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DECONTAMINATION OF NBC-PROTECTIVE GARMENTS - Part 5: Thermal decontamination of the protective suit NM143

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8) ABSTRACT Thermal decontamination of the NBC-protective suit (NM143) has been studied. The NM143 overgarment consists of a liquid repellent outer layer and an inner layer of activated charcoal fixed on polyurethane foam. Small pieces of fabric were exposed to 10 g/m ² sulphur mustard (HD) and decontaminated in a tube oven at temperatures from 80 to 150 °C for 1,4 and 24 hours, respectively. The airflow was kept constant at 2 l/min in all experiments. These small-scale laboratory experiments show that decontamination with respect to HD can be obtained using temperatures from 80 to 150 °C and heating for 1 hour. Heat-treatment for 4 and 24 hours also gave satisfactory results at the lower temperatures 80 and 100 °C. Introducing humidity in the airflow at 100 °C gave significantly better protection against HD after decontamination than for decontamination in dry air. This effect was not observed at 130 °C. Decontamination by heat-treatment was compared with the recommended laundrying procedure from previous studies. This was heat-treatment for 4 hours at 100 °C in humid air (airflow 2 l/min) and laundrying at 60 °C for 45 min, and subsequent rinsing three times in cold water. Experiments performed the same day on the samples taken from the same NM143 suit showed that both procedures gave satisfactory decontamination.		
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CONTENTS

	Page	
1	INTRODUCTION	7
2	BACKGROUND	7
3	EXPERIMENTAL SECTION	8
3.1	Material	8
3.2	Thermal decontamination procedure	8
3.3	Laundering	10
3.4	Penetration measurements	10
3.5	Thermogravimetric analysis	10
4	RESULTS AND DISCUSSION	11
4.1	Protective properties of NM143 fabric compared to previous studies	11
4.2	Parameters in the penetration measurement procedure	12
4.3	The thermal stability of NM 143	14
4.3.1	Thermogravimetric analysis of NM143 inner layer	14
4.3.2	Isothermal heat-treatment of NM143 inner layer	15
4.4	TGA experiments on contaminated cloth	17
4.5	Thermal decontamination of NM143	18
4.5.1	Decontamination with dry air	19
4.5.2	Decontamination with humid air	20
4.6	Comparison between decontamination by hot air and laundering	23
5	CONCLUSIONS	25
APPENDIX		
A	EFFECT OF DROP SIZE AND POSITION ON PENETRATION DATA	26
B	DECONTAMINATION DATA FOR DRY AIR THERMAL TREATMENT	27
C	DECONTAMINATION DATA FOR THERMAL TREATMENT IN HUMID AIR	29
	References	30
	Distribution list	31

DECONTAMINATION OF NBC-PROTECTIVE GARMENTS - Part 5: Thermal decontamination of the protective suit NM143

1 INTRODUCTION

Protective suits for protection against chemical warfare (CW) agents incorporate a highly adsorbing layer containing activated carbon. The main advantage of using activated carbon is its ability to adsorb a wide variety of toxic substances even at very low concentrations. After a number of chemical attacks the remaining adsorption capacity of the activated carbon is reduced to a level that renders the protective properties of the suit unsatisfactory. For economic and logistic reasons, it would be advantageous to decontaminate the suits and re-establish the protective properties, thereby enabling the suits to be reused. The objective of the decontamination procedure is to remove the adsorbed substances from the activated carbon and thereby restoring, as far as possible, its initial adsorptive capacity. The two main routes for decontamination are *evaporation* of the CW agents induced by hot air, steam or exhaust or *laundering* using water or organic solvents with detergents or other additives.

This study is a continuation of earlier published work on laundering and thermal decontamination of NBC-protective garments (1-6). The aim is to investigate decontamination procedures for NBC-protective garments used by the Norwegian Army (NM143). Challenging agent is sulphur mustard (HD). It was shown previously that NM143 garments can be decontaminated one time by laundering at 60°C (5,6). Initial investigations have also indicated that it is possible to decontaminate NBC-protective garments by a thermal method (2-4). The most important parameters are temperature and time. The main objective of the present study is to optimise these parameters for hot air decontamination of NM143 garments and to compare the results from thermal decontamination with laundering experiments.

2 BACKGROUND

The study to identify possible methods for decontamination of NBC-protective garments was initiated in 1994. Two possible decontamination methods, thermal decontamination and laundering, were studied. A historical survey up to 1997 is given in reference (1). It is relevant to summarise the previous results of thermal decontamination.

Hot air treatment has been carried out on two types of NBC-protective garments, NM143 and the NBC-suit used by the Norwegian Navy (SM3) (2-4). Protective clothing for military purposes have to be able to protect against toxic chemicals, against rain, to repel oil and retard flames. Such characteristics can seldom be incorporated in single layer and therefore multi-layer systems are used. Both NM143 and SM3 consist of two layers. The Norwegian Army and Air force ground personnel use the NM143 garments manufactured by the Norwegian

company K Stormark and the French manufacturer Paul Boyé. It is made of activated carbon impregnated on polyurethane foam with a liquid-repellent outer layer. For SM3 the inner layer that adsorbs toxic vapours is based on activated carbon fixed on a non-woven material with a wicking outer layer.

