

Metals and arsenic in eye shadows

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The regulations relating to cosmetic products give no limit values for toxic elements such as metals or arsenic occurring as impurities in cosmetic products. The present study of metals (lead, cobalt, nickel, chromium) and arsenic in eye shadows in 88 colors of 25 brands and 49 products provides a basis for assessing the safety of eye shadow. 66 out of 88 (75%) of the colors contained more than 5 ppm of at least one of the elements, and all 49 products contained more than one 1 ppm of at least 1 of the elements. In one color, the amount of all elements was less than 1 ppm. The highest levels of cobalt and nickel were 41 and 49 ppm, respectively. These levels were high enough to cause allergic symptoms in those previously sensitized. Furthermore, long-term exposure to such levels may probably sensitize. The concentrations of arsenic were extremely low, 2.3 ppm at most. The level of lead was less than 20 ppm in all products. Accordingly, the concentrations of arsenic and lead seemed to be safe. 9 colors had concentrations of water-soluble chromium exceeding 2 ppm, and a very high level, 318 ppm, was encountered in 1 case. The overall results indicate that eye-shadows probably have no significant systemic toxicological effects. The groups at greatest risk are those already sensitized to the allergenic elements. Such consumers will have difficulties in choosing suitable products, since these elements in the form of impurities are not declared in the list of ingredients. Manufacturers should demand certification that the raw materials they buy contain no toxic elements. Although some of the products studied were acceptable, many had excessive levels of the elements from the consumer's viewpoint.

Key words: heavy metal; nickel; cobalt; lead; chromium; arsenic; eye; cosmetics; allergic contact dermatitis; safety assessment. © Munksgaard, 2000.

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In the manufacture of colors, toxic elements such as metals and arsenic are retained in pigments. Eye shadow is a typical example of a group of cosmetic products in which the significance of pigments is great. Some toxic elements and their compounds are water-soluble and moist skin can therefore promote the percutaneous absorption of elements occurring as impurities in pigments. The excipients used also affect the absorption through the skin (1, 2). On the other hand, eye shadow is applied dry to the skin, lowering the risk of percutaneous absorption of pigments and thus also of toxic elements. The skin of the eyelids is thin and eczemas of the eyelids are common. Absorption of elements into the circulation is possible but percutaneous absorption of elements is slower than that of fat-soluble substances (3).

Some elements and their compounds may cause skin allergy or even cancer (1, 4–15). According to the Finnish Resolution of the Ministry of Labour

(838/1993) (4), lead (5), arsenic (6), nickel and chromium compounds (trivalent and hexavalent) may present a carcinogenic risk at high concentrations (8–15).

Nickel is one of the commonest causes of allergic contact dermatitis (16), and the prevalence of nickel allergy is on the increase (17). The first case of nickel allergy caused by eye shadow was reported about 10 years ago (18). Minimal amounts of heavy metals, even 1 ppm of nickel, may trigger a pre-existing allergy (19). It is therefore recommended that cosmetic products should not contain more than 5 ppm of metals as impurities (19). Nickel and cobalt allergy usually occur in the same individual because nickel compounds contain cobalt as an impurity (20).

The aim of this study was to determine the concentrations of lead, cobalt, nickel, total and water-soluble chromium and arsenic in samples of eye shadow, and to assess the safety of eye shadows on the basis of the results.

Materials and Methods

Samples were acquired in stores in Helsinki in October 1997. 2 eye shadows were randomly selected from each cosmetic series.

Processing of samples

The samples were processed using acid-washed containers and devices. The 65% nitric acid and 30% hydrochloric acid used for digestion were of Suprapure quality (Merck Darmstadt, Germany). The lead, cobalt, nickel and chromium standards were prepared from commercial 1000 ppm Merck Titrisol solutions (Merck Darmstadt, Germany). Other reagents were of analytical grade quality. The water used was ultra-pure water (distilled and treated using the MilliQPlus system, Millipore Corporation, USA).

