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### **1.1.1 Nail varnishes / colourants, preservatives, nitrosamines, formaldehyde, phenol, ethyl pyrrolidone, hydroquinones and phthalates**

Priority programme at the border SPP 2016\_6; joint campaign by the Federal Food Safety and Veterinary Office, the customs authorities, and the cantons of Aargau and Basel-Stadt (test laboratory)

Samples/sets studied: 87 Number of samples/sets objected to: 35 (42%)  
(individual samples studied) 107

Reasons for objection: *Prohibited or impermissible colourants (25), impermissible preservatives (15), prohibited solvents (3), prohibited monomer (1), technically avoidable quantities of nitrosamines (5) Undeclared preservatives (15), undeclared colourants (35), undeclared hydroquinones (3).*

#### **Initial position and objectives of study**

In recent years, nail varnishes have repeatedly been associated with high objection rates as identified by our studies of decorative children's cosmetics (2015<sup>1</sup>). In 2012, carcinogenic nitrosamines were identified in nail varnishes for the first time<sup>2</sup>. Surprisingly, we found not only N-nitrosodiethanolamine (NDELA), a nitrosamine already identified in various product categories such as decorative cosmetics for the eyes, cleansing agents for skin and hair, and both hair dyes and hair gel, but also N-nitrosodimethylamine (NDMA) and N-nitrosomorpholine (NMOR). The nitrosamine findings occurred again in 2014 and 2015. The suppliers concerned were unable to provide any explanations for these results. Based on the contents declared, we assumed the presence of nitrosamines was attributable to nitrocellulose. In collaboration with the Federal Food Safety and Veterinary Office (FSVO) and the customs authorities, a priority campaign was carried out at customs. In addition, further nail products were also collected in the cantons of Aargau and Basel-Stadt, including both products for public and professional use.

#### **Legal principles**

The requirements for cosmetic agents are stipulated in the Ordinance on Cosmetics (VKos).

<sup>1</sup> Hauri, U. Children's Cosmetics 2015; A Campaign by the Cantons of Aargau, Basel-Landschaft, Basel-Stadt (Main Laboratory), Bern, Solothurn, and Zurich and the Customs Authorities; <http://www.kantonslabor.bs.ch/dms/kantonslabor/download/berichte/berichte-2015/Kinderkosmetik-2015.pdf>  
<sup>2</sup> Hauri, U.: Children's Cosmetics 2012 / Preservatives, Colourings, Fragrances, Nitrosamines; A Joint Campaign by the Cantons of Aargau, Zurich, and Basel-Stadt (Main Laboratory), [http://www.kantonslabor.bs.ch/dms/kantonslabor/download/berichte/berichte-2012/Kinderskosmetik\\_2012.pdf](http://www.kantonslabor.bs.ch/dms/kantonslabor/download/berichte/berichte-2012/Kinderskosmetik_2012.pdf)  
Hauri, U. Children's Cosmetics 2014; A Campaign by the Cantons of Aargau, Basel-Stadt (Main Laboratory), and Zurich; <http://www.kantonslabor.bs.ch/dms/kantonslabor/download/berichte/berichte-2014/Kinderkosmetik-2014.pdf>

Parameters	Assessment
Colourants	VKos, art. 2 para. 1, appendix 2 and appendix 4
Preservatives, UV filters, substances permitted subject to conditions (hydroquinones)	VKos, art. 2 para. 2, appendix 3
Allergenic fragrances	VKos, art. 2 para. 3, appendix 3
Prohibited substances (nitrosamines)	VKos, art. 2 para. 4, appendix 4
Carcinogenic, mutagenic, and reproductively toxic substances (ethyl pyrrolidone, phenol)	VKos, art. 2 para. 5
Labelling	VKos, art. 3

### Description of samples

Most of the products were traditional nail varnishes. 29 professional nail products were also collected, including at least 19 UV-curing products. The products were collected at customs (17), as well as at importers, warehouses, toy stores, and shops in the cantons of Aargau (34) and Basel-Stadt (36).