Initial investigations indicated that protective garments NM143 and SM3 can be decontaminated using either hot air or laundering (2,3). The thermal stability of the NM143 fabric was studied by thermogravimetric analysis (TGA), and both the inner and outer layers are stable up to 150°C (2). These initial investigations indicated that it is possible to decontaminate NM143 and SM3 garments both by a thermal procedure and by laundering. The result of hot air decontamination depends strongly on temperature, time and airflow. A minimum temperature of 100°C is necessary (3). The effect of temperature combined with humidity for decontamination of NM143 was studied (4). Decontamination with humid air at 110°C and dry air at 150°C gave the best results (4). However, penetration analyses showed that the protective ability was not completely re-established, probably due to incomplete removal of the HD. Due to the limited number of experiments further investigations have been recommended.

The focus of the present study is NM143. The objectives are to:

- investigate the thermal stability of the NM143 fabric during hot air decontamination
- to optimise parameters such as temperature, humidity and time in order to maximize the decontamination effect
- to compare hot air decontamination with laundering

3 EXPERIMENTAL SECTION

3.1 Material

The NM143 (delivered by the companies K Stormark and Paul Boyé) is made of an inner layer of activated carbon impregnated on polyurethane foam and a liquid-repellent outer layer. The NM143 yard goods used in the present study was stored at K Stormark since 1991 and received at FFI January 2000. For the comparative study of decontamination by laundering and a thermal method, samples were taken from a suit, produced by Paul Boyé in 1990.

3.2 Thermal decontamination procedure

A Heraeus tube oven with a tube diameter of 6 cm was used for hot air decontamination and for the decontamination with humid air. Humid air was generated by bubbling air through a round-bottomed flask filled with boiling water. In order to give good contact between the water and the air, the flask was filled with washed gravel. The airflow was kept constant at 2 l/min (linear velocity 12 mm/s). The air was taken from the laboratory-pressurised air

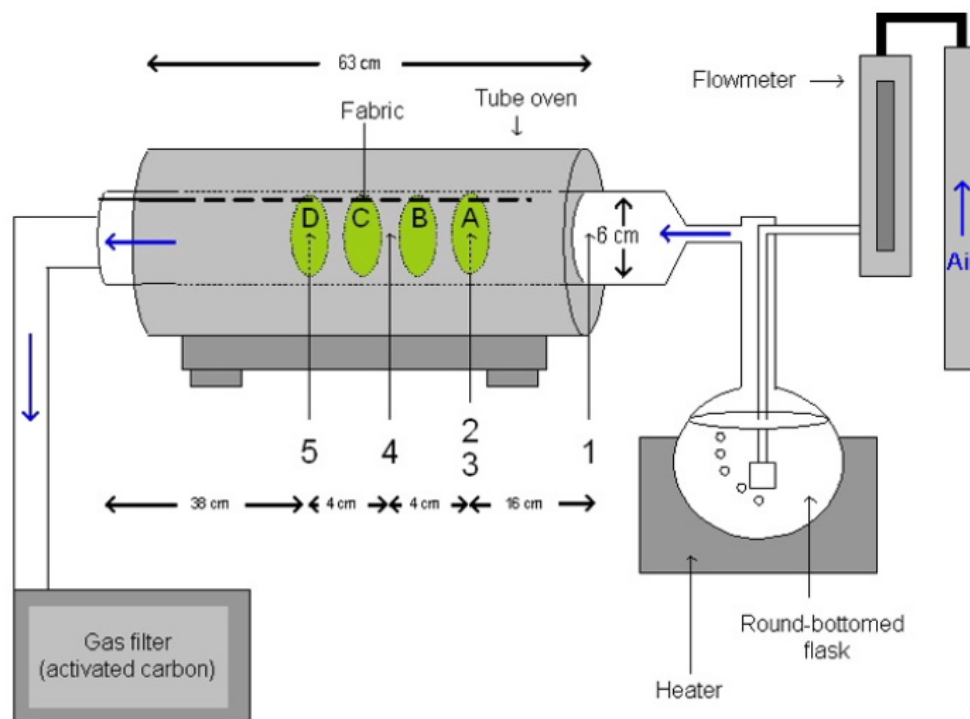


Figure 3.1 Experimental set-up for hot air decontamination and decontamination with humid air. The numbers 1–5 indicate the temperature measuring points

Temperature setting of the oven controller	Measuring points for temperature				
	1	2	3	4	5
80 °C dry air	22	70	77	79	82
100°C dry air	24	89	100	100	100
130°C dry air	30	122	126	135	135
150°C dry air	34	147	149	153	154
100°C with humid air	95	95	95	95	95
130°C with humid air	95	95	110	122	127

Table 3.1 Temperature variation in the oven. The positions where the temperature was measured are indicated in Figure 3.1

system, and had a water vapour content of ca 1 mg/l (ca 5%). Four pieces of fabric were decontaminated in each experiment (see Figure 3.1).

The temperature was checked with four pieces of fabric placed in the oven. The results are presented in Table 3.1. The temperature was measured along the centerline in the tube oven. The positions were at the entrance (position 1), just before (position 2) and after (position 3) patch A, between patches B and C (position 4) and just after patch D (position 5).

The patches were placed in a row in the oven, perpendicular to the airflow. This leads to transportation of contaminated air from patch A to B, from B to C and C to D, thus may give

transfer of contamination. The data showed no systematic variation between the various positions of the patches in the oven.

3.3 Laundering

Decontamination by laundering was performed in glass containers, which were rotated in water under temperature control in order to simulate a laundering machine (5). The amount of water corresponded to that used in a laundering machine (8 litres of water per kg textile). Four pieces of fabric were laundered at 60 °C for 45 min using 40 ml of water. The commercial detergent Blenda Sensitive (0.099 g) was added. After laundering, the patches were rinsed three times in cold water (40 ml each time).