Lead, total chromium, cobalt, nickel and arsenic

Approximately 150 mg of each sample was accurately weighed into a beaker, into which 5 ml of strong nitric acid and 5 ml of strong hydrochloric acid were added. The samples were covered with a watch-glass and heated slowly in a sand bath nearly to dryness. After cooling 2.5 ml of both acids were added, and the samples were re-heated until the production of acid fumes ceased. The residues were dissolved in an ultrasonic bath for 30 min into 2 ml of 65% nitric acid and rinsed with water into 10 ml volumetric flasks filling to the mark.

Water-soluble chromium

Water-soluble chromium was determined in the samples with total chromium exceeding 10 ppm. Approximately 100 mg of each sample were accurately weighed into a test tube into which 5 ml of water was added. The samples were kept in a water bath at +80°C for 1 h and thereafter in an ultrasonic bath for 15 min at room temperature. Finally the samples were centrifuged and filtered. Determinations were carried out on the filtrate.

Methods of analysis

The samples were analyzed by electrothermal atomic absorption spectroscopy using Zeeman background correction, matrix modification and pyrolytic tubes with wall or platform atomization indicated in Table 1 utilising the concept of stabilized temperature platform furnace (STPF) introduced by Slavin et al. (21). The STPF conditions and matrix modifiers used were based on the methods giving reliable results in analysing several

elements, even easily volatilizing arsenic in such difficult matrixes as in urine, blood, milk powder and other feedstuff (22–24). A Perkin-Elmer Zeeman 3030 AAS (Perkin-Elmer & Co, GmbH, Germany) instrument was used for the analysis of lead, arsenic, total (Cr_{tot}) and water soluble chromium (Cr_{ws}), and a Perkin-Elmer Zeeman 5100 AAS (Perkin-Elmer & Co, GmbH, Germany) instrument for the analysis of cobalt and nickel.

Each element was determined from the sample solutions by means of 2 parallel assays using appropriate dilutions with nitric acid solution (2 ml of 65% nitric acid and 8 ml of water) or water for soluble chromium to keep each sample within the linear range of calibration curve (Table 2). Quality control procedures included reagent blank analyses from which the detection limits of methods were determined as the mean value plus $3 \times$ the standard deviation (SD). The precisions were calculated as the means of relative standard deviation (RSD) of duplicate analyses of 20 samples. The accuracy was tested by analysing the recoveries from the Standard Reference Material 1572 (SRM 1572), Citrus Leaves (NIST, National Institute of Standards and Technology, USA). The concentration of cobalt in the reference material was too low for accuracy testing and the concentration of water-soluble chromium was not notified. Hence, the recovery of cobalt was tested by analysing 2 samples of an international comparison programme (Proficiency testing for measurements of elements in workplace air filters, National Institute of Occupational Health, Norway) showing a mean recovery and SD of $95 \pm 0.4\%$ from the reference value. For water-soluble chromium recovery experiments were carried out by analysing 2 duplicate samples spiked with potassium dichromate (3.3 and 6.6 $\mu\text{g/g}$) giving a mean recovery and SD of $92 \pm 7\%$.

Results

2 eye shadows were randomly selected for analysis from each cosmetic series (Table 3). Each product was given a running number (Table 3), but a single product may contain several colors which have been identified with the letters a, b, c and d. Altogether the study covered 88 colors of 25 brands and 49 products.

The concentrations of the metals and arsenic in the eye shadows are presented in Table 3. 66 out of 88 (75%) of the colours contained more than 5 ppm of at least one element, and all 49 products contained more than 1 ppm of at least one element. In one color the amount of all elements was less than 1 ppm.

The lowest value for lead was below the detection limit (6 colors), the highest value was 16.8

ppm (Lumene, color 34 a). In the majority of the products, the lead content was close to a few ppm. Arsenic was detectable in 15 colors, although in minute quantities (highest value 2.3 ppm).

