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Determination of Heavy Metals in Eyeliner, Kohl Samples

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It is well known that heavy metal impurities in cosmetic products are unavoidable due to the ubiquitous nature of these elements, but should be removed wherever technically feasible. Heavy metal concentrations in cosmetic products are seen to be technically avoidable when they exceed a certain limits. In this work we chose eight sample of the most expensive eyeliner, kohl brand from the Saudi market. Twenty eight elements were determined by using inductively coupled plasma mass spectrometer (ICP-MS) and a flow injection mercury system (FIMS). Most of the elements concentration were found in the normal level but aluminum and nickel were high in some samples.

Key Words: Cosmetic, Eyeliner, Kohl, Heavy metal.

INTRODUCTION

Most of people specially females use cosmetic, personal care products (PCP) and their ingredients on a daily basis. Although human external contact with a substance rarely results in its penetration through the skin and significant systemic exposure, personal care products produce local (skin, eye) exposure and are used in the oral cavity, on the face, lips, eyes and mucosa. Therefore, human systemic exposure to their ingredients can rarely be completely excluded. In addition, natural and synthetic substances may produce local effects in human skin, such as irritation, sensitization or photoreactions. Given the significant and relatively uncontrolled human exposure to personal care products, these products must be thoroughly evaluated for their safety prior to their marketing[1].

Kohl is a traditional eyeliner which has been widely used as an eye cosmetic in the Middle East, Far East and Northern Africa[2]. It is both used for beautification and as a traditional ethnic remedy to relieve eyestrain, pain, or soreness. In addition, Kohl is known to prevent sun glare, thus it was used by Bedouins in the Arab Peninsula. Previous studies[3-5] have shown that Kohl contains toxic heavy metals, such as lead and case studies have revealed that blood levels were significantly higher in individuals who used Kohl compared to ones who did not. It was also shown that blood levels in infants of Kohl using mothers were considerably higher than those of non-Kohl using mothers (5.2 µg/dl *versus* 2.8 µg/dl)[6]. However, there have been a few published reports where Kohl is mentioned. One is where the application of Kohl to a child's eyes and/or umbilicus at birth was found to be one of the causes of elevated blood lead levels in Saudi Arabian school girls[7].

Lead is harmful to all adults, children and infants. It is particularly harmful to the developing brain and nervous system[8]. Lead mainly enters the body through oral ingestion or inhalation of lead dust. Of lead that reaches the digestive tract, adults absorb *ca.* 11 % and children absorb 30-75 %. Less than 1 % of lead is known to be absorbed through the skin[9]. Lead poisoning is a global problem, considered to be the most important environmental disease in children[10]. Pregnant women and children under 6 years of age absorb lead in the highest quantities and even low levels of lead exposure are considered hazardous to pregnant women[11]. Lead exposure during the first trimester of pregnancy has been found to cause alterations in the developing retina, thus leading to possible defects in the visual system in future[12]. Lead poisoning has been linked to juvenile delinquency and behavioural problems. Young children are particularly susceptible to lead poisoning due to their normal hand-to-mouth activity and because of the high efficiency of lead absorption by their gastrointestinal tracts[13]. Chronic low-dose lead exposure was found to cause renal tubular injury in children[14], while in adults, it was associated with poorly controlled hypertension[15]. A blood lead level of 10 mg/dl is of concern[8]. Shaltout *et al.*[16] found 20 patients aged between 1 and 18 months suffering from lead encephalopathy in Kuwait. The blood levels in 19 children ranged between 60 and 257 mg/dl. Two of these patients died before starting treatment and three children died during treatment. Among the children who recovered, four had neurological sequalae. The source of lead in 11 patients was confirmed to be Kohl[16]. Recently, a 7-month-old baby was found to have a blood lead level of 39 mg/dl due to use of Kohl[17]. In the USA, Kohl and 'Kajal' from the middle east were considered among the unapproved dyes in eye cosmetics that contained potentially harmful amounts of lead[18]. Similarly, certain traditional digestive remedies also contain harmful levels of lead[17]. Little is known about lead poisoning in Saudi Arabia. Studies have suggested that Kohl in Saudi Arabia might be a cause of lead toxicity[7,19] but no detailed investigation has been undertaken.