3.4 Penetration measurements

The patches (21 - 22 cm² circular pieces) were contaminated (12.5 cm²) with liquid drops (10 x 1 µl) of HD, corresponding to a contamination load of 10g/m², which is in accordance with the NATO NBC-protective cloth triptych AEP-38. The protective properties were analysed before and after decontamination. According to the specification of the Norwegian Army, NBC-protective garments must yield a protection against liquid HD (10 g/m²) corresponding to 4 µg/cm² maximum accumulated penetration after 24 hours from exposure (skin irritating level).

The analytical apparatus used to test the penetration of HD through the protective suits consist of 16 permeation cells coupled to a gas chromatograph (GC). The HD that penetrates through the fabric is transported to the GC by a nitrogen gas flow. The nitrogen flow under the test cells was approximately 30 ml/min. HD is analysed by a flame ionisation detector. A thin polyethylene film (12 µm) was used under the test pieces to avoid nitrogen carrier gas to flow out through the fabric. The analyses were performed at room temperature (21-23 °C). The horizontal wind speed above the sample cells was approximately 0.1 m/s. Further details of the 16 cells penetration analysis system are given in reference (7).

The pieces of fabric were decontaminated 25 - 26 hours after contamination. In order to avoid evaporation of HD in the period after decontamination the pieces of fabric were wrapped in aluminium foil and stored in a freezer. It was shown that the time in the freezer did not have any effect on the result of the penetration analysis. It made no difference whether the fabrics were stored in a freezer or analysed immediately after decontamination.

3.5 Thermogravimetric analysis

Thermogravimetric analysis (TGA) was carried out on a TGA 2950 from TA Instruments. The 16 - 30 mg samples were placed in platinum crucibles and nitrogen was used as atmosphere. The temperature range was 50 - 300 °C and the heating rate was 10 °Cmin⁻¹.

4 RESULTS AND DISCUSSION

4.1 Protective properties of NM143 fabric compared to previous studies

The lifetime of the NBC- protective fabric is limited due to gradual degrading as a result of storage. The penetration data from this study are compared with earlier investigations of the NM143 (K Stormark) fabric (Table 4.1). It has been pointed out earlier that the protection against liquid HD offered by the K Stormark fabric received at FFI in the year 2000 is statistically significantly better than that of yard goods stored at FFI since 1991 (see Table 4.1). The results in this study when compared with reference (5) show no statistically significant degrading in the period from 2000 to 2001.

The codes A, C, D, E and F are used to distinguish between difference in the year of manufacture and storage conditions of the fabrics. These labels are in agreement with the notation used in reference (5). The NM143 E was used in this study for hot air decontamination and investigation of the effect of various parameters. NM143 F was used for the comparative study of hot air decontamination versus laundering. The notations NM143 E and NM143 F were simplified in the subsections, referring only to NM143.

Code	Garment description	Instrument	Year of analysis	Parallels	P_{24h} $\mu\text{g}/\text{cm}^2$	S_{P24h}	Ref.
A	K Stormark fabric stored at FFI	1 cell*	1998/99	11	1.23	0.56	(5)
C	K Stormark fabric received 2000	16 cells	2000	9	0.536	0.086	(5)
D	K Stormark fabric stored at FFI	16 cells	2000	19	1.76	0.71	(7)
E	K Stormark fabric received 2000	16 cells	2000/01	76	0.802	0.212	This study
F	NM143 suit produced in 1990 by Paul Boyé. Opened 22.11.2000, analysed 12.03.2001	16 cells	2001	8	5.09	0.55	This study

*Penetration on a single sample cell. For further details see reference (5).

Table 4.1 Penetration data before decontamination. Data from previous investigations are included for comparison. P_{24h} is the accumulated amount of penetrated HD 24 hours after initial exposure of $10 \text{ g}/\text{cm}^2$ HD, with standard deviation S_{P24h}

It is observed that the protective properties of NM143 F exceed the criterion of maximum penetrated amount $4 \mu\text{g}/\text{cm}^2$. The same effect has been reported in reference (6). It is a general trend that the protective quality of NM143 garments lies close to and sometimes just above the protection criterion immediately after unpacking. It was shown that after storage one to several months on a cloths hanger in the laboratory the protective properties of these garments tend to improve significantly (6).

4.2 Parameters in the penetration measurement procedure

The protective properties of the fabrics before and after heat treatment were analysed by measuring the rate of penetrated HD for a period of 24 hours. The penetration analyses were performed on a system that measures the vapour penetration through NM143 after challenge with liquid HD (see the experimental section and reference (7)). To gain more insight in the penetration analyses, two different parameters that may influence the rate of penetrated HD have been studied; drop size and location of the HD drops.

The drops of HD were gently placed on the surface of the fabric with a syringe. Various drop sizes were tested: 20 x 0.5 μl , 10 x 1 μl , 2 x 5 μl and 1 x 10 μl . The results are summarised in Figure 4.1 and Figure 4.2. There is a significant difference between the different drop sizes. The penetrated amount with drop size 5 μl is about 11 times higher than the level using drop size 1 μl . The result for a drop size of 5 μl is higher than for a drop size of 10 μl . The surface area of one 10 μl drop is smaller than for two 5 μl drops, which leads to less HD evaporation from the larger drop. A considerable portion of the 10 μl drop was still visible after 24 hours. In addition, the larger drop covers a smaller area than the two 5 μl drops and therefore a slower rate of penetration is observed. Penetration curves for 5 and 10 μl drops show that the penetration is not complete after 24 hours (Figure 4.2).