There was no cobalt (i.e., the amount was below the determination limit) in 2 colors (Pierre Robert, color 7a, Yves Saint Laurent, color 48 c). The highest levels of cobalt were found in the products of Isadora (product 22) (41 ppm), Christian Dior (product 12, a, c) (26 and 29 ppm) and Joe Blasco (product 24) (25 ppm). Approximately 25% of the eye shadows contained less than 1 ppm of cobalt.

The highest nickel contents were measured in the products of The Body Shop (product 3) (42 ppm), Christian Dior (colour 12 a) (40 ppm), Isadora (product 22) (49 ppm) and Joe Blasco (product 24) (44 ppm). The levels of nickel were generally higher than those of cobalt.

The chromium content was surprisingly high in some products. Chromium had presumably been used as a pigment in the products with the highest concentrations. The total chromium content in Soho 13 (product 45) was 5470 ppm, and in Lancome product 31 b and c, 2680 ppm and 2370 ppm, respectively. The levels of soluble chromium were determined only in products with total chromium content exceeding 10 ppm. 9 of the 88 colors contained more than 2 ppm of soluble chromium, but only 2 more than 10 ppm. The highest content of

soluble chromium, 318 ppm, was found in Soho 13 (product 45).

Discussion

The present report is a continuation of a series of studies related to the safety of cosmetics for consumers (25–27). Eczema of the eyelids is common and may be caused by cosmetics. Mascara, eyeliner, eye shadow, and eyebrow pencil or powders are the most commonly used eye make-ups (15, 28). The metals which may be present in the coloring agents are often overlooked as causes of allergic contact dermatitis of the eyelids.

Eye shadow should not be applied to broken skin. Eye shadows contain particles which differ in size and have various types of edges. The eyelids can therefore be mechanically broken, enabling the penetration of pigments and thus also of metals into the body. Metals are also absorbed into the organism through healthy skin (2, 20, 29).

The regulations relating to cosmetic products give no limit values for toxic elements such as metals or arsenic occurring as impurities in cosmetic products. It has been proposed that the levels of nickel or cobalt should not exceed 5 ppm and should preferably be below 1 ppm (1,19). The levels of arsenic and lead should not exceed 5 and 20 ppm (30), respectively, and the level of water-

Table 1. Instrumental parameters and matrix modification used in the study

Analyte	Wavelength (nm)	Slit (nm)	Sample volume (μl)	Matrix modifier mass/volume	Tube/atomization
lead	283.3	0.7	10	0.2 mg/5 μl PO ₄ and 0.01 mg/5 μl Mg(NO ₃) ₂	pyrolytic/platform
Cr _{tot}	357.9	0.7	10	0.05 mg/5 μl Mg(NO ₃) ₂	pyrolytic/wall
Cr _{ws}	357.9	0.7	10	0.05 mg/5 μl Mg(NO ₃) ₂	pyrolytic/wall
arsenic	193.7	0.7	10	0.05 mg/5 μl Ni(NO ₃) ₂	pyrolytic/platform
cobalt	242.5	0.2	10	0.05 mg/5 μl Mg(NO ₃) ₂	pyrolytic/platform
nickel	232.0	0.2	10	0.05 mg/5 μl Mg(NO ₃) ₂	pyrolytic/platform

The samples were analyzed by electrothermal atomic absorption spectroscopy using Zeeman background correction, matrix modification and pyrolytic tubes with wall or platform atomization (Cr_{tot}=total chromium; Cr_{ws}=water-soluble chromium).

Table 2. Analytical quality parameters of the methods: linear calibration range, detection limit (mean of blanks+3 times SD), precision (RSD, relative standard deviation of 20 samples), accuracy (recovery from standard reference material)

	Linear calibration range (μg/l)	Detection limit (μg/g)	Precision (RSD) mean±SD (%)	Accuracy (SRM 1572)	
				recovery (μg/g)	certified (μg/g)
lead	10–300	0.5	1.8±1.4	11.2	13.3±2.4
Cr _{tot}	3–50	0.3	2.1±1.7	0.79	0.8±0.2
Cr _{ws}	3–50	0.25	3.2±4.9	–	*
arsenic	2–100	0.3	1.8±2.3	3.08	3.10±0.3
cobalt	3–100	0.5	2.2±1.5	–	0.02**
nickel	3–200	0.5	2.0±1.9	0.61	0.6±0.3

Cr_{tot}=total chromium; Cr_{ws}=water-soluble chromium.

