	Sr	He & Air	86, 88
Be	Air	9	Mn	He	55	Ta	Air	181
Ca	He	44	Mo	Air	95, 97, 98	Tb	Air	159
Cd	He	111-113	Na	Air	23	Te	Air	125
Ce	Air	140	Nd	Air	146	Th	Air	232
Co	Air & He	59	Ni	He & Air	60	Ti	Air	47
Cr	Air & He	52, 53	Os	Air	189	Tl	He	203, 205
Cs	Air	133	P	Air	31	Tm	Air	169
Cu	He	63	Pb	Air	206-208	U	Air	238
Fe	He	56	Pd	Air	105	V	He	51
Ga	Air	71	Pr	Air	141	W	Air	182
Gd	Air	156	Pt	Air	195	Y	Air	89
Ge	He	74	Rb	Air	85	Yb	Air	172
Hf	Air	178-180	Re	Air	187	Zn	He	68
Hg	Air	201	Rh	Air	103	Zr	Air	90
Ho	Air	165	Ru	Air	101			
In	Air	115	Sb	Air	121, 123			
Ir	Air	193	Sc	He	45			

Samples

Forty-eight lipstick samples representing 14 brands were donated by SPEX CertiPrep employees. The price range for samples was between $5 and $35 per container. Four types of lip colorants were represented among the samples: lip stain, lip gloss, lip balm, and lipstick and included liquids, gels and solid material. The samples were classified and grouped according to finish and color. The lip product finishes designated were:

- Clear: containing no color or finishing material
- Matte: contained colorant but lacked any other type of finishing material
- Pearl: contained color and an iridescent or white top finish
- Metallic: contained color with a gold, silver or other metal color finish

The lipsticks were also grouped into one of seven color groups: Beige/Tan (3), Brown (7), Light Pink (3), Dark Pink (7), Corals/Peaches (11), Berry/Wine (11), and White or Colorless (6).

Sample Preparation

The general sample preparation method followed the methods reported by Hepp et. al. (1). The method follows a two-step process in which samples are digested by microwave digestion.

Step 1:

0.3 g of sample, 7 mL high purity nitric acid and 2 mL high purity HF are added to a microwave vessel and the samples are heated over 15 minutes to 130 °C. Samples are held at 130 °C for 3 minutes before the temperature is ramped to 200 °C over 15 minutes and held at 200 °C for 30 minutes.

Step 2:

30 mL of 4% high purity boric acid solution is added to the vessels and the samples are heated again in the microwave to 170 °C over 15 minutes and held for 10 minutes at 170 °C. The samples were then diluted to 50 mL using DI H_2O.

Digestion blanks were run in between sample digestions to clean out the vessels and to minimize carryover. A final 1000x dilution was performed prior to ICP-MS analysis.

Instrument Conditions

Screening of the samples for macroelemental composition was performed on a Perkin Elmer ICP-OES Optima 7300. The trace element analysis was performed using an Agilent ICP-MS 7700. The system was operated in air mode and collision mode. Collision mode using helium was employed for the examination of elements with atomic weights under 100. The line selection and mode were chosen to reduce possible interferences. See Table 1 for the list of elements and the analysis mode and selected lines.

The conditions for the ICP-MS operation were:

- Power: 1550 W
- Plasma Gas: 15 L/min.
- Aux Gas: 0.2 L/min.
- Nebulizer: 0.8 L/min.
- Sampling Rate: 0.3 mL/min.

Results

Macroelements

The elements with the highest concentrations overall in the lipstick were aluminum, calcium, titanium, silicon, and potassium. The lipstick samples contained over 10,000 ppm on average of each of these elements. Table 2 shows the average and maximum concentration levels for the macroelements examined.

Table 2. Results Macroelements (ppm)

Element	Average	Max
Al	38,283.69	360,790.97
Ba	2,734.56	26,494.60
Ca	28,248.93	345,753.07
Fe	8,262.9	63,177.23
K	11,917.92	128,129.69
Li	19.51	144.52
Lu	107.65	335.41
Mg	610.38	5,566.33
Mn	20.52	90.38
Na	380.01	2,962.92
P	13.36	97.81
Rb	22.94	82
Sc	3.9	101.37
Si	13,387.93	47,661.39
Sn	21.77	359.28
Sr	30.28	255.13
Ti	14,664.03	38,477.15
Zn	40.13	1,084.94
Zr	8.1	39.37

Concentration of Macroelements (ppm)