Origin	Samples collected
USA	17
France, unknown origin	16
Switzerland	12
China	11
Germany	6
Europe	4
Luxembourg, Spain	2
Sweden	1
<b>Total</b>	<b>87</b>

Product type	Number of individual samples
Nitrocellulose-based nail varnishes containing solvents	62
Effect products for nail varnish	3
Watery nail varnishes	15
Nail products for professional use	27
<b>Total</b>	<b>107</b>

### Test procedure

Parameter group	Method
Multi-method for UV-active agents: <ul style="list-style-type: none"> <li>• Preservatives</li> <li>• UV-active fragrances</li> <li>• UV filters</li> <li>• Colourants and pigments</li> </ul>	UHPLC-DAD following extraction with 1% methanolic phosphoric acid and other solvents (UV filters; pigments)
Colourants and pigments	<ul style="list-style-type: none"> <li>• Ion-pair reversed-phase HPLC-DAD following extraction with DMF or other suitable solvents</li> <li>• LC/DAD/HRMS(/MS) as required</li> <li>• UV spectroscopy following dissolution in sulphuric acid or chloronaphthalene as required</li> <li>• LDI-TOF as required</li> </ul>
Formaldehyde	HPLC-DAD following pre-column derivatisation with 2,4-dinitrophenylhydrazine
Isothiazolinones/ polar preservatives	UHPLC-DAD following extraction with 0.1% phosphoric acid
N-nitrosamines	HPLC-HRMS(/MS) following extraction with water

Parameter group	Method
Phthalates	UHPLC-DAD following extraction with acetonitrile
Hydroquinones	HPLC-DAD following extraction with methanol

## Results and measures

The results of the joint campaign showed significant deficiencies in conformity: 35 of the 87 (42%) products collected were objected to. For around a quarter of products (22), either a ban on sales was declared or the manufacturers voluntarily took the products off the market following disclosure of our test results. The reasons for non-conformity were very diverse: besides many cases of inadequate declarations, objections were based on the presence of banned colourants and preservatives, prohibited solvents and monomers, and contaminants (nitrosamines and phenol).

### Impermissible or restricted ingredients, contaminants

#### *Impermissible colourants*

In total, seven (8%) samples, including two sets for teens, a low price set, and four products for commercial purposes, contained a multitude of colourants that are not permitted (table 1).

Table 1 – Impermissible colourants in nail varnishes

Impermissible or prohibited colourants	Number of individual samples	Number of samples/sets
C.I. 12315	5	2
C.I. 12485	2	2
C.I. 21090	1	1
C.I. 21095	2	1
C.I. 21110	3	1
C.I. 45161	4	4
C.I. 45170	2	2
C.I. 45174	4	4
C.I. 73900	1	1
C.I. 73915	1	1
<b>Total</b>	<b>25</b>	<b>7</b>

Two products from Switzerland were also among the products concerned.

The two nail varnish sets for teens are worthy of particular mention for the fact that none of the 15 nail varnishes included contained a single one of the organic pigments declared!

#### *Benzisothiazolinone*

Two nail varnish sets for teens contained the impermissible preservative benzisothiazolinone (BIT) at concentrations between 224 and 436 mg/kg. The substance was not declared, whereas the preservative phenoxyethanol – although declared – was not contained in the product. Cosmetics may only contain preservatives listed in appendix 3 of the Ordinance on Cosmetics. The European Union's SCCS (Scientific Committee on Consumer Safety) assessed the use of BIT as a preservative in cosmetic agents on 26/27 June 2012<sup>3</sup>. Given a potential for skin sensitisation comparable to that of methylisothiazolinone (MI), the fact that MI at a concentration of 0.01% in cosmetics causes contact allergy and allergic contact dermatitis, and because BIT at a concentration of 20 mg/kg in gloves has also resulted in sensitisation, the use of BIT at a concentration of 100 mg/kg was classified as unsafe. All nail varnishes in these two sets exceeded this value by a factor of two to four.

<sup>3</sup> Scientific Committee on Consumer Safety (SCCS); Opinion on Benzisothiazolinone (COLIPA n° P96), The SCCS adopted this opinion at its 15th plenary meeting of 26-27 June 2012; [https://ec.europa.eu/health/scientific\\_committees/consumer\\_safety/docs/sccs\\_o\\_099.pdf](https://ec.europa.eu/health/scientific_committees/consumer_safety/docs/sccs_o_099.pdf)

### *Hydroquinone and hydroquinone monomethyl ether (MEHQ)*

Hydroquinone and MEHQ (also known as 4-methoxyphenol or p-hydroxyanisole) are used as inhibitors to stabilise acrylate-based nail products. Acrylate monomers are polymerised in most products when exposed to UV light. Their use is only permitted in professional products and only for artificial nail systems at a concentration of up to 0.02%.

Of 27 products for professional use, 25 products contained between 0.0029 and 0.021% MEHQ and one product 0.0057% hydroquinone. With some products, however, the labelling was either inadequate or there was no guarantee that private customers would be unable to access these products (e.g. via the Internet). Although UV-curing nail varnishes applied directly to the nail do not actually constitute artificial nail varnish systems in the original sense of the term, we have tolerated the use of these substances in these relatively new products. Vendors are obliged, however, to ensure the products are not given to private individuals and are correctly declared to be 'For professional use only'. This led to a change in sales practices, particularly for Internet providers.