* Not available.

** Not determined, too low for the method.

Table 3. The products studied, and the metals and arsenic found in them

Product and number of color	Lead $\mu\text{g/g}$ (ppm)	Cobalt $\mu\text{g/g}$ (ppm)	Nickel $\mu\text{g/g}$ (ppm)	Cr _{tot} $\mu\text{g/g}$ (ppm)	Cr _{ws} $\mu\text{g/g}$ (ppm)	Arsenic $\mu\text{g/g}$ (ppm)
1. Arcancil 147 Chataigne	1.7	5.9	7.6	5.6		<0.3
2. Arcancil 145 Astrakan	1.0	24.6	26.1	4.2		<0.3
3. The Body Shop Colourings 24 Damson	1.5	22.6	42.4	54.6	11.3	<0.3
4. The Body Shop Colourings 37 Olive	0.9	12.8	12.9	272	8.8	<0.3
5. Chanel/90 Planètes/Les 4 ombres						
a) (brown)	1.7	11.4	21.1	8.0		1.0
b) (brown)	3.0	4.1	11.0	36.1	0.6	2.3
c) (light brown)	2.2	1.9	14.9	49.5	<0.25	1.6
d) (chocolate brown)	0.9	8.9	12.8	4.7		<0.3
6. Chanel/25 Variations/Les 4 ombres						
a) (chocolate brown)	0.8	19.3	22.3	6.6		<0.3
b) (light brown)	3.2	0.5	2.1	2.8		1.7
c) (brown)	2.7	1.2	30.5	68.5	2.4	<0.3
d) (golden brown)	1.9	0.5	16.5	43.4	<0.25	<0.3
7. Pierre Robert						
a) 19 Sky Blue	1.5	<0.5	0.5	3.4		<0.3
b) 20 Sandy Beige	1.7	0.6	1.3	4.7		<0.3
8. Clarins Les Ombres Duo Multi Éclat 09						
a) Champagne / Bois Précieux	0.7	0.7	0.5	3.5		<0.3
b) Seashell/Mahogany	0.7	8.2	21.3	15.7	<0.25	<0.3
9. Clinique 78 Cloud	1.9	0.5	<0.5	2.6		0.8
10. Clinique 89 Peach silk	1.3	0.5	0.8	6.1		0.7
11. Christian Dior 864 Rosewood and Plum						
a) Rose Teck/Teak Rose	2.7	14.3	16.5	5.4		0.6
b) Rose Cedre/Cedar Rose	2.4	6.8	13.5	6.3		0.4
c) Rose Bambou/Bamboo Rose	2.6	6.9	5.3	5.1		0.8
d) Blanc Rose/Shell Rose	1.8	0.6	1.9	4.7		<0.3
e) Eyeliner Effet Prune/Plum	3.0	15.0	39.5	41.0	<0.25	<0.3
12. Christian Dior 714 Browns and Blue						
a) Chene/Oak	1.7	26.9	40.6	7.4		0.9
b) Brun Cendre/Ash Brown	2.1	16.8	25.6	57.5	<0.25	1.3
c) Liege/cork	1.8	29.6	37.1	3.8		1.2
d) Ivoirine/Ivoire	2.0	1.0	0.7	2.8		<0.3
e) Effet Bleu/Blue	2.8	2.6	15.4	47.3	<0.25	<0.3
13. Estée Lauder Compact Disc Orange2 42	0.8	0.8	0.9	1.2		<0.3
14. Estée Lauder Compact Disc Brown8 56	0.7	3.2	25.4	45.1	<0.25	<0.3
15. Givenchy Diamant 1						
a) (light)	1.2	0.8	0.6	0.6		<0.3
b) (reddish-brown)	1.3	0.9	1.5	1.6		<0.3
c) (grey)	1.4	3.5	3.1	4.3		<0.3
d) (brown)	1.3	5.3	6.1	7.9		<0.3
e) (white)	0.9	0.7	1.2	2.8		<0.3
16. Givenchy Onyx 5						
a) (bluish-grey)	0.6	0.8	2.9	3.9		<0.3
b) (mid grey)	1.1	7.4	5.3	2.1		<0.3
c) (grey)	1.0	13.8	14.9	2.1		<0.3
d) (dark grey)	1.2	21.6	32.4	8.4		<0.3
e) (white)	0.7	0.8	3.0	12.9	<0.25	<0.3
17. Guerlain Beige No 1	1.1	0.5	1.4	4.0		<0.3
18. Guerlain Cuivre No 5	1.3	1.1	10.8	12.2	<0.25	<0.3
19. Helena Rubinstein Colour Fusion Duo 01 Sheer Kaki Duo						
a) (brown)	0.9	3.0	6.3	238	2.6	<0.3
b) (greenish-yellow)	0.5	2.8	4.1	38.0	<0.25	<0.3
20. Helena Rubinstein Colour Fusion Mono 06 Black Spell	<0.5	11.7	18.6	19.0	<0.25	<0.3
21. Helena Rubinstein Loose Eye Colour 04 Red Devil	2.7	15.6	23.7	4.7		<0.3
22. Isadora 57 Soft Black	<0.5	41.2	49.7	18.2	<0.25	<0.3
23. Isadora 52 Gold	1.1	1.5	4.9	7.9		<0.3
24. Joe Blasco True Brown	<0.5	25.6	44.8	16.1	1.4	<0.3
25. Joe Blasco Periwinkle	3.0	3.4	14.8	92.0	<0.25	0.9
26. Juvena Stylo-duo Dusty Coral 10 74887						
a) (brown)	1.2	4.0	5.0	3.2		<0.3
b) (greyish-brown)	0.7	2.8	3.1	0.4		<0.3