The results show that the suits no longer meet the protection criteria of 4 $\mu\text{g}/\text{cm}^2$ when the drop size is increased from 1 to 5 μl . The drop size 1 μl is the standard used at FFI, and this size has been used throughout this study.

Previous results showed that after a second exposure to 10 g/m^2 HD the NM143 garments do not yield a satisfactory protection (3). In this work, the effect of superimposing the drops exactly on the “old” position was compared with avoiding placing the drops at the same spot. Figure 4.3 shows that a significant smaller penetration is observed in the latter case. A second exposure leads to penetration above the criterion only when the drops are superimposed. For this reason the worst-case scenario, i.e. superimposing the drops, was used in all subsequent decontamination experiments.

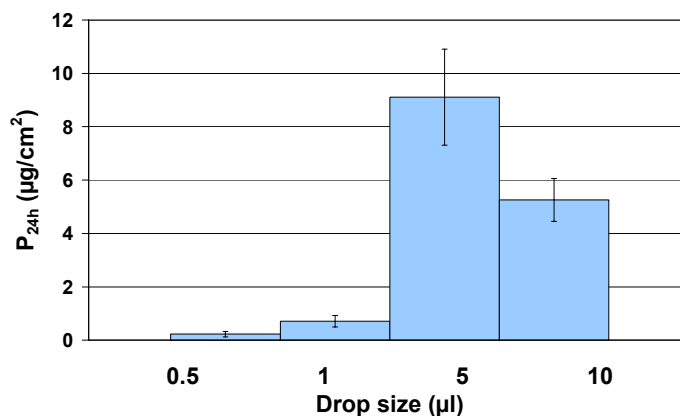


Figure 4.1 Accumulated penetrated amount of HD (P_{24h}) based on 4 parallels with standard deviation indicated as bars. The drop size was varied from 0.5 to 10 µl

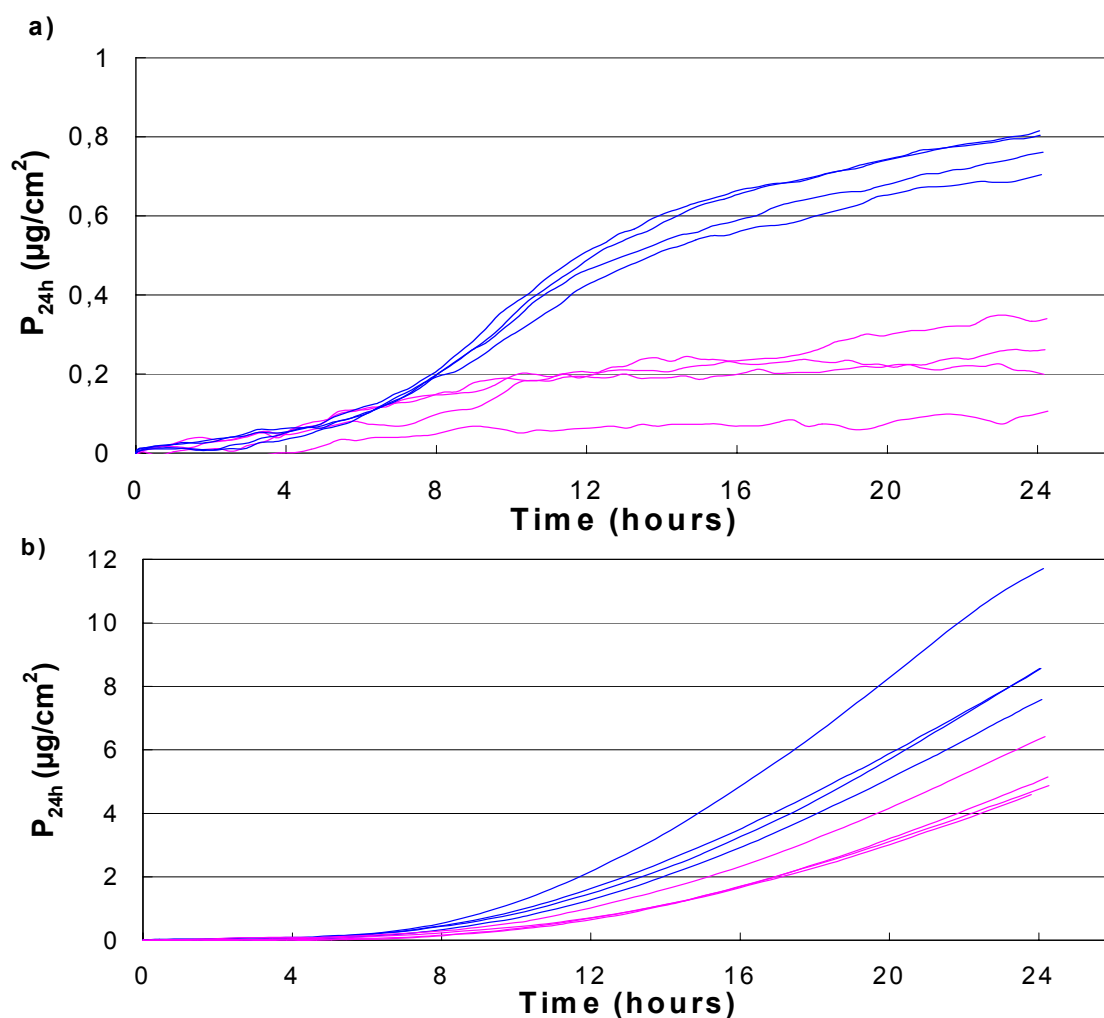


Figure 4.2 Accumulated penetration through NM143 exposed to a) 20 drops of 0.5 µl HD (pink curves) and 10 drops of 1 µl HD (blue curves) b) 2 drops of 5 µl HD (blue curves) and 1 drop of 10 µl HD (pink curves)

