

Survey of Chemical Substances in Consumer Products

Survey no. 25, 2003

Survey of the content of certain chemical substances in mattress pads

Carried out by:

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Preface

This survey of the content of total tin and total antimony as well as triclosan and permethrin in mattress pads has been carried out for the Danish Environmental Protection Agency based on the tender dated April 2001 from the Danish Environmental Protection Agency.

Summary and conclusions

The analysed mattress pads have been chosen and bought in consultation with the Danish Environmental Protection Agency on the basis of an investigation of the supply at the Danish market. Eight mattress pads and two top mattresses with foam filling have been bought. All the mattresses have been tested for content of total tin and total antimony while five of the mattresses have also been tested for content of triclosan and permethrin.

We could not establish the presence of triclosan or permethrin in the tested goods. For two out of five samples, it was not possible to carry out the tests as they were impeded by an interference, which gave a large background in the chromatography.

Dealers of washable mattress pads and mattresses with detachable, washable covers often use the concepts “antibacterial” and “allergy-friendly”. Only one mattress pad is marketed as having real antibacterial properties. This mattress is declared in co-operation with The Asthma and Allergy Association.

The content of total tin is in some samples considerable. The top mattresses contain definitely more tin than the mattress pads. The content has been measured to 120 mg/m^2 ($110 \text{ }\mu\text{g/g}$) and 180 mg/m^2 ($120 \text{ }\mu\text{g/g}$) respectively in the top mattresses, while the highest measured content of tin in the mattress pads is 12 mg/m^2 ($9.7 \text{ }\mu\text{g/g}$). However, there are large deviations in the results measured at the mattress pads – for one sample $> 100\%$ whereas the deviations of the measurements from the top mattresses are low. This indicates that the tin compounds do not come from the production of the fibre materials in the mattress pads but is applied after the production. Tin compounds is used as catalyst in the production of polyurethane polymers /5/. This might explain the high but equally distributed content of tin compounds in the top mattresses.

The measured content of total antimony is $9 - 150 \text{ mg/m}^2$ ($15 - 150 \text{ }\mu\text{g/g}$) with a deviation of up to 13%. Antimony is used in connection with the production of polyester, wherefore it can be expected that antimony may be found in products containing polyester.

In addition, the test shows that:

- The most used types of fibres are cotton and polyester as well as blends of cotton/polyester.
- Jysk A/S (Danish bedding firm) has the largest and most varying selection of mattress pads in Denmark.
- Based on the present investigation, it is not possible to state the exact quantities of mattress pads sold in Denmark.

Sammenfatning og konklusioner

De analyserede rulle madrasser er udvalgt og indkøbt i samråd med Miljøstyrelsen på baggrund af en undersøgelse af udbuddet på det danske marked. Der er indkøbt 8 rulle madrasser samt 2 topmadrasser med en kerne af skummateriale. Alle er analyseret for indhold af total tin og total antimon, mens 5 af de indkøbte varer ligeledes er analyseret for indhold af triclosan og permethrin.

Der er ikke påvist indhold af triclosan eller permethrin i de analyserede varer. For 2 ud af 5 prøver var det dog ikke muligt at gennemføre analyserne, idet disse blev besværliggjort af en interferens, som gav en stor baggrund ved kromatograferingen.

Begreberne ”antibakteriel” og ”allergivenlig” anvendes ofte af forhandlerne i forbindelse med vaskbare rulle madrasser og madrasser med aftageligt, vaskbart betræk. Kun én rulle madras markedsføres som havende egentlige antibakterielle egenskaber. Denne er deklareret i samarbejde med Astma- og Allergiforbundet.

Indholdet af total tin er i visse prøver betydeligt. Topmadrasserne indeholder klart mere tin end rulle madrasserne. Der er målt indhold på hhv. 120 mg/m^2 ($110 \text{ } \mu\text{g/g}$) og 180 mg/m^2 ($120 \text{ } \mu\text{g/g}$) i topmadrasserne, mens det højeste målte indhold af tin i rulle madrasser er 12 mg/m^2 . Der er dog stor spredning på resultaterne målt på rulle madrasserne - i et enkelt tilfælde er spredningen således $> 100\%$, hvorimod spredningen på målingerne foretaget på topmadrasserne er lav. Dette indikerer, at tinforbindelserne ikke stammer fra produktionen af de i rulle madrasserne indgåede fibermaterialer, men må være påført efter at disse er fremstillet. Tinforbindelser anvendes også som katalysatorer i forbindelse med produktion af polyuretan skum /5/. Dette kan forklare det høje, men jævnt fordelte indhold af tinforbindelser i topmadrasserne.

