

Cobalt release from inexpensive jewellery: has the use of cobalt replaced nickel following regulatory intervention?

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Background: Before the introduction of the EU Nickel Directive, concern was raised that manufacturers of jewellery might turn from the use of nickel to cobalt following the regulatory intervention on nickel exposure.

Objectives: The aim was to study 354 consumer items using the cobalt spot test. Cobalt release was assessed to obtain a risk estimate of cobalt allergy and dermatitis in consumers who would wear the jewellery.

Methods: The cobalt spot test was used to assess cobalt release from all items. Microstructural characterization was made using scanning electron microscope (SEM) and energy-dispersive spectroscopy (EDS).

Results: Cobalt release was found in 4 (1.1%) of 354 items. All these had a dark appearance. SEM/EDS was performed on the four dark appearing items which showed tin–cobalt plating on these.

Conclusions: This study showed that only a minority of inexpensive jewellery purchased in Denmark released cobalt when analysed with the cobalt spot test. As fashion trends fluctuate and we found cobalt release from dark appearing jewellery, cobalt release from consumer items should be monitored in the future. Industries may not be fully aware of the potential cobalt allergy problem.

Key words: cobalt allergy; cobalt dermatitis; cobalt ions; jewellery; screening; spot test. © John Wiley & Sons A/S, 2010.

Conflict of interests: Jacob P. Thyssen, Per Møller, and Morten S. Jellesen sold the cobalt spot test methodology, after its development, to SmartHealth (Phoenix, Arizona). The company did not participate in its development and had no influence on the study. The authors will receive royalty fee annual net sales of the spot test. Torkil Menné, Jeanne D. Johansen, Carola Lidén, and Anneli Julander have no conflict of interest to disclose.

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In response to the growing nickel problem in the 1970s–1980s (1), the Danish government and the EU began regulating nickel release from various consumer products in 1990 and 1994, respectively (2, 3). We have consistently shown that the prevalence of nickel allergy has decreased in Danish women and female patients with dermatitis after the regulation (4–9). We have also shown that the EU Nickel Directive, which entered into full force

in 2001, has led to a decrease in the prevalence of dimethylglyoxime (DMG) test positive consumer items in Sweden, and thus, a reduction in skin exposure to nickel after introduction of the Directive compared with the situation before its entry into force (10, 11).

In 1992, before the introduction of the EU Nickel Directive, concern was raised that manufacturers of jewellery might turn from the use of nickel

to cobalt following the regulatory intervention on nickel exposure (12). This would eventually result in higher prevalence of cobalt allergy caused by increased skin exposure to cobalt release from consumer products (13). In 2005, Hindsén et al. described a patient with allergic contact dermatitis caused by a brown-plated necklace that released high amounts of cobalt but not nickel (14). The authors discussed whether this was a unique case or whether similar cases should be expected in the future following the regulation on nickel exposure. In 2009, we published an article on nickel and cobalt allergy in Danish female patients with dermatitis (7). The prevalence appeared stable for cobalt allergy in the youngest age group (5–30 years), whereas a slight increase was observed in the age groups 30–49 years and >50 years. It was discussed whether the increasing prevalence of cobalt allergy among older patients to some degree could reflect the increasing prevalence of isolated cobalt allergy in this age group and also whether the increase could reflect a cohort effect as observed for nickel allergy (7). As the prevalence of cobalt allergy among young patients remained stable rather than decreasing (as observed for nickel allergy), it was suggested that cobalt exposure could possibly have increased over the observation period (7). We concluded that to further explore the cobalt issue, cobalt release from various consumer products should be investigated.

Recently, we developed, described, and applied a spot test for the detection of cobalt release from metallic items (15). We showed that the cobalt spot test was able to detect cobalt release from metallic items that had elicited positive patch test reactions in cobalt-allergic patients (15, 16). This study aimed to analyse 354 consumer items recently purchased in Copenhagen, Denmark, for a study on nickel exposure, using the cobalt spot test (17). This was performed to obtain a risk estimate of cobalt allergy and dermatitis in consumers who are in prolonged skin contact with these items. The current study is of relevance as it may increase our knowledge on the effects of regulatory interventions on nickel exposure and estimate the risk of cobalt allergy and dermatitis following skin contact with inexpensive jewellery.