MEHQ was not declared in three cases, which resulted in an objection.

### *Phenol*

Nine products contained between 0.015 and 0.12% phenol. Phenol counts as a class 2 mutagen and is therefore prohibited in cosmetics. The companies concerned were asked to provide a statement. The products concerned were a traditional nail varnish with the plasticiser triphenyl phosphate, as well as eight products for professional use only, which were from two manufacturers. In one case, the manufacturer declared he had found the source of the phenol contamination and had already adapted production. In a second case, the source of the phenol remained unexplained.

### *Ethyl pyrrolidone and 1-vinyl-2-pyrrolidone*

Ethyl pyrrolidone is a polar solvent frequently used for paints and varnishes. Since being classified as a category 1B reproductive toxicant, its use in cosmetics has been prohibited. We found the substance was still in three products from the same manufacturer. Sales of the products were prohibited.

1-vinyl-2-pyrrolidone is a monomer classified as a category 2 carcinogenic substance. One product for professional use only contained between 5 and 10% of this substance according to the safety information leaflet. Sales of the product were prohibited.

### *Nitrosamines*

We first found nitrosamines in nail varnishes for children in 2012<sup>4</sup>. Recent years have confirmed this was no exception. Although the providers of these products were unable to give any explanations, we assumed nitrocellulose to be responsible for the findings given the contents declared. Based on the generally lower quality of cosmetics for children (used as toys), we also assumed the problem relates to cheap products from beyond Europe. This is why the findings of the present campaign came as a surprise: of 104 nail products studied, 61 products contained nitrosamines in quantities in excess of 20 µg/kg. These kinds of quantities in cosmetics are usually deemed to be technically avoidable (table 2).

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<sup>4</sup> Hauri, U.: Children's Cosmetics 2012 / Preservatives, Colourings, Fragrances, Nitrosamines; A Joint Campaign by the Cantons of Aargau, Zurich, and Basel-Stadt (Main Laboratory), [http://www.kantonslabor.bs.ch/dms/kantonslabor/download/berichte/berichte-2012/Kinderskosmetik\\_2012.pdf](http://www.kantonslabor.bs.ch/dms/kantonslabor/download/berichte/berichte-2012/Kinderskosmetik_2012.pdf)

Table 2 – Nitrosamines in nail varnishes

	NDELA	NDMA	NMOR	NDEA	Total
Number of samples > LOD (5-10 µg/kg)	49	51	48	18	62
Number of samples > 20 µg/kg	33	46	36	8	61
Number of samples > 50 µg/kg	17	29	7	8	50
<b>Highest value (µg/kg)</b>	<b>6010</b>	<b>497</b>	<b>255</b>	<b>266</b>	<b>6507</b>
Lowest value (µg/kg)	7	10	8	8	20
Median (µg/kg)	34	59	30	19	124
<b>90th percentile (µg/kg)</b>	<b>168</b>	<b>269</b>	<b>59</b>	<b>262</b>	<b>466</b>

A close look at the results confirmed the assumption that nitrocellulose was the reason for finding nitrosamines: of the 63 samples declared to contain nitrocellulose, only two products contained no nitrosamines. There was one product containing nitrosamines where the contents were not declared. In this case, however, the presence of nitrocellulose can be inferred from the remaining ingredients identified after analysis (table 3).

Table 3 – Nitrosamine findings broken down by product type

Contents as declared	Samples as per decl.	Nitrosamine-containing samples	Nitrosamine-free samples
Traditional nitrocellulose-based nail varnishes	63	61 97%	2 3%
Products where something was not declared	2	1 50%	1 50%
Water-based nail varnishes	15	0 0%	15 100%
Professional nail products without nitrocellulose (non traditional nail varnishes)	24	0 0%	24 100%
<b>Total</b>	<b>104</b>	<b>62 60%</b>	<b>42 40%</b>

Of 58 'traditional' nail varnishes collected (one set = one sample), 55 products (95%) were solvent-containing nitrocellulose-based products according to what was declared. Only two sets for young people contained water-based nail varnishes, and we assume nitrocellulose was also an ingredient of one product as mentioned above. None of the professional nail products based on acrylate monomers were contaminated with nitrosamines.