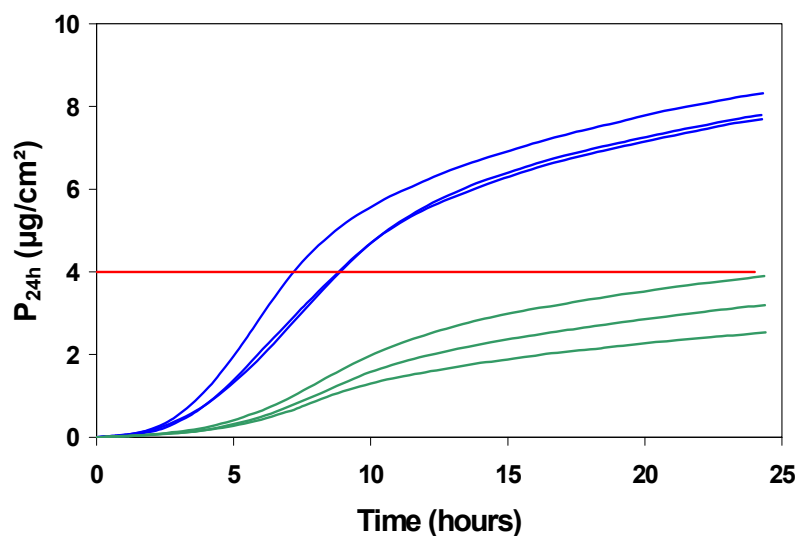


Figure 4.3 Penetration through NM143 fabric when a second set of drops is superimposed (blue curves), respectively not superimposed (green curves), on a first set of drops. The red line indicates the protection criterion (skin irritating level)

4.3 The thermal stability of NM 143

4.3.1 Thermogravimetric analysis of NM143 inner layer

The thermal stability and the relative mass loss for the inner layer of the NM143 fabric were established by TGA. The result from a TGA experiment for the NM143 inner layer, heated from 50 to 300 °C at a heating rate of 10 °C/min is shown in Figure 4.4. The onset of the degradation of NM143 is poorly defined, as expected for a polymer material with a complex composition. Weight loss of the sample occurs over the entire temperature range from 50 to 300 °C. The rate of weight loss increases slightly at ca 130 - 150 °C. This is consistent with the earlier findings in reference 2. Figure 4.5 compares TGA curves for the NM143 inner layer reported in reference 2 with results obtained in this study. The curves differ significantly above 150 °C. The heating rate was the same (10 °C/min), but different sample sizes were used. In this study the sample size was 10.2 mg, while a smaller sample (4-5 mg) was used in reference 2. The weight loss may be due to evaporation of adsorbed liquids (e.g. water, softeners and solvents) or decomposition or oxidation of the material. Measurements identifying the evolved gases are planned in the future. However, it is reasonable to suppose that the polyurethane foam is stable up to the onset of increased weight loss rate around 140 °C.

During heating of a solid sample at a programmed rate, a temperature gradient is developed within the sample. The temperature gradient increases with increasing sample mass and the scanning rate. Hence, for homogenous samples a small sample size is advantageous to minimize the variation in the results due to temperature gradients. The NM143 inner layer is however inhomogeneous and a large sample is required to obtain a representative sample. The

difference between previous and present results indicates that both the sample size and the heating rate clearly affect the TGA results for the polyurethane foam. It would be interesting to perform a more thorough analysis to investigate the influence of these parameters.

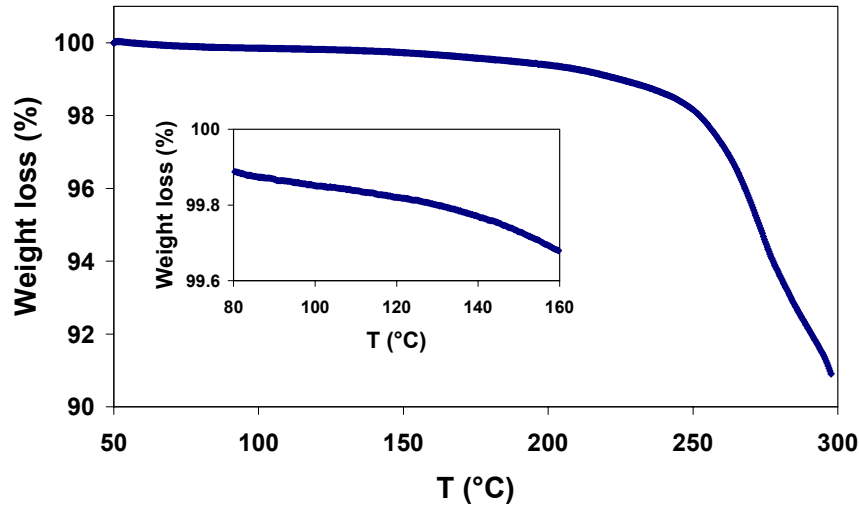


Figure 4.4 TGA data for NM143 polyurethane foam. Inset shows the same TGA data blown up in the region 80 to 160 °C