MATERIALS AND METHODS

Purchase strategy

This study was performed between December 2008 and March 2009 in Copenhagen, Denmark, and has previously been described in detail (17). The study was initially performed to investigate the proportion of inexpensive jewellery and hair clasps that released an excessive amount of nickel but

at the time of purchasing, we had the intention of investigating the jewellery for cobalt release as well. When buying earrings, we mainly looked for inexpensive metallic pieces with perforating piercing posts (hoops, studs, and drops). However, metallic finger rings, bracelets, necklaces, and hair clasps were also randomly purchased.

We spent approximately 3000 € (range 5–50 €) buying 354 metallic jewellery items and hair clasps from a total of 36 stores, street vendors, and jewellery Web pages. The great majority of purchased jewellery was intended for girls and women. The accumulation of jewellery and hair clasps was believed to be representative of possible cobalt exposure in consumers from all age groups. In total, we bought 170 (48%) earrings, 117 (33.1%) hair clasps, 33 (9.3%) necklaces, 18 (5.1%) bracelets, and 16 (4.5%) finger rings (17). A total of seven items had a dark appearance.

Test strategy

The cobalt test was prepared at the laboratory at the Department of Mechanical Engineering, Technical University of Denmark, Lyngby. Individual parts of the jewellery and hair clasps that came into direct and prolonged skin contact were examined. All 354 items were initially tested for cobalt release with the cobalt spot test applied on cotton sticks at the laboratory at Gentofte Hospital. Furthermore, a subsample of 40 pieces of jewellery items was retested with the cobalt gel test at the Department of Mechanical Engineering, Technical University of Denmark. This was performed to confirm the initial results as it may be easier to visually detect cobalt release when using the cobalt gel test when compared with the cotton stick test (15). Selection of test items for the gel test was based on previous DMG test outcome (20 were DMG test positive and 20 were DMG negative) and on the outcome of the cobalt cotton stick test (4 positive reactions were reinvestigated of which 1 was also DMG positive). All samples were rinsed and washed with tap water before cobalt gel testing.

The cobalt cotton stick test

Chemicals. The cobalt cotton stick test was prepared by mixing 0.1 wt% oxalic acid, 0.02 wt% disodium-1-nitroso-2-naphthol-3,6-disulfonate (nitroso R salt), and 5.0 wt% sodium acetate in deionized water.

Equipment. The equipment used was white cotton-wool sticks. One drop of the cobalt test solution, a clear yellow solution, was added on the tip of a white cotton stick, which was then rubbed for 20–30 seconds against the test item. If the cotton

stick changed colour from yellow to red, it indicated that cobalt ions were released in a concentration that had elicited positive patch test reactions in cobalt-allergic patients (15).

The cobalt gel test

Chemical. The test was performed using cobalt cotton stick test and aqueous agar solution (Sigma-Aldrich Chemie GmbH, Steinheim, Germany). To 100 ml agar solution, 15 ml of the cobalt cotton stick test was added and stirred with low rotation speed for a few minutes to produce 115 ml of the cobalt gel ready for use. (The aqueous agar solution was prepared by placing 3 g of agar powder in a flask and adding 100 ml of demineralized water. Using a stir hot plate and agitation with a stirring magnet, the agar powder was dissolved by heating to 80°C. The stirring magnet was started on low rotation and the rotation speed was slowly increased to avoid vigorous stirring that might have produced unwanted bubbles.)

Equipment. White plastic dishes approximately 50 ml volume.