In addition to lead, as a non-essential element, aluminium might also be toxic at both environmental and therapeutic levels[20-22]. Aluminium exposure, apart from causing cholinotoxicity, can induce changes in other neurotransmitter levels since neurotransmitter levels are closely interrelated[22]. Al-Saleh and Shinwari[23] highlighted the adverse developmental effects of aluminium on children and infants. Antimony, on the other hand, has been found to induce DNA strand lesions but not DNA-protein crosslinks[24]. Fumes from melting antimony cause dermatoses and skin lesions[25]. Bearing in mind the reports on aluminium and antimony toxicity and many alarming reports on the association of Kohl with lead poisoning in different countries, it was considered essential to examine the Kohl found in Saudi Arabia. In this work we chose eight sample of the most expensive eyeliner, Kohl brand from the Saudi market. Twenty eight elements were determined by using inductively coupled plasma mass spectrometer (ICP-MS) and a flow injection mercury system (FIMS).

EXPERIMENTAL

Sample preparation: Accurately weighed portion (0.1- 0.2 g) of the eyeliner of Kohl brnad was transferred to a Teflon digestion tube (120 mL) and 7.0 mL of the acid mixture (HNO_3/HF/HCl, 4.5:2:0.5) was introduced. The tube was sealed and the sample was digested inside a microwave oven (Milestone ETHOS 1600) following a heating program shown in Table-1.

TABLE-1
MICROWAVE HEATING PROGRAM USED FOR DISSOLUTION OF SAND, SOIL AND SEDIMENT SAMPLES

Step	1	2	3	4
Power (W)	400	0	300	400
Time (min)	15	2	10	15
Temp. (ºC)	195	195	195	195

After being cooled to ambient temperature, the tube was opened. The inside of the lid was rinsed with distilled and de-ionized water (DDW) and the mixture heated on a hotplate (120 ºC) for 0.5 h to drive off the residual HF and HCl. The resulting digest was filtered in a polypropylene flask using 1 % HNO_3 and made up to 50 mL volume. For ICP-MS measurement the clear digest obtained were diluted 10 times incorporating 10 μg L^{-1} solution of ^{103}Rh. In general, samples of the eyeliner, Kohl and standard reference materials (SRM) were prepared in a batch of six including a blank (HNO_3/HF/HCl) digest.

High purity water (DDW) (Specific resistivity 18 MΩ cm^{-1}) obtained from a E-pure water purification system (Barnsted, USA) was used throughout the work. HNO_3, HF and HCl used for sample digestion were of Suprapureâ grade with certified impurity contents and were purchased from Merck, Germany. A multi-element standard containing 27 elements were prepared from Perkin-Elmer single-element ICP standards (1000 or 10000 ppm). The standard reference material (SRM), IAEA-SOIL-7 was purchased from the International Atomic Energy Agency, Vienna.

Measurements were carried out by means of a Perkin-Elmer Sciex ELAN 6100 inductively coupled plasma mass spectrometer (ICP-MS). The instrument is equipped with a quadrupole mass filter, a cross-flow nebulizer and a Scott type spray chamber.

Quality assurance: To assess of the analytical process and make a comparative analysis, standard reference materials (soile 7) from the International Atomic Energy Agency (IAEA),Vienna, Austria was used. The quantitative analysis result is shown in Table-2. The results are generally in good agreement with certified values of the reference materials.

TABLE-2
CONCENTRATION OF ELEMENTS IN SOIL 7

Elements	Certified values 95 % Confidence interval in ppm	This work	
		ppm	rsd
Li	15-42	39.1	3.07
B	–	28.3	5.4
Na	2300-2500	2090	0.96
Mg	11000-11800	11200	1.05
Al	44000-51000	47900	0.287
K	11300-12700	11500	0.878
Ca	157000-174000	155000	1.09
V	59-73	73.7	0.982
Cr	49-74	62.8	3.33
Mn	–	648	1.13
Fe	25200-26300	25100	0.623
Co	8.4-10.1	12.4	4.32
Ni	21-37	17.2	2.22
Cu	9.0-13	11.2	1.16
Zn	101-113	115	0.0825
As	12.5-14.2	14	2.23
Se	0.2-0.8	1.3	34.6
Rb	47-56	50.2	0.327
Sr	103-114	102	1.35
Mo	0.9-5.1	1.03	3.47
Ag	–	0.484	3.3
Cd	1.1-2.7	1.13	0.726
Ba	131-196	131	1.36
Pb	55- 71	61.7	0.262
U	2.2-3.3	2.07	0.544
Sb	1.4-1.8	1.57	1.91
Sn	–	2.84	2.79

Hg analyses: A flow injection mercury system (FIMS) from Perkin-Elmer FIMS-400 was used for determination of Hg in eyeliner of Kohl brand.

The FIMS is a complicated technique depending up on synchronization of mechanical, chemical and optical operations. The system contain three major units namely the spectrophotometer coupled with the flow injection circuitry, the amalgamation unit and the computer unit for automated control of the operation and measurements. The FIAS program was optimized and the program is saved as "Mercury 2" in the computer (Table-3). The FIMS pumps program is shown in Table-4.

TABLE-3
THE FIMS PROGRAM

Method name	Mercury 2	Slit width	0.7 nm
Technique	FIAS-MHS	Read time	15.0 s
Wavelength	253.4 nm	Read delay	0.0 s
BOC time	2.0 s	Signal type	AA
Measurement	Peak height	Calibration	Linear, zero intercept

The blank used in this process contained 2 v/v % H_2SO_4, 2 v/v % HNO_3 and *ca.* 1.0 mg L^{-1} $KMnO_4$ in de-ionized water. All the measuring standard and sample solutions were stabilized in the same medium.

RESULTS AND DISCUSSION

Acceptable limits for heavy metals vary according to the subpopulation of interest (*e.g.* children are more susceptible to heavy metal toxicity than adults; have greater exposure potential due to hand-to-mouth activity); the amount of product used and the site of application (*e.g.* arms *vs.* lips). Assessment of dermal absorption by a single component in a cosmetic product is complex[26,27] and depends on factors such as the concentration in the product, the amount of product applied, the length of time left on the skin and the presence of emollients and/or penetration enhancers in the cosmetic product[27]. Given this complexity and the lack of well-conducted dermal absorption studies incorporating these factors, determination of heavy metal limits in cosmetics based on human health risk alone is a challenge.

There are currently no international standards for impurities in cosmetics. Limits have been established in Germany[28]. Rather than taking a risk-based approach, the German limits are based on levels that could be technically avoided. Thus, heavy metal impurities were limited to anything above normal background levels.

The German Federal Government conducted tests to determine background levels of heavy metal contents in toothpastes and other cosmetic products. Based on their studies, it was determined that heavy metal levels in cosmetic products above the values listed below are considered technically avoidable[28]: lead: 20 ppm, arsenic: 5 ppm, cadmium: 5 ppm, mercury: 1 ppm, antimony: 10 ppm.

In addition, following a survey of its member companies, the German Industrial Association for Personal Care and Detergents Inc. confirmed that heavy metal contents in toothpastes are at least a decimal power lower than for other cosmetic products[28]. Therefore, the Commission for Cosmetic Products at the Federal Ministry of Health in Germany concluded that the following values are the maximum acceptable concentration for toothpastes: lead: 1 ppm, arsenic: 0.5 ppm, cadmium: 0.1 ppm, mercury: 0.2 ppm, antimony: 0.5 ppm.

In Germany, a program is in progress to obtain updated values for traces of heavy metals in cosmetics[29].

Health Canada has taken a similar approach in the establishment of heavy metal impurity limits, as the department has always maintained that impurities in cosmetics should be reduced to the extent that is technically feasible. A review and analysis of the results of heavy metal testing conducted in the Health Canada Product Safety Laboratory on a number of cosmetics sold in Canada lead to the determination of limits. Furthermore, comparison of conservative estimates of exposure to Canadians from use of cosmetics and the established tolerable intakes, demonstrated that these levels provide a high level of protection to susceptible subpopulations of consumers (*e.g.* children)[29].

It is acknowledged that heavy metal impurities in cosmetic products are unavoidable due to the ubiquitous nature of these elements, but should be removed wherever technically feasible. Heavy metal concentrations in cosmetic products are seen to be technically avoidable when they exceed the following limits: lead: 10 ppm, arsenic: 3 ppm, cadmium: 3 ppm, mercury: 3 ppm, antimony: 5 ppm.

These levels are based on background levels found in cosmetic products sampled in Canada and are in line with acceptable levels of impurities in other jurisdictions. In addition, comparison of conservative estimates of exposure to Canadians from use of cosmetics and the established tolerable intakes for these metals demonstrated that these limits provide a high level of protection to susceptible subpopulations of consumers (*e.g.* children)[29].

Levels of heavy metals in some facial cosmetics in some other parts of the world are shown in Table-5[30].

Table-6 show the concentration of 28 elements on the eyeliner of Kohl brand from the Saudi market. Comparing the results with the literature it is clear that lead, arsenic, cadmium, mercury and antimony level in the samples under investigation are within the normal level. The nickel concentration is high. It reach 80.5 ppm in sample C47 and the concentration range was 23.5 to 80.5 ppm. Aluminium is also high among the sample under investigation. The range of aluminium concentration was between 926 ppm in sample C38 and 38,700 ppm

TABLE-4
FIMS PUMPS PROGRAM

Step	Time	Pump 1 speed	Pump 2 speed	Valve position	Read	Heat	Cool	Argon
Pre-fill	8	100	40	Fill	–	–	X	X
Step 1	5	100	40	Fill	–	X	–	X
Step 2	25	100	40	Fill	–	–	X	X
Step 3	20	0	40	Inject	–	–	X	X
Step 4	20	0	40	Inject	–	–	X	X
Step 5	10	0	40	Fill	–	–	X	X
Step 6	20	0	40	Fill	X	X	–	–
Step 7	10	0	40	Fill	–	–	X	X
Step 8	1	0	0	Fill	–	–	–	–

Steps to repeat: 1 to 4; Number of repeats: 0

TABLE-5
LEVELS OF HEAVY METALS IN SOME FACIAL COSMETICS IN SOME OTHER PARTS OF THE WORLD (ND = NOT DETECTABLE)

Country	Class/Name of cosmetics	Pb	Cd	Ni	Fe	Zn	Ref.
Saudi Arabia	Henna	1.29-16.48 µg/g	–	–	–	–	31
Saudi Arabia, India, Middle East	Kohl, eyeliner pencils	2.9-100 % ND	–	–	–	–	32
Morocco, US, Mauritania, Pakistan, India, UK and Saudi Arabia	Kohl	0.6-50 %	–	–	46%	–	33
Bulgaria	Eye shadow, lipstick and powders eye shadows	ND-41.1 µg/g < 20 µg/g	–	1-49 µg/g	–	–	34,35
Oman and UAE	Bint al dhahab	~91 %	~0.05%	–	–	–	36
Bahrain	Suma and kohl surma kohl	< 0.16 % ~88 % ~53 %	–	–	–	–	37-39
Nigeria	Galena based kwali graphite-based kwali	58.8-62.4 % 23-32 µg/g	- 14-30 µg/g	–	0.98-1.2 % 0.43-0.46 %	–	40
Nigeria	Local eye shadows	–	–	–	6.15%	35%	41

TABLE-6
CONCENTRATION OF ELEMENTS ON THE EYELINER, KOHL SAMPLES (> DL = BELOW DETECTION LIMIT)

Element	Brand 1 (C2)		Brand 2 (C10)		Brand 3 (C18)		Brand 4 (C27)		Brand 5 (C38)		Brand 6 (C47)		Brand 7 (C56)		Brand 8 (C64)	
	Conc. (ppb)	rsd	Conc. (ppb)	rsd	Conc. (ppb)	rsd	Conc. (ppb)	rsd	Conc. (ppb)	rsd	Conc. (ppb)	rsd	Conc. (ppb)	rsd	Conc. (ppb)	rsd
Li	55400	1.8	96100	0.3	82000	0.7	13400	0.7	84200	3.0	115000	1.1	25300	1.4	42800	1.6
B	17000	2.5	38800	2.5	31200	1.8	18700	1.7	> DL	–	27300	2.1	32900	0.9	> DL	–
Na	751000	1.6	2020000	2.2	10300000	1.7	779000	0.3	213000	1.6	950000	1.8	8520000	1.4	488000	0.7
Mg	2820000	1.9	8770000	2.2	6960000	1.6	854000	1.1	30900000	0.7	7430000	1.8	7200000	1.5	7200000	0.8
Al	7020000	2.0	38700000	1.6	19100000	2.7	11400000	1.1	926000	1.5	26900000	2.6	21400000	1.4	5560000	0.3
K	3270000	0.8	20500000	1.6	4040000	2.1	5560000	1.0	62200	2.5	16700000	1.4	1700000	0.6	2410000	1.3
Ca	245000	4.5	193000	3.5	143000	8.2	> DL	–	397000	7.5	207000	5.0	421000	3.8	253000	2.2
V	438	2.6	5300	1.2	926	3.7	1230	1.7	804	1.7	4500	1.4	1800	0.5	284	2.0
Cr	10800	2.2	4480	3.6	5260	3.2	1590	5.0	3620	1.8	3200	2.1	4180	2.3	6570	2.3
Mn	275000	1.0	765000	0.6	482000	0.1	509000	0.9	512000	1.3	697000	1.5	494000	1.2	497000	0.7
Fe	1.73E+08	1.3	3.09E+08	1.4	2.03E+08	0.6	2.53E+08	0.5	1.73E+08	0.8	315000000	0.8	2.47E+08	1.1	2.11E+08	0.7
Co	21300	1.1	72100	0.6	48900	1.1	57300	0.6	6160	0.8	71700	0.8	49100	0.9	50600	0.8
Ni	35200	1.1	74000	0.9	50900	1.7	55700	1.3	23500	0.9	80500	0.5	55300	1.2	57900	0.7
Cu	679	5.8	8030	2.4	2110	0.7	> DL	–	4820	0.8	1980	3.9	4430	0.4	1390	2.4
Zn	29200	0.9	22500	1.8	12600	2.0	25600	0.8	5660	4.5	24600	0.6	15700	1.0	18000	1.0
As	811	7.1	2330	17.9	1440	8.2	1520	20.9	1290	1.7	2640	5.4	1910	9.3	1160	21.7
Se	37.7	–	613	–	223	–	57.5	–	426	12.5	608	–	268	–	83.4	–
Rb	23500	1.1	133000	0.1	37800	1.0	49300	0.3	715	1.1	12000	1.4	24100	1.1	17200	0.5
Sr	2820	1.2	6270	0.5	12700	1.0	1560	1.3	5570	1.2	3890	1.5	22300	1.9	2500	0.0
Mo	905	1.4	141	10.4	302	3.4	122	8.0	174	5.3	344	0.9	226	3.4	281	18.0
Ag	298	2.4	14	25.4	32	1.8	12.6	–	> DL	–	> DL	–	138	6.7	141	3.9
Cd	21.5	21.6	14.1	41.1	10.3	16.3	9.87	–	12.6	–	27.3	9.0	26.8	–	14.2	–
Ba	4690	1.4	77100	0.3	20100	0.4	22600	0.8	924	2.9	48800	1.1	32500	1.8	4420	0.9
Pb	1510	1.0	5790	0.5	2830	1.1	1710	0.2	82.3	5.4	7900	1.8	4250	1.4	1330	2.5
U	189	1.3	192	2.5	667	1.4	77.5	4.5	258	3.1	309	0.7	1260	0.7	146	3.7
Sb	42.5	5.8	22.9	9.3	48.7	5.5	> DL	–	4.24		24.8	13.1	111	10.4	16.5	–
Sn	29100	0.6	55600	0.7	24100	1.1	26500	1.2	17700	0.5	55500	0.5	24500	1.2	30600	0.4
Hg	> DL	–	0.04	–	> DL	–	0.04	–	0.03	–	0.096	–	42	–	12	–

in sample C10. Aluminium concentration in Kohl samples from Saudi Arabia was reach to 5570 ppm in one of the sample analyzed by Al-Ashban *et al.*[38].

Conclusion

The results of this study clearly demonstrated that most of the elements under investigation are in the normal level. The concentration of lead in the eyeliner of Kohl brand investigated in this work is low. The highest concentration of lead was 7.9 ppm in sample C47. Aluminum and nickel in some samples were high. The highest aluminum concentration was 38.700 ppm in sample C10. Nickel highest concentration was 80 ppm in sample C47.

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