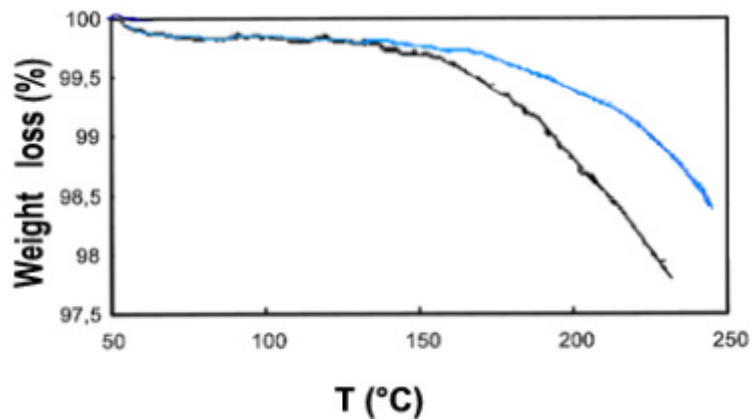


Figure 4.5 TGA curves for the NM143 inner layer reported in reference 2 (black curve) and this study (blue curve)

4.3.2 Isothermal heat-treatment of NM143 inner layer

To gain more insight in the long-term thermal stability and degradation of NM143, new clothes were kept in a climatic chamber at 85 °C for several days. Figure 4.6 shows the accumulated penetrated amount of HD for the clothes as a function of number of days at 85 °C. It is observed that the protective properties of the clothes gradually worsen. After 15 days at 85 °C the penetration of HD exceeds the protection criterion. The results show a linear relationship between time and the accumulated penetrated amount of HD.

Prolonged heating probably leads to liberation of binding materials as well as other components in the material, which might poison the activated carbon. It is important to study the thermal behaviour of each component (activated carbon, binders, polyurethane and the outer layer) in order to understand the thermal behaviour of NM143.

Heat treatment for 4 hours at fixed temperatures of 130 and 150 °C has also been performed. The results for accumulated penetrated amount of HD 20 hours after exposure were 2.3 and 2.7 $\mu\text{g}/\text{cm}^2$ for 130 and 150 °C, respectively. The average value is 0.8 $\mu\text{g}/\text{cm}^2$ for cloths that have not been subject to heat-treatment. It was thus confirmed that heat-treatment results in a higher HD penetration. The NM143 fabric still met the protection criterion; implying that the garment can stand heating at up to 150 °C for 4 hours.

Figure 4.6 shows that heating of the inner layer at the relatively low temperature of 85 °C results in serious degrading of the NM143 garment when the treatment is kept over a period of days. A polymeric and composite material will always release volatiles and age over time. An increased temperature accelerates this process. In order to optimise the decontamination procedure, it is therefore necessary to identify the minimum temperature and time needed for a satisfactory evaporation of HD from the activated charcoal layer to assure a minimum of degradation of the material itself.

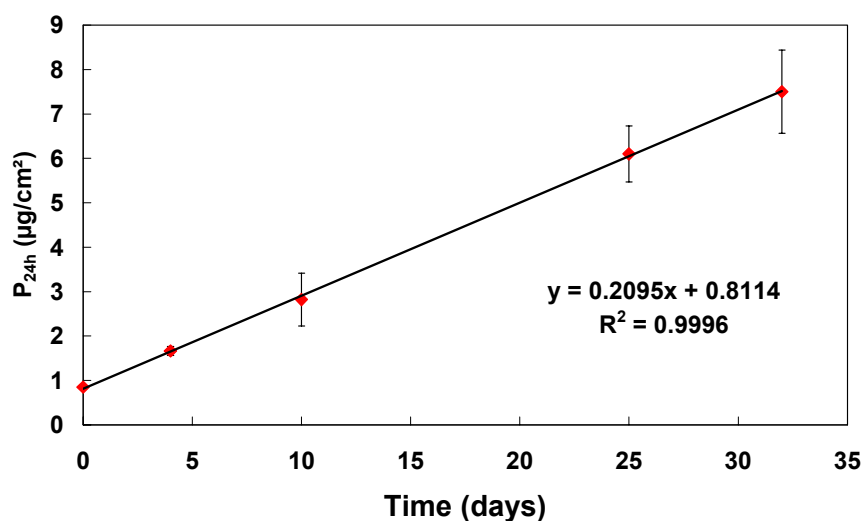


Figure 4.6 Effect of penetrated amount of HD after heat treatment of NM143 at 85°C, each data point is the mean of six parallel experiments and the standard errors are given as bars

This investigation has shown that the temperature changes the garments properties. Further investigations in the temperature range 30 to 80 °C are necessary. Important questions are: What will happen to the cloth in a tropical atmosphere of 35 °C with high humidity for several months? What is the effect on the protective properties after storage in a vehicle or a similar container in the temperature range 40 – 60 °C?

4.4 TGA experiments on contaminated cloth

The polyurethane foam was contaminated with methyl salicylate (MS) vapour and subsequently analysed by TGA. MS has a vapour pressure very similar to HD, thus it is often used as a non-toxic simulant for HD. Figure 4.7 shows the weight loss curves (a) and their derivative thermogravimetric (DTG) curves (b) for both the contaminated sample and a reference sample. Evaporation of MS occurs over a wide temperature range. The TGA curves indicate that evaporation of MS starts at ca 90 °C. Most MS have evaporated at ca 250 °C, since there is no difference in the rate of weight loss between the contaminated and the reference samples above that temperature, as shown by the DTG data.