Det målte indhold af total antimon er på $9 - 150 \text{ mg/m}^2$ ($15 - 150 \text{ } \mu\text{g/g}$) med en spredning på op til 13%. Antimon anvendes i forbindelse med fremstillingen af polyester, hvorfor det må forventes at forekomme i produkter indeholdende polyester.

Der ud over viser undersøgelsen, at

- de hyppigst anvendte fibertyper er bomuld og polyester samt blandinger heraf.
- Jysk Sengetøjslager (nu Jysk A/S) har langt det største og mest varierede udbud af rulle madrasser i Danmark.
- det er ikke muligt på baggrund af nærværende undersøgelse at angive mængderne af rulle madrasser solgt i Danmark.

1 Background

There is a steadily growing interest in the chemistry, which we are exposed to, in our everyday life. According to various surveys, substances with unwanted effect have been found in common products available on the market. Therefore, the Danish Environmental Protection Agency wants to contribute greatly to a survey of chemical substances in consumer products – including survey of the below mentioned chemical substances in mattress pads.

1.1 ANTIMONY

On the list of dangerous substances, 10 different antimony compounds are classified. Of this "S,S',S',S'-teraphenylthiobis-(4,1-phenylene)-disulfoniumhexafluoro-antimonate(1), diphenyl-(4-phenylthiophenyl)-sulfoniumhexafluoro-antimonate(2) are compounds of 1 and 2 (CAS No. 159120-95-3)" classified with R43 (may cause allergy at skin contact).

Antimony trioxide (CAS No. 1309-64-4) is classified as carcinogenic in group 3 with R40 (limited evidence of a carcinogenic effect). This substance is also on the "List of unwanted substances".

In Ökotest it has been referred several times that antimony is used in connection with the manufacturing of polyester, and as, among other things, 2.5 mg has been found in a bra with an air pump inserted, it will be interesting to make a survey of the content of antimony in textiles. /1/.

1.2 TIN

Ökotest has found residues of organic tin compounds in mattress pads. It is the opinion that it is used in connection with the production of polyester. /1/

According to experiments with animals, organic tin compounds are suspected of giving long-term effect to the immune response. Tributyltin compounds have shown effects to the capacity for reproduction for organisms in the maritime environment. /1/.

1.3 TRICLOSAN

Ökotest refers to investigations where triclosan has been found in sheets, which are promoted as being antibacterial. Triclosan is added to products in order to be bactericidal. /1/.

1.4 PERMETHRIN

Permethrin has been approved as a pesticide. Greenpeace has found permethrin in some carpets, where it is used against dust mites. It is therefore also possible that permethrin may be used in mattress pads against dust mites. /1/.

2 Purpose

The purpose of the project is to achieve more knowledge about the content of total tin and antimony compounds as well as triclosan and permethrin in mattress pads. This knowledge should subsequently make the basis of further surveys of specific tin and antimony compounds. Furthermore, this knowledge may form the basis for information to consumers and manufacturers and for a possible regulation within this field.

3 Mattress pads in the Danish market

3.1 THE DEFINITION OF MATTRESS PADS

Before the implementation of the survey, a definition of mattress pads was laid down in co-operation with the Danish Environmental Protection Agency:

A mattress pad is in this context a relatively thin and washable type of mattress, which only is to be used on top of another mattress and not as a mattress alone. The purpose is partly to be a washable protective layer to the underlying mattress and partly to give additional comfort.

The mattress pads can be covered with cotton, man-made fibres or blends. As filling cotton wool made of synthetic fibres (often polyester), cotton or wool are used.

Under this definition, we also find the so-called electric blanket made of 100% wool.