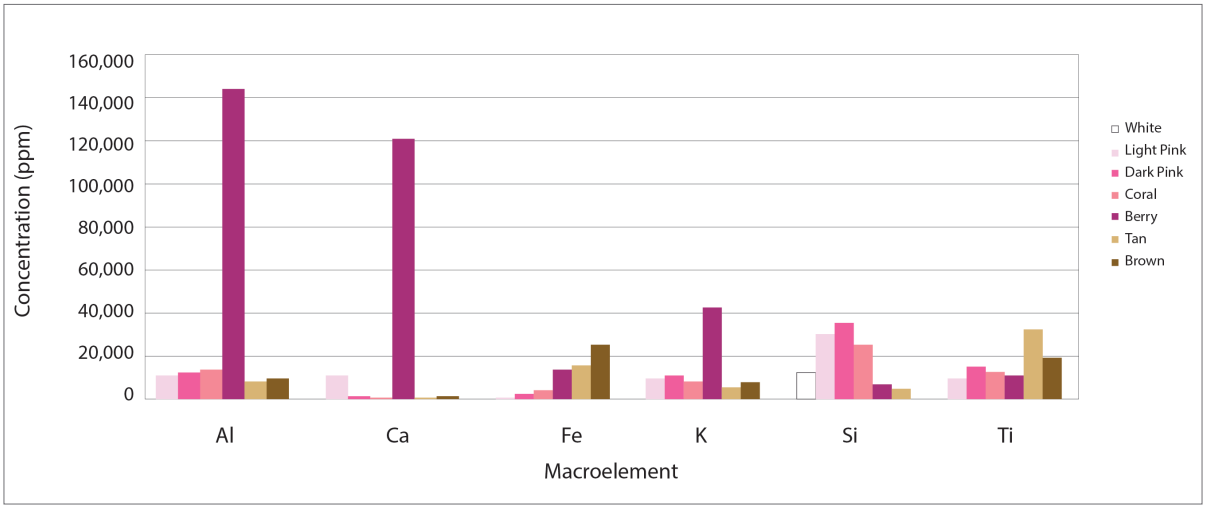

Figure 1. Concentration of each macroelement in each shade of lipstick

Some correlations were found between color groupings and finish type with some of the macroelements. The darker colors contained higher concentrations of the macroelements. The brown grouping had high concentrations of manganese and potassium and lower concentrations of silicon and zirconium. The brown colors contained the highest concentration of iron and zinc. Wine or berry colors contained higher concentrations of aluminum, calcium, silicon, zinc, potassium and manganese. The highest overall concentrations of aluminum, calcium and magnesium were found in the berry colors. Pink colors contained the highest concentrations of silicon. White or light colors contained the highest levels of zirconium.

The metallic finish types were found to have high concentrations of magnesium, iron, aluminum, and potassium. Matte finish types had higher concentrations of silicon, iron and zirconium but low concentrations of aluminum and magnesium. Pearlescent finishes contained higher amounts of silicon and magnesium but low amounts of iron, potassium and aluminum.

Trace Elements

Small trace amounts of a wide variety of elements were found in the lipstick samples and included a variety of precious metals. Precious metals such as silver, gold, platinum, and palladium were found in the range of 0.1 to 0.7 ppm. These elements were predominantly found exclusively in one lipstick brand.

Silver was found in concentrations up to 0.2 ppm in peach colors and pearl finishes. Gold was found in brown and berry colors with predominantly metallic finishes in levels up to 0.2 ppm. Platinum was measured up to 0.7 ppm in white or brown colors. Platinum was not found in berry colors. Palladium was found to be the lowest concentration of the precious metals up to 0.1 ppm in berry and dark pink colors.

Table 3. Results Trace Elements (ppm)

Element	Average	Max
Ag	0.04	0.21
Au	0.02	0.22
Cu	1.47	7.2
Ga	2.01	4.28
Gd	0.03	0.15
Ge	0.25	1.37
Hf	0.15	1.13
In	0.07	0.95
Ir	0.47	3.62
La	0.4	1.29
Nd	0.07	0.68
Pd	0.02	0.1
Pr	0.01	0.1
Pt	0.03	0.74
Re	0	0.04
Rh	0.03	0.21
Se	0.13	0.52
Sm	0.03	0.13
Ta	0.86	1.9
Th	0.06	0.27
W	3.04	8.63
Y	0.07	0.26
Yb	0.01	0.04

Toxic Elements

Eleven potentially toxic elements were examined in lipstick (Table 4) and found to be in the ppm range. Three additional toxic elements, cadmium, mercury and uranium, were found to be in the low ppb concentration range.