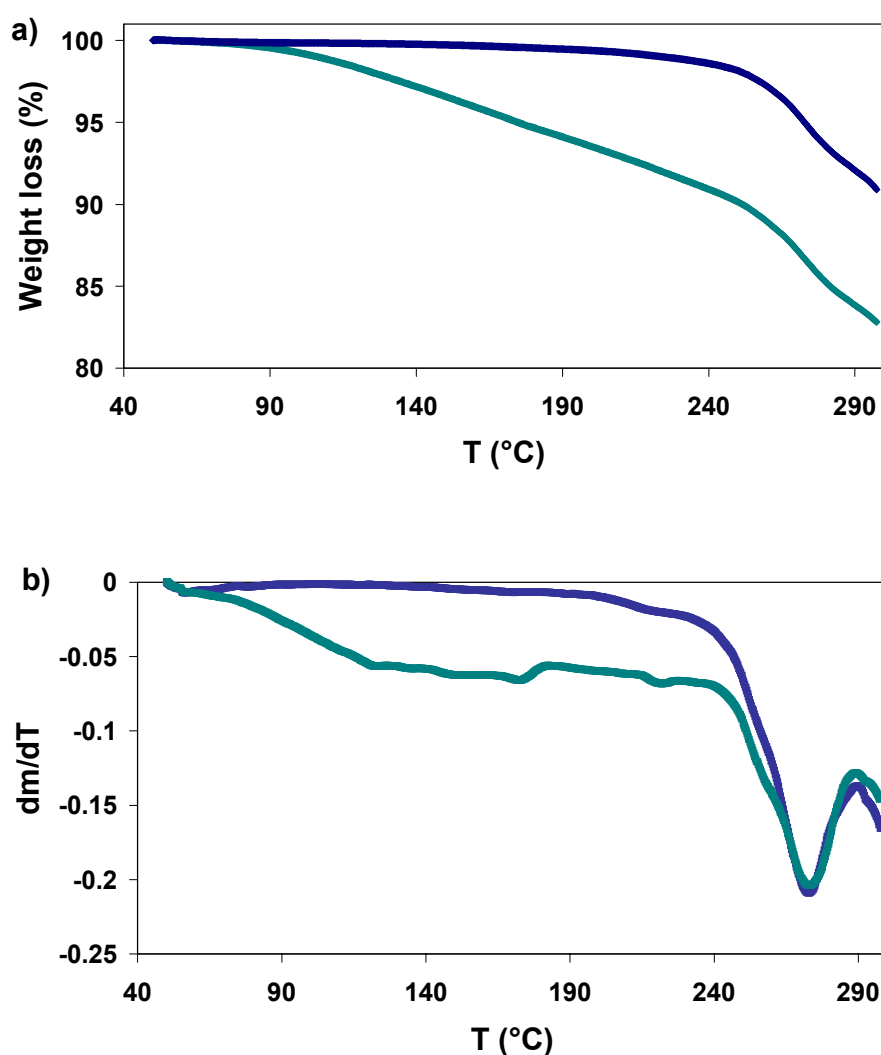


Figure 4.7 a) TGA and b) DTG data for NMI43 (blue curve) and NMI43 contaminated with 12 weight % methyl salicylate (green curve). Heating rate 10 °C/min

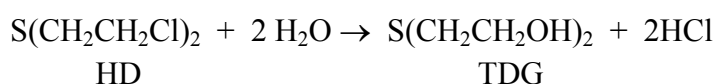
These results are included here to give an idea of the potential of TGA experiments in studying thermal decontamination and thermal stability of various materials. Further experiments, isothermal as well as with different heating rates and different heat-treatment time intervals, would give insight into the evaporation of MS or HD from the NM143 fabric. Such experiments, also with the real CW agent HD and other CW agents, are now possible using a new instrument at FFI, a TGA coupled to a mass spectrometer. In such experiments it is possible to identify the off-gassing components and at which temperatures various components evaporates.

4.5 Thermal decontamination of NM143

During thermal decontamination several parameters that may influence the protective properties were systematically varied. Since the NM143 fabric has a complex composition and morphology of activated carbon fixed on polyurethane foam, it is important not to overlook parameters, which may influence the adsorptive properties. Altogether four important parameters were identified; time, temperature, humidity and gas flow. The first three were varied one at a time to identify the most favourable values for decontamination. The gas flow was kept constant at 2 l/min.

The decontamination temperature plays an important role for the success of the decontamination efficiency. The applicable temperature range is from 25 to 150°C due to the limited thermal stability of the NM143 fabric. Together with the temperature, the time is also critical for the decontamination efficiency. Heat-treatments for 1, 4 and 24 hours were performed.

Heating of NM143 fabric induces desorption of HD from the activated carbon surface. Humid air may lead to hydrolysis of HD to thiodiglycol (TDG):



It was shown by Watterud that introducing humidity at 110°C gave better results than dry air at the same temperature (4). Hence, experiments with and without humidity were performed also in this study.

Textiles are quite inhomogeneous and the values of accumulated penetrated amount of HD may vary a great deal. Hence, it is important to carry out several parallel experiments before statistically significant conclusions can be drawn. In this work, four to six parallels were measured. The penetration data before and after decontamination are given in Appendices B and C.

4.5.1 Decontamination with dry air

Contaminated pieces of fabric were heated at selected temperatures (80, 100, 130 and 150 °C) for 1, 4 and 24 hours, respectively. For reference, cloths that were stored at room temperature (25 °C) were also analysed. The resulting protective properties are summarised in Figure 4.8.

At 80 and 100 °C satisfactory protective properties are obtained for heat-treatment for 1, 4 and 24 hours. At 130 °C the protective properties are re-established after heating for 1 and 4 hours, while heating for 24 hours gave results above the protection criterion, probably due to thermally induced degradation. At 150 °C, only the shortest heating time of 1 hour gave satisfactory protection properties.

In conclusion, decontamination of NM143 with respect to HD is achieved over a wide temperature range of 80 to 150 °C and heating for 1 hour. This minimises the degradation of the fabric and it is also the fastest procedure.

The most important conclusion from this study as compared to previous results is that sufficient thermal decontamination can be achieved both at lower temperatures and shorter time periods than previously investigated. The present results are promising as the basis for a decontamination procedure based on thermal treatment of garments.

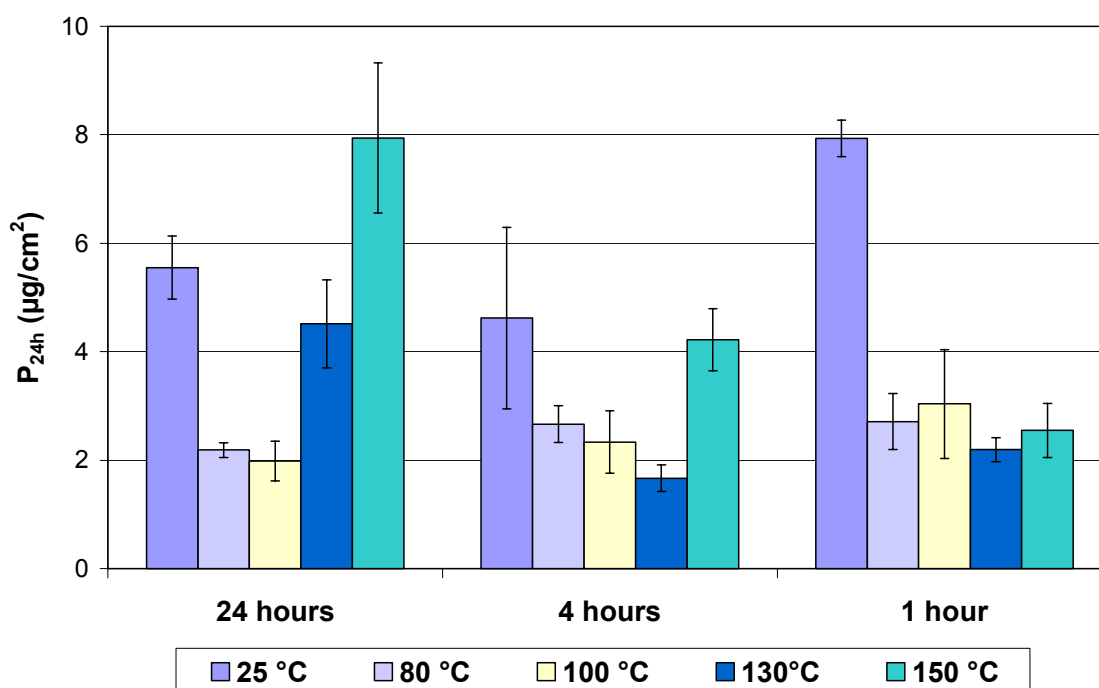


Figure 4.8 Accumulated penetrated amount of HD (P_{24h}) for NM143 after heat treatment for 1, 4 and 24 hours, respectively

4.5.2 Decontamination with humid air

Selected favourable decontamination procedures (100 and 130 °C for 1 and 4 hours) were also tested with humid air atmosphere. The results are summarised in Table 4.2 and Figures 4.9 and 4.10, which show that the experiments gave good results.

The results at 130 °C show that the protective properties after decontamination are not affected by introduction of humidity in the air (Figure 4.9). The difference in the mean values of the decontamination with and without humid air is not large enough to rule out that the difference is due to random sampling variability. There is no statistically significant difference between the accumulated penetrated amounts of HD.

Temp. / °C	Decontamination time / hours	P _{24 hours} / µg/cm ² dry air	P _{24 hours} / µg/cm ² humid air
100	1	3.0	1.4
100	4	2.3	1.1
130	1	2.2	1.9
130	4	1.7	1.6

Table 4.2 Average accumulated penetrated amount of HD (P_{24h}) after decontamination at 100 and 130 °C with and without humid air

After the pieces of fabric have been treated with humid air at 130 °C, the liquid repellent quality of the outer material is reduced. It was observed that the droplets were spread on the material. The penetration curves in Figure 4.9 show an earlier breakthrough of HD after treatment in humid air. However, the total penetrated amount after 24 hours are similar. Wicking of the HD drops accelerates the evaporation. Also, a larger area of the fabric is exposed. Both effects contribute to less total HD penetration.

After heat treatment at 100 °C with humid air, a significant decrease in accumulated penetrated amount is observed as compared to heating in dry air (Figure 4.10). This is in accordance with previous studies (4). The reason for the improved results with humid air as compared to dry air at 100 °C is most probably due to enhanced removal of HD from the carbon by the effect of