Based on the survey of products in the Danish market, we noticed, however, that the designation "top mattress" in some cases has been used for products, which fall within the above mentioned definition. Except from this, there is in general a differentiation between mattress pads and top mattresses as the last mentioned often has a core of latex, PU-foam or similar.

3.2 SURVEY OF THE SUPPLY OF MATTRESS PADS IN THE DANISH MARKET

In connection with purchase of test material, we have carried out a survey of the product supply in the Danish market. We have visited seven distributors (mainly national distribution chains), contacted three other distributors by telephone and two Danish manufacturers.

The survey shows that the individual distributors normally have 1- 2 different mattress pads in all sizes for normal beds. The only exception is Jysk Sengetøjslager (now Jysk A/S), which has a much larger and more varying quantity.

Cotton, polyester and their blends are the most used types of fibres.

All mattress pads of cotton/polyester are washable. Some of them carry a label stating that they withstand washing at the boil.

The general impression is that the distributors often compare the terms "anti-bacterial" and "allergy-friendly" with the fact that the products are washable or have detachable, washable covers.

In connection with a specific inquiry for antibacterial-treated mattress pads from two individual distributors, both sales assistants referred to top mattresses, which were designated as being "antibacterial-treated". At an additional inquiry to the manufacturers, it appeared to be a misunderstanding. The cover is detachable and washable and produced in a special tight construction. According to the information given, no antibacterial agents have been used in connection with the production.

The survey showed that only one mattress pad, which according to the marketing specifically states that this mattress pad consists of "...special treated textile with fantastic antibacterial properties ... stuffed with 1000 g of the finest and soft antibacterial polyester down per m²". /3/. This mattress pad is declared in co-operation with The Asthma and Allergy Association, which means that The Asthma and Allergy Association has assessed the substances of the product with regard to allergy characteristics. Furthermore, it is a requirement that the product is washable at 60°C at least. /4/.

The manufacturer informs that triclosan or permethrin have not been used in this product, which also has been confirmed from the analysis results (sample No. 8).

3.3 NUMBERS OF MATTRESS PADS SOLD

It is difficult to find the exact quantity of used/sold mattress pads in Denmark, as mattress pads do not have their own KN-product number (customs tariff) but are included in the group: 9404,90,90, Bedding and similar, except with a filling of feather and downs. This category includes therefore i.a. mattresses, down quilts, quilts, quilted bedcovers, eiderdowns, mattress covers (thin mattresses as pads to the real mattresses), wedge-shaped bolster, pillows, decorative pillows etc. /2/.

The import for the whole group made a total in 2000 of 4762.3 tons with Germany (1633.2 t), Poland (517.7 t), Norway (511.7 t), Lithuania (490.9 t) and China (434.7 t) as the most important. The export made a total of 10439.4 tons with Sweden (2030 t), Norway (1658.1 t), Germany (1587 t), USA (721 t) and The Netherlands (665.5 t) as the most important export countries. /2/.

Based on the present survey, it is not possible to estimate the total number of mattress pads included in this product group. In order to get an overview of the exact quantities, it is necessary to carry out a much more thorough analysis of the market situation than it has been possible in this survey.

The following might give a rough estimate:

If each Dane on average buys a mattress pad of 400 g/m² in a size of 90×200 cm every 15 years, this will give a turnover of approx. 240 tons/year.

4 Test method

The following is a description of the test methods used:

4.1 TRICLOSAN AND PERMETHRIN

In the preliminary experiments, extractions were carried out with four different solvents in order to find the most suitable solvent. The sample (consisting of mattress 6) was diluted with solvent and was left at room temperature for more than 24 hours, whereupon the solvent was separated and evaporated. During the evaporation material precipitated for all solvents. Pentane was the solvent, which resulted in the minimum precipitated material and therefore pentane was selected as extraction solvent. It was established with "Thin Layer Chromatography" that there was only one large interference.