The cobalt gel test is similar to the cobalt cotton stick test except that the yellow fluid has been replaced with a yellow gel and no rubbing is involved. The samples were placed on white plastic dishes and the gel was poured to cover the samples. The gel was left for 30 min and was inspected for a colour change. A red colour indicated cobalt release, whereas no change of the yellow colour indicated absence of cobalt release. We made final interpretation of the gel test after 30 min to confirm results from the cobalt stick test (15, 16).

Scanning electron microscopy and energy-dispersive spectroscopy analysis

Microstructural characterization and chemical analysis of four cobalt-positive items with a dark metallic appearance were made using scanning electron microscope (SEM) (Model JEOL 5900; JEOL Ltd, Tokyo, Japan) in combination with energy-dispersive spectroscopy (EDS) (Oxford Link ISIS analyzer, Oxford, UK) using an INCA Energy EDS software. After initial SEM/EDS characterization of the four items, they were cut with a nipper, hot moulded in a conductive embedding material, and grinded with grit SiC paper size 320, 500, 1000, 2000, and 4000 before polishing to a mirror-like surface. The polished cross-sections were then analysed using SEM/EDS.

RESULTS

From the entire study group of jewellery and hair clasps, 1.1% (4 of 354 items) gave a positive

reaction with the cobalt spot test. As none of the hair clasps released cobalt, the proportion of positive reactions in the jewellery group was 1.7% (4 of 237 items). Cobalt releasing jewellery included three earrings and one necklace, which all had a dark coloured appearance (Fig. 1). Thus, we found cobalt release from four of seven items that had a dark appearance.

Among cobalt releasing jewellery, only the piercing post of the earring that had a decorative pendant (butterfly) also gave a positive DMG test reaction (Fig. 1). Confirmatory testing with the gel test in a subsample of 40 jewellery items showed no further positive cobalt reactions. One earring was purchased from a street vendor at the busiest pedestrian street in downtown Copenhagen (Stroget), whereas the remaining pieces were purchased from three different accessory stores within the centre of Copenhagen.

Cross-sectional SEM/EDS analyses of cobalt spot test positive jewellery showed a thin surface layer (under 1 µm thickness) that contained cobalt in all items, except for the piercing post used for the earring that had a butterfly pendant (Table 1 and Fig. 2). One example of a cross-sectional SEM/EDS analysis is shown for the butterfly pendant (Fig. 3) but not for remaining jewellery. It showed a bulk made mainly of lead. Similar results were obtained when cross-sections of remaining cobalt releasing items were analysed, although copper instead of lead was used as bulk material in these. The surface results seemed to be in accordance with the cobalt spot test results. Thus, all items contained cobalt in the surface and also released cobalt when investigated with the cobalt spot test. Of interest, we found tin–cobalt plating on the surface of all items, probably as a decorative coating to obtain a 'black pearl' colour effect (Table 1).

DISCUSSION

This study showed that only a minority of jewellery purchased in Denmark released cobalt when analysed with the cobalt screening test. The sample was initially collected for a study on nickel exposure and represents jewellery from a total of 36 stores, street vendors, and jewellery Web pages (17). We therefore think that the collection of jewellery was relatively unbiased, in the sense that the main purpose of the study was not screening for cobalt release. However, the collecting author (J.T.) was aware of the case report by Hindsén et al., which described allergic contact dermatitis following skin exposure to a brown-plated necklace (14). Jewellery with a similar dark appearance was therefore purchased when they were observed. In this study, we only detected cobalt release by the cobalt spot



Fig. 1. Three earrings and one necklace that released cobalt as assessed by the cobalt gel test. Red coloration of the yellow gel indicates cobalt release. All items had a dark metallic appearance.