According to the Ordinance on Cosmetics, cosmetics may contain traces of prohibited substances if these are not added deliberately, present no danger to health, and cannot technically be avoided. Toxicology estimates from the Federal Food Safety and Veterinary Office (FSVO) and various manufacturers revealed the contents identified present a 'tolerable health risk'. Currently, nitrocellulose is a main ingredient of all commercially available nail varnishes, and no brand on the market is free of nitrosamines. This could imply that traces of nitrosamines are currently unavoidable. Data does, however, reveal significant differences between individual products, which suggest it may be possible to reduce these undesirable substances. Content levels in excess of 466 µg/kg of nitrosamines in total were classified as technically avoidable as the content levels for 90% of the products studied were found to be below this figure. Sales of these products were prohibited.

Given that NDELA, NDMA, NDEA, and NMOR are genotoxic carcinogens, exposure to these substances should be kept to a minimum, as there are many other sources of nitrosamines, such as food, tobacco, rubber items, and other cosmetics. The manufacturers were therefore instructed to ascertain the cause and take measures to minimise nitrosamines levels in future. As a result of our objections in recent years and discoveries of NDELA by German supervisory authorities in late 2015, some manufacturers have recently become aware of the nitrosamine issue, at least in relation to the substance NDELA. The clarification work and measures undertaken have already resulted in a reduction, but have so far failed to resolve the problem completely. Besides the choice and combination of raw materials, storage also seems to play an important role. This is also known to be the case for nitrosamines in mascara or skin cleansing products. Consequently, we have pointed out that the practically unlimited shelf life which manufacturers state for these products – a statement based only on microbiological studies – needs to be reconsidered.

### *Phthalates*

Plasticisers are added to nail varnishes to stop these products becoming brittle too quickly. Dibutyl phthalate was often used for this purpose in the past, before being prohibited since dibutyl phthalate was classified as a reproductive toxicant. While European products have long been free of phthalates, products containing DBP keep turning up from beyond Europe. This campaign, however, failed to identify any phthalate content level > 100 mg/kg.

### *Formaldehyde*

These days, nail hardeners may still contain up to 5% formaldehyde. It is also used as a starting material for the plasticiser known as tosylamide/formaldehyde resin. Apart from the usual traces of formaldehyde, no conspicuous concentrations were identified.

### Inadequate declaration

#### *Colourants, preservatives, and hydroquinones*

It is important for those with allergies that contents are correctly declared. Erroneous declarations also point to deficiencies in production and/or quality assurance for the products concerned and indicate the presence of these agents was not taken into account in the safety assessment.

The extremely high rate of incorrectly declared colourants is worthy of remark, with a total of 35 undeclared colourants being identified in 26 individual samples. This suggests that quality assurance in this respect is nonexistent. Products with inadequate declarations were objected to and corrections demanded.

	Preservatives	Failure to declare Colourants	Hydroquinones
Number of products collected	2 (2.4%)*	13 (15%)*	3 (11%)**
Samples studied	5 (14%)**	26 (24%)**	

\* Based on samples collected

\*\* Based on samples actually studied

\*\*\* Based on commercial products

### *Tosylamide*

We found tosylamide (p-toluenesulphonamide) in four nail varnishes at conspicuous levels of 0.25 to 0.46%. Since this substance was not declared, we asked the companies concerned for an explanation. According to these statements, the tosylamide/formaldehyde resins used contained 10% of this starting material. Since those with allergies – due to the polymer being declared – have already been made aware of the possible presence of p-toluenesulphonamide and the substance is only present as contamination, we refrained from taking further measures in. One manufacturer informed us that they will nonetheless make an effort to look into purer raw materials.

### *Other declaration deficiencies*

- Various products for professional use made inadequate declarations. For example, contents such as 'phosphine oxide' or 'alpha-hydroxy ketone' were listed without stating the chemical substances actually contained.
- Some products for professional use didn't mention the reason for this restriction.

## Conclusions

- Nitrosamines in nail varnishes are not only a problem of low price products. This is the most important finding of this campaign. With the exception of two water-based nail varnish sets for teens, all nail varnish samples contained the film former nitrocellulose. Although efforts to clarify how nitrosamines form in nail varnishes are still ongoing, it may be assumed that the use of nitrocellulose is the main reason for the presence of nitrosamines in these products.
- The onus is on industry to find solutions for avoiding nitrosamines in nail varnishes or reducing their contents to an acceptable level. The quantities identified however do not represent an unacceptable health risk. Since the four substances identified are genotoxic and given that there are other sources of nitrosamines besides cosmetics (e.g. food, tobacco and rubber products), the quantities of these substances need to be reduced to a technically unavoidable level.
- Apart from the issue of nitrosamines, nail products for professional use are of much poorer conformity than traditional nail varnishes. Particularly striking are the use of impermissible colourants and preservatives and a poor declaration of ingredients.
- The high objection rate calls for further market controls.