The analyses were carried out by means of GC-MS. By using "On Column", OC, injection, the detection limit was improved by approx. 25 times (permethrin from 25 to 1 µg/ml and triclosan from 250 to 10 µg/ml). It was found that the reaction for triclosan was almost doubled by derivatization with diazomethane (triclosan from 10 to 5 µg/ml). These figures have been found by injection of pure standards. The detection limits for the stated results are only for standard solutions, which have undergone the sample preparation. The detection limits are stated in the tables in Chapter 5.

The pure analytes were used as standard solutions and the concentration was determined in relation to the internal standards. Fenpropathrin, α -cypermethrin and esfenvalerate were used as internal standards, as all three substances have a structure, which can be compared to the analytes. Three internal standards were used to measure the concentration of the analytes in order to reduce the possibility of interference for all the internal standards.

In order to extract larger material quantities, each mattress part was separated into three fractions according to the most natural stratification. Please see the results in Chapter 5.

The analyses of permethrin and triclosan were impeded by an interference, which gave a large background in the chromatography. The extracts were saturated with this interference after having been concentrated.

4.2 TOTAL TIN (SN) AND TOTAL ANTIMONY (SB)

4.2.1 Test preparation

Three representative samples were cut out from each mattress in the entire thickness. These three samples are designated A, B and C in table 5.2 and 5.3. The size of the samples was chosen to a weight of approx. 2 g. After this, each sample was separated into small pieces with a diameter of approx. 1-2 mm. The entire separated sample was weighed and transferred to a crucible. The material in the crucibles was moistened with 7 M HNO₃ (sub boiling quality)

and was left for two hours to fix the organic tin compounds. Then the crucibles were heated slowly in a muffle furnace to 500°C. After cooling, the incineration residue was collected in 20 ml 4 M HCl.

The blank specimens were prepared similarly.

4.2.2 Analyses

All the samples were subsequently analysed for Sn with FI-ICP-MS (Flow Injection Inductively Coupled Plasma Mass Spectrometry) on an Elan 5000, in that internal standards were added to the samples and standards on-line via Flow Injection. The detection limit was 0.01 µg/g.

The content of Sb was analysed by ICP-AES (Inductively Coupled Plasma Atomic Emission Spectrometry). The detection limit was 1 µg/g.

4.2.3 Degree of accuracy

The uncertainty for this type of analysis is empirically estimated to be below 10% for concentrations higher than 5 times DL. %RSD for Sb determinations for the mattress material is approx. 10%, while the variation for Sn is considerably higher – this variation must primarily be ascribed to an uneven distribution in the mattresses, as Sn determinations typically have a %RSD below 10% with this method for homogeneous materials.

%RSD is the standard deviation of the three determinations in percent of the average value.

5 Results

5.1 TRICLOSAN AND PERMETHRIN

Test results for samples stated in mg/kg

| Client No. | 6 | 6 | 6 | Recommended detection limit |
|-------------------------|-----------------------|-----|-----|-----------------------------|
| Chemical Technology No. | 6-1 | 6-2 | 6-3 | |
| Triclosan | Could not be analysed | | | 5 |
| Permethrin | | | | 1 |
| | | | | |

”-” means below the detection limit

6-1: Fabric from the surface, outer insulation, and separation layer (from one side of the mattress)

6-2: Foam rubber

6-3: Fabric from the surface, outer insulation, and separation layer (from the other side of the mattress)

Test results for samples stated in mg/kg

| Client No. | 7 | 7 | 7 | Recommended detection limit |
|-------------------------|-----------------------|-----|-----|-----------------------------|
| Chemical Technology No. | 7-1 | 7-2 | 7-3 | |
| Triclosan | Could not be analysed | - | - | 5 |
| Permethrin | | - | - | 1 |
| | | | | |

”-” means below the detection limit

7-1: Fabric from the surface (from both sides of the mattress)

7-2: Insulating material from the middle

7-3: Insulating material from the middle

Test results for samples stated in mg/kg

| Client No. | 8 | 8 | 8 | Recommended detection limit |
|-------------------------|-----|-----|-----|-----------------------------|
| Chemical Technology No. | 8-1 | 8-2 | 8-3 | |
| | | | | |
| Triclosan | - | - | - | 5 |
| Permethrin | - | - | - | 1 |
| | | | | |

”-” means below the detection limit

8-1: Fabric from the surface, outer insulation, separation layer (one side of the mattress)