Table 3 (cont'd)

Product and number of color	Lead $\mu\text{g/g}$ (ppm)	Cobalt $\mu\text{g/g}$ (ppm)	Nickel $\mu\text{g/g}$ (ppm)	Cr _{tot} $\mu\text{g/g}$ (ppm)	Cr _{ws} $\mu\text{g/g}$ (ppm)	Arsenic $\mu\text{g/g}$ (ppm)
27. Juvena Stylo-duo Flanell Grey 60 74892						
a) (blue)	1.3	16.3	15.0	2.4		<0.3
b) (light brown)	0.6	2.7	3.3	0.3		<0.3
28. Kanebo Bio BE-89	2.6	1.0	6.8	11.1	<0.25	<0.3
29. Kanebo Bio BE-6	1.4	7.5	23.7	52.8	<0.25	<0.3
30. Lancome Ombre subtile Duo 10 Brun d'écorce						
a) (brown)	1.0	17.1	20.4	6.8		<0.3
b) (light brown)	1.0	2.1	4.4	10.0	<0.25	<0.3
31. Lancome Ombre subtile Trio 58 Bleus planète						
a) (dark bluish-grey)	1.0	22.8	20.6	15.8	<0.25	<0.3
b) (light blue)	1.0	9.4	8.0	2680	3.2	<0.3
c) (turquoise)	0.9	1.1	1.6	2370	4.6	<0.3
32. L'Oréal Perfection 20-Ciel	1.0	0.5	1.2	1.0		<0.3
33. L'Oréal Perfection 43-Doré	0.7	0.5	1.2	1.5		<0.3
34. Lumene 19 Harsopilvi, duo						
a) (yellowish-brown)	16.8	2.2	3.6	3.0		0.9
b) (blue)	14.7	0.8	3.5	8.5		<0.3
35. Lumene 20 Iltaisin, duo						
a) (green)	16.8	0.5	1.0	16.4	0.3	0.4
b) (brown)	10.4	3.2	6.0	5.8		<0.3
36. Max Factor 101 Pale Pebble	0.7	0.5	0.8	1.1		<0.3
37. Max Factor 105 Terra Firma	0.7	4.8	19.2	14.8	<0.25	<0.3
38. No7 trio 05 Rum Barley						
a) (brown)	0.9	3.6	6.2	5.0		<0.3
b) (light brown)	1.2	4.3	14.6	4.1		<0.3
c) (reddish-brown)	0.6	5.6	22.0	11.7	<0.25	<0.3
39. No7 trio 30 Willow Moss						
a) (brown)	<0.5	13.8	20.3	8.7		<0.3
b) (light brown)	0.5	3.2	3.5	0.7		<0.3
c) (green)	0.8	7.1	6.6	114	2.5	<0.3
40. Revlon 60 Frosty White	1.9	0.7	1.6	3.4		<0.3
41. Revlon 30 Vintage Wine	2.0	2.2	13.9	37.4	<0.25	0.3
42. Rimmel 12 Blueberry Ice	1.1	4.9	1.5	2.9		<0.3
43. Rimmel 56 Matte Sage	<0.5	3.1	5.0	6.6		<0.3
44. Soho 12	0.9	4.4	8.8	19.0	8.5	<0.3
45. Soho 13	1.2	0.6	1.9	5470	318.0	<0.3
46. Yves Rocher Outremer 04032	1.1	0.9	3.7	15.7	1.0	<0.3
47. Yves Rocher Acajou 02687	0.5	1.0	5.5	3.0		<0.3
48. Yves Saint Laurent Duo/Enlumineur 3						
a) (brown beige)	<0.5	2.7	7.2	7.3		<0.3
b) (brown)	<0.5	4.0	7.2	4.7	<0.25	<0.3
c) (champagne)	<0.5	<0.5	0.5	0.4		<0.3
49. Yves Saint Laurent Duo 11						
a) (golden brown)	1.0	1.9	4.8	5.7		<0.3
b) (brown)	0.9	13.7	41.1	18.1		<0.3
Detection limit	0.5	0.5	0.5	0.3	0.25	0.3

(Cr_{tot}=total chromium; Cr_{ws}=water-soluble chromium).