Table 4. Results Toxic Elements (ppm)

Element	Average	Max
As	0.3	0.69
Be	0.49	1.23
Co	0.44	4.3
Cr	2.44	31.45
Cs	1.68	4.76
Mo	0.27	4.12
Ni	2.88	23.36
Pb	0.96	2.39
Sb	0.33	9.58
Tl	0.08	0.23
V	4.68	50.72

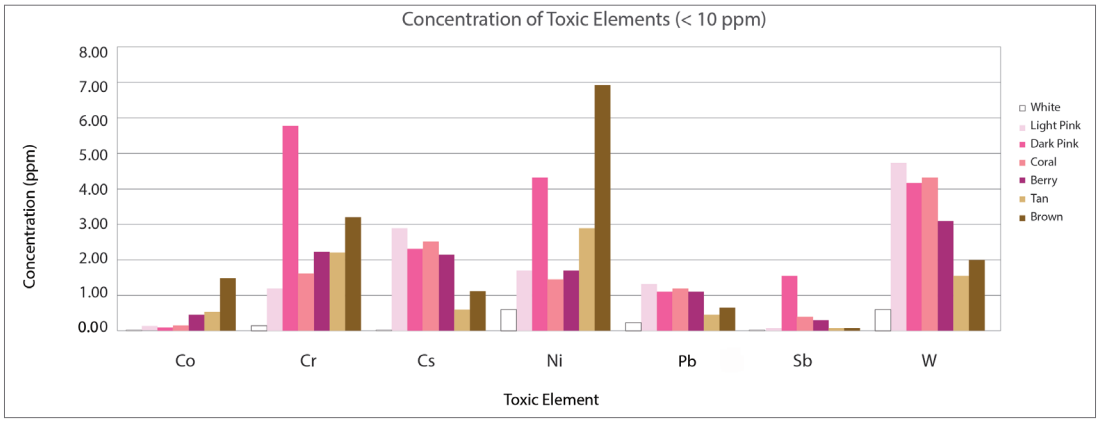

Figure 2. Concentration of each toxic element (< 10 ppm) in each shade of lipstick

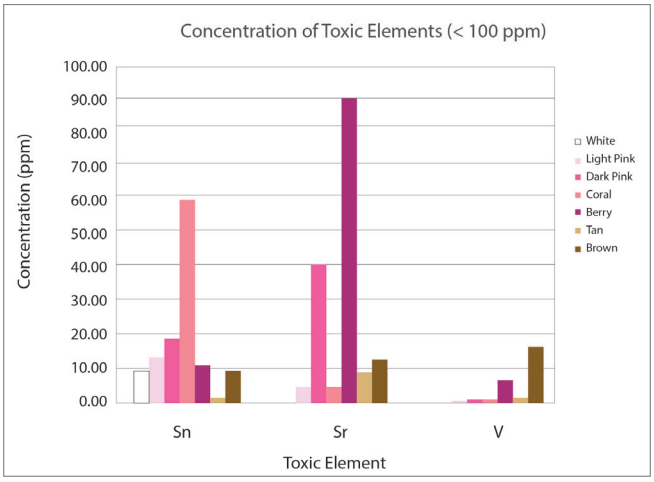

Figure 3. Concentration of each toxic element (< 100 ppm) in each shade of lipstick.

Prior studies of lipstick reported up to 3 ppm of lead found in lipstick. The maximum concentration of lead from samples in this study was 2.4 ppm with an average concentration of 1 ppm.

As was found in the macroelements, the toxic metals had some correlations in regards to color and finish. The darker colors, such as browns and berries, contained higher concentrations of arsenic and vanadium. The highest lead levels were found in the lighter colors and the red hued shades, such as pinks and corals. The brown colors and white or clear colors contained smaller concentrations of lead. Lead was also found in the highest concentration in the metallic and pearl finishes.

Conclusions

The lipsticks studied contained a wide array of elements from percentage levels of macroelements such as aluminum and calcium to small amounts of precious metals such as gold, silver, palladium, and platinum. Lead has been the concern in previous studies of lipstick. The maximum level of lead found in this study (2.4 ppm) was within the range found in previous work. Some correlation was found between lead levels and pink colors but a larger sample set would have to be tested to validate the correlation. Other potentially toxic elements were found in lipstick at ppm levels.

Despite the ppm concentrations of potentially toxic elements, the overall potential exposure to the consumer is relatively low. The average application of lipstick is less than 100 µg. Even if a consumer were to apply multiple applications up to 0.1 g in a day, the amount of exposure would be minimal. In the case of lead, the maximum concentration found was 2.4 µg/g. A total daily use of 0.1 g of lipstick would be 0.24 µg or 0.009% of the allowable daily exposure according to the EPA reference dosages (RfD) for lead. A person would have to consume almost forty tubes of lipstick with the maximum lead levels to meet or exceed recommended reference dosage levels.

References

1. Hepp, N. M., Mindak, W. R., and Cheng, J., Journal of Cosmetic Science, Vol. 60, No. 4, July/August, 2009.

Additional Resources

To watch our webinar on The Analysis of Lipstick for Toxic Elements, visit our YouTube channel at www.youtube.com/spexcertiprep. For additional product information, please contact us at +1.732.549.7144 or via email at USMet-CRMSales@spex.com.