8-2: Fabric from the surface, outer insulation, separation layers (the other side of the mattress)

8-3: Insulating material from the middle

Test results for samples stated in mg/kg

| Client No. | 9 | 9 | 9 | Recommended detection limit |
|-------------------------|-----|-----|-----|-----------------------------|
| Chemical Technology No. | 9-1 | 9-2 | 9-3 | |
| | | | | |
| Triclosan | - | - | - | 5 |
| Permethrin | - | - | - | 1 |
| | | | | |

”-” means below the detection limit

9-1: Insulating material from the middle

9-2: Fabric, outer insulation, and separation layer (from one side of the mattress)

9-3: Fabric, outer insulation, and separation layer (from the other side of the mattress)

Test results for samples stated in mg/kg

| Client No. | 10 | 10 | 10 | Recommended detection limit |
|-------------------------|-----------------------|------|------|-----------------------------|
| Chemical Technology No. | 10-1 | 10-2 | 10-3 | |
| | | | | |
| Triclosan | Could not be analysed | | | 5 |
| Permethrin | | | | 1 |
| | | | | |

”-” means below the detection limit

10-1: Insulating material from the middle

10-2: Fabric from the surface, outer insulation and separation layer (from one side of the mattress)

10-3: Fabric from the surface, outer insulation and separation layer (from the other side of the mattress).

5.2 TOTAL TIN (SN)

The test results for content of total tin. A, B and C designate 3 samples from the same product (see 4.2.1)

| Sample | Sn $\mu\text{g/g}$ | Average $\mu\text{g/g}$ | Sn mg/m^2 | Average mg/m^2 | %RSD |
|--------|--------------------|-------------------------|--------------------|-------------------------|------|
| 1A | 4.3 | 2.5 | 2.6 | 1.5 | 108 |
| 1B | 0.58 | | 0.34 | | |
| 1C | - | | - | | |
| 2A | 0.28 | 0.28 | 0.23 | 0.23 | 1 |
| 2B | 0.28 | | 0.23 | | |
| 2C | 0.28 | | 0.23 | | |
| 3A | 5.4 | 6.0 | 3.7 | 4.1 | 90 |
| 3B | 12 | | 7.9 | | |
| 3C | 0.91 | | 0.61 | | |
| 4A | 8.7 | 5.8 | 3.7 | 2.5 | 78 |
| 4B | 8.0 | | 3.4 | | |
| 4C | 0.59 | | 0.25 | | |
| 5A | 0.21 | 0.29 | 0.14 | 0.19 | 57 |
| 5B | 0.19 | | 0.12 | | |
| 5C | 0.49 | | 0.32 | | |
| 6A | 120 | 120 | 170 | 180 | 6 |
| 6B | 120 | | 170 | | |
| 6C | 130 | | 190 | | |
| 7A | - | 4.3 | - | 1.7 | 32 |
| 7B | 4.6 | | 1.8 | | |
| 7C | 3.9 | | 1.5 | | |
| 8A | - | 9.7 | - | 12 | 79 |
| 8B | 16 | | 20 | | |
| 8C | 3.1 | | 3.8 | | |
| 9A | 3.9 | 3.6 | 2.9 | 2.7 | 84 |
| 9B | 0.45 | | 0.34 | | |
| 9C | 6.5 | | 4.8 | | |

| | | | | | |
|-----|-----|-----|-----|-----|---|
| 10A | 110 | 110 | 120 | 120 | 4 |
| 10B | 110 | | 120 | | |
| 10C | 100 | | 110 | | |

5.3 TOTAL ANTIMONY (Sb)

The test results for content of total antimony. A, B and C designate 3 samples from the same product (see 4.2.1).