soluble chromium should preferably be less than 2 ppm (12). The last paper simulates the situation when cement, containing water-soluble chromium, comes into contact with the skin. Therefore this limit value for water-soluble chromium may be used as the limit in cosmetics.

Our results indicate that eye shadows are not likely to cause systemic effects. The contents of arsenic were very low and those of lead less than 20 ppm. These are clearly lower than the limit values given (30). The levels of cobalt and nickel at their highest (nearly 50 ppm) were fairly low as regards systemic toxicological effects.

Water-soluble chromium can be absorbed through the skin and high levels can cause undesirable effects on the skin. In some products, chromium compounds had presumably been used as actual pigments. The levels of chromium compounds in the form of impurities varied considerably. Since water-insoluble chromium does not affect the skin as does water-soluble chromium, and the products include also insoluble chromium, the levels of soluble and total chromium were determined separately. 9 colors had concentrations of water-soluble chromium exceeding 2 ppm, and a very high level, 318 ppm, was encountered in 1

case. The authorities are negotiating with the importer about the possible risks of the product.

It is difficult to estimate whether a person not previously sensitized to nickel or cobalt might acquire an allergy from the products studied. In individuals sensitized to these metals, concentrations as low as 1 ppm of nickel or cobalt may cause an allergic reaction (1). No product contained less than 1 ppm of the elements examined. Only 30% of the eye shadows examined (25/88) contained less than 5 ppm of each element. Contents as high as these have not previously been found in cosmetic products (1, 30, 31) but earlier studies on eye shadows are relatively few.

Consumers cannot determine which products contain metals by reading the labels. Individuals previously sensitized to metals should completely avoid the use of eye shadow. On the other hand, manufacturers should pay more attention to the color purity requirements. Guidelines on the good manufacturing practice of cosmetic products require the absence of harmful levels of impurities in the ingredients.

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