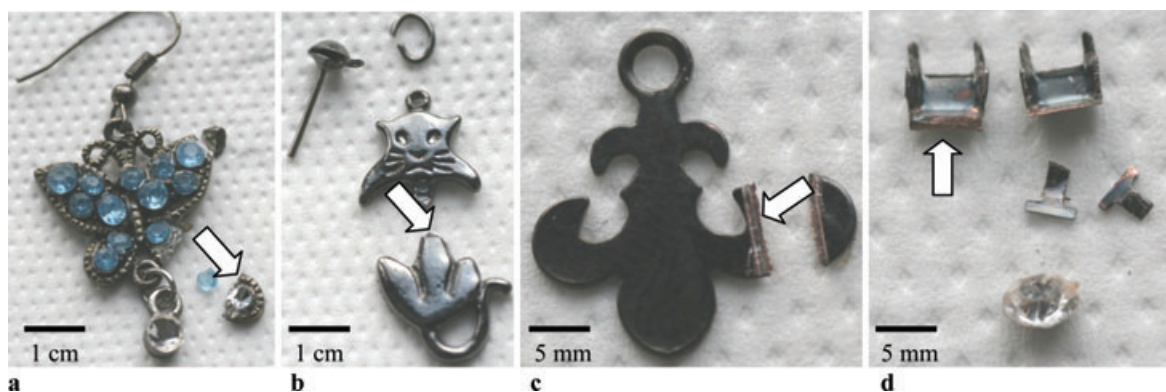


Fig. 2. Pieces of the dark appearing jewellery that gave positive cobalt spot test results were mounted in a conductive embedding material and polished to a mirror-like finish. This embedding method made it possible to investigate the cross-sections (marked with arrows) with scanning electron microscopy (SEM) and energy-dispersive spectroscopy (EDS) analysis.

test from jewellery with a dark appearance (Fig. 1). Thus, we found that four of seven dark appearing jewellery released cobalt. Of note, nickel may also be used to obtain dark coatings. Black nickel plating gives a black, matt surface with high nickel release, used for optical equipment and decoration (18). It is our impression that dark appearing jewellery, like the ones that released cobalt in this study, was in fashion 3–5 years ago in Denmark,

whereas jewellery with silvery and gold appearances are currently more in style. As fashion trends fluctuate, we cannot out rule that jewellery with a dark appearance may once again be popular, possibly resulting in increased cobalt exposure. SEM/EDS analysis showed that all four items had black cobalt–tin platings, which are often used to obtain a pearl-like effect (Fig. 3 and Table 1). The Ekem Chemical Industry Co, Ltd, Xiamen, China, produces BTC platings and recommends

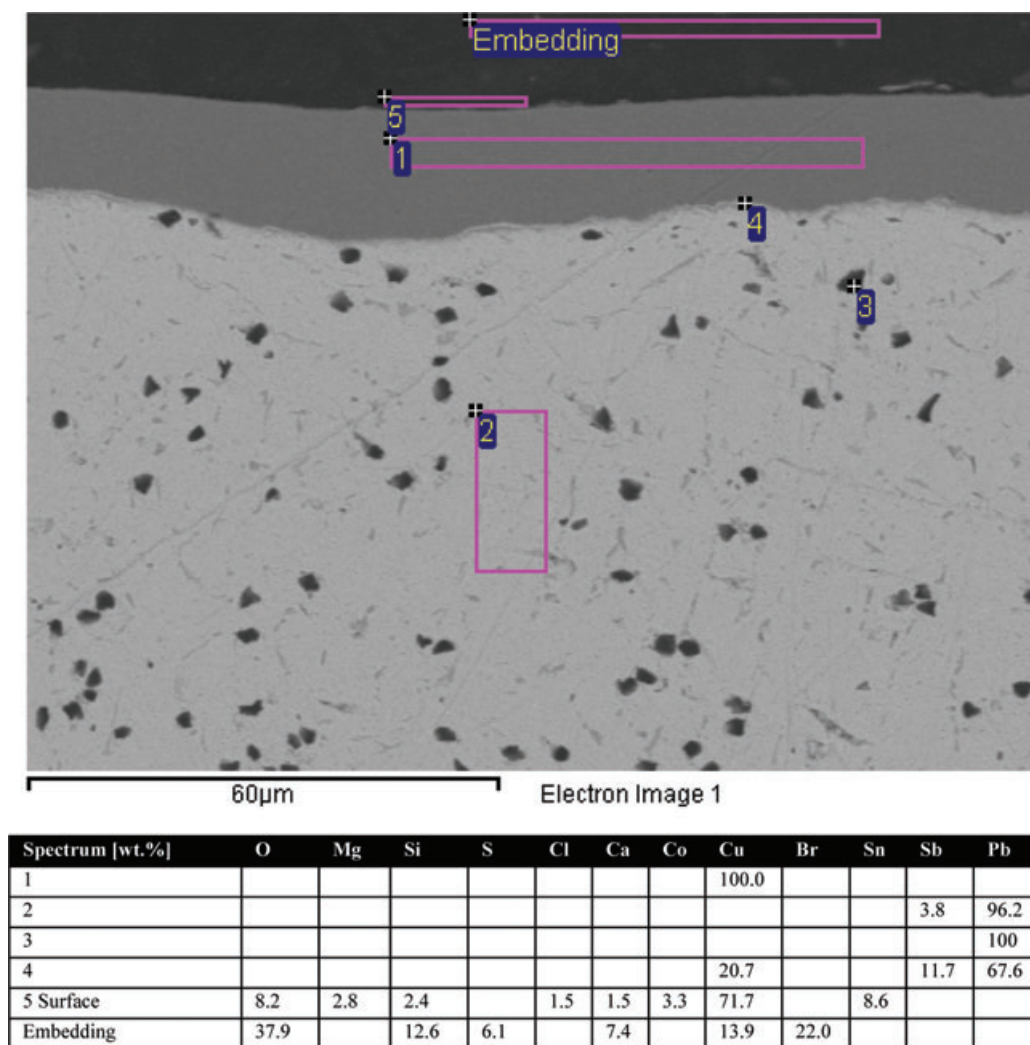


Fig. 3. Scanning electron microscopy (SEM) micrograph of the cross-section of the butterfly pendant (Fig. 2a). Energy-dispersive spectroscopy (EDS) analysis indicates that lead (spectra 2 and 3) was coated with copper (spectrum 1) and then with a thin layer (below 1 μ m) of a cobalt- and tin-containing alloy (spectrum 5).

their use in jewellery, opticals, and watches as they are nickel free and applicable for export to Europe (<http://www.ekem.com.cn/en/ArticleShow.asp?ArticleID=134>). Because we recently showed cobalt release, as assessed by the cobalt spot test, from cobalt-containing materials which elicited positive patch test reactions in cobalt-allergic patients (15, 16), we warn against the use of cobalt–tin platings in consumer products intended to be in prolonged or repeated skin contact. In 2010, J.T. and colleagues have initiated a new DMG test study in other parts of Europe to estimate nickel exposure from inexpensive jewellery. In Poland, most accessory stores and clothing stores targeting young girls had large proportions of jewellery with dark appearance (publication under preparation).

Besides black cobalt–tin platings, cobalt can also be used for other platings in jewellery. Cobalt is used in platinum cobalt plating, acid gold plating

(green to yellow), and acid gold cobalt plating (bright yellow) (Martin Baker, Allgemeine Gold und Silberscheideanstalt AG, 2010, personal communication). Thus, in a recent study, we examined release of nickel and some other metals in artificial sweat from 73 pairs of inexpensive earrings (19). We detected release of cobalt from several pairs, most with bright colour (Lundgren et al., in preparation). We therefore suggest that dermatologists and consumers should be aware of the fact that cobalt might be released from bright as well as dark jewellery and from inexpensive as well as expensive jewellery. Finally, one should be aware that we, in the confirmatory test, left the subsample of 40 items in the gel test for 30 min only. We have previously shown that it may be necessary to do repeated readings after several days to detect positive reactions (15), if the concentration of cobalt is low. In 2006, Perryman and Fowler patch tested a novel metal alloy made

Table 1. Distribution of metals after surface energy-dispersive spectroscopy analysis on items that showed cobalt release (Figs. 1 and 2)

Element spectrum (wt%)	Co	Ni	Co	Sn	Fe	Zn	O
Earring, piercing post (a)*	0	83.9	0	6.1	9.5	0	0
Earring, decorative part (a)	3.3	0	90.7	5.8	0	0	0
Neck of cat used as a decorative part (b)	3.0	0	85.8	11.2	0	0	0
Stud used for earring with a cat as decorative part (b)	6.0	0	77.2	15.8	0	0	0.8
Decorative anchor (c)	23.0	0	44.7	28.8	0	2.0	1.5
Setting for stone (d)	2.9	2.0	84.9	10.2	0	0	0

*a–d refers to the 4 jewelry items displayed in Figure 2.

of cobalt and platinum on 30 cobalt-allergic patients and found that 18% had allergic reactions (20). Taken together, the above findings underscore the possibility of a future cobalt problem.

It might seem surprising that only 1 of 78 DMG-positive items also released cobalt in the present study. Cobalt is often produced as a by-product of nickel and copper mining activities. Thus, traces of cobalt may frequently occur in nickel alloys and coatings. This is probably one explanation to concomitant cobalt and nickel allergy often observed among patients with dermatitis (7, 21) as well as among subjects from the general population (22). Data analysis of 11 516 patch-tested patients with dermatitis showed that 79% of cobalt-allergic patients also reacted to nickel and that 23% of nickel-allergic patients also reacted to cobalt (21). Concomitant cobalt and nickel allergy is probably in most cases a result of dual sensitization and not cross-reactivity (23–25). The association between jewellery exposure and cobalt allergy has been supported by studies which documented that pierced men had a higher prevalence of cobalt allergy than do non-pierced men (22, 26). However, concomitant nickel and cobalt allergy has also been observed among hard metal workers with hand eczema (27). The increasing costs of cobalt when compared with nickel may to some degree explain our study observation (i.e. cobalt release was not detected from DMG-positive items). Within 2009, the price of nickel was between 4 and 10 US\$/lb, whereas the price of cobalt was between 25 and 40 US\$/lb (<http://www.metalprices.com>). The increasing price of cobalt over the last decades has led to increased industrial incentive to separate cobalt more efficiently from nickel (Bruce McKean, Nickel Development Institute, 2003, personal communication), which may have again resulted in decreasing prevalence of concomitant cobalt and nickel allergy in the future.

Cobalt patch test data from 1985 to 2007 in Denmark showed a stable prevalence of cobalt allergy among young Danish female patients with dermatitis (7). As cobalt and nickel allergy are often concomitantly observed, one would expect the prevalence of cobalt allergy to decrease together with

the prevalence of nickel allergy following the Danish nickel regulation. The stable prevalence of cobalt allergy in the youngest age group could be explained by random variation or possibly by increased exposure to cobalt from consumer products (7). Data from two cross-sectional patch test studies performed in the general population in Denmark 16 years apart (1990 versus 2006) showed that the prevalence of cobalt allergy decreased from 0.7% to 0.1% in men and from 1.5% to 0.4% in women (28). However, because participation rates in the youngest age group were very low in the 2006 study, we have limited insight into the prevalence of cobalt allergy in the youngest age groups in Denmark.

Based on data from the current study, a recent study on metal ion release from 73 inexpensive earrings purchased in San Francisco (19), and from 2 recent epidemiological patch test studies from Denmark (7, 28), one may conclude that (i) cobalt release from inexpensive jewellery occurs; (ii) cobalt release should be suspected from inexpensive jewellery with a dark metallic appearance and also from bright jewellery; (iii) the prevalence of cobalt allergy has to some degree followed the development of nickel allergy, that is, a decrease of nickel allergy in young women and an increase of nickel allergy in middle-aged and older women, because of a cohort effect (6, 7, 29); (iv) the EU Nickel Directive has resulted in changing epidemiology of nickel allergy but has apparently not resulted in a large increase in the cobalt allergy problem, which however, might be the result if the use of cobalt in jewellery increases.

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