| Sample | Sb $\mu\text{g/g}$ | Average $\mu\text{g/g}$ | Sb mg/m^2 | Average mg/m^2 | %RSD |
|--------|--------------------|-------------------------|--------------------|-------------------------|------|
| 1A | 15 | 15 | 9.1 | 8.6 | 8 |
| 1B | 14 | | 8.1 | | |
| 1C | - | | - | | |
| 2A | 110 | 120 | 89 | 97 | 13 |
| 2B | 110 | | 90 | | |
| 2C | 130 | | 110 | | |
| 3A | 130 | 120 | 88 | 81 | 9 |
| 3B | 120 | | 80 | | |
| 3C | 110 | | 74 | | |
| 4A | 120 | 110 | 50 | 47 | 11 |
| 4B | 98 | | 41 | | |
| 4C | 120 | | 51 | | |
| 5A | 57 | 59 | 38 | 39 | 3 |
| 5B | 61 | | 40 | | |
| 5C | 60 | | 40 | | |
| 6A | 27 | 32 | 39 | 45 | 13 |
| 6B | 33 | | 47 | | |
| 6C | 35 | | 50 | | |
| 7A | 140 | 150 | 54 | 59 | 8 |
| 7B | 150 | | 60 | | |
| 7C | 160 | | 63 | | |
| 8A | 120 | 130 | 140 | 150 | 9 |
| 8B | 140 | | 170 | | |
| 8C | 120 | | 150 | | |
| 9A | 14 | 16 | 10 | 12 | 13 |
| 9B | 18 | | 13 | | |

| | | | | | |
|-----|----|----|-----|----|---|
| 9C | 15 | | 11 | | |
| 10A | 85 | 86 | 97 | 98 | 2 |
| 10B | 88 | | 100 | | |
| 10C | 85 | | 97 | | |

6 Discussion

6.1 PURCHASE OF TEST MATERIAL

Through many years of work with textiles and clothing at the Danish Technological Institute, we have experienced that permethrin is often used for mothproofing of wool. It would therefore be relevant if the survey also included different wool products.

In connection with the survey, it became obvious that a very great part of the marketed mattress pads consist of a combination of cotton/polyester, we decided to include a top mattress made of wool in the survey.

Equally, another top mattress was included in the survey as the dealer presented the product as being anti-bacterial treated. However, it became apparent that it was a misunderstanding, but this circumstance was not clarified until after we bought the test material.

6.2 PERMETHRIN AND TRICLOSAN

The analyses of permethrin and triclosan were impeded by an interference, which gave a large background in the chromatography. The extracts were saturated with this interference after having been concentrated. The results from the analysis of tin showed a high quantity of tin in the mattresses, and we presume that this compound made it impossible to carry out the permethrin and triclosan analyses.

Nothing in the survey indicates that triclosan and permethrin are being used as antibacterial substances in the mattress pads. Two major Danish manufacturers of mattress pads and top mattresses, respectively, inform that these substances are not being used, which also appears from the test results.

6.3 TOTAL TIN

Basically, it is not likely that organotin compounds have been used in textiles, but the survey shows clearly that there is a considerable content of tin compounds in the mattress pads.

The large deviation in the results of the individual mattress pads indicates that the tin compounds do not come from the production of the fibre materials in the mattress pads, but must have been applied after the production. For sample No. 1 the deviation is > 100%.

These types of chemicals, which are used as anti-fouling substances for ship's bottoms, may be added to products, that either are not being washed at all or not very frequently, and that at the same time are exposed to perspiration or other humidity exposure in use. These types of chemicals may be used on such products in order to prevent or impede the growth of micro-organisms.

It is, however, also a question whether the chemicals can be used as transport preservation of different textiles or as preservation in different auxiliary chemicals for textiles as well.

The content of tin is 10 to 15 times higher in the top mattresses with a foam/latex filling than in the mattress pads of cotton and polyester. As the deviation of the content measured in the top mattresses is low compared to the deviation measured on the individual mattress pads, this indicates that the tin compounds might have been applied during the production of one or several of the included materials. It could be the foam filling which is the most essential difference between the two types of products, and as known, tin catalysts are used in the production of PU-foam in order to increase the urethane formation.

6.4 ANTIMONY

Antimony is used in connection with the production of polyester, wherefore it can be expected that antimony may be found in products containing polyester. It has also been seen that the content is equally distributed in the products.

Furthermore, it is known that antimony oxides are used as fire-retardant. This might possibly explain the fact that the product, consisting of 100% wool, contains just as much antimony as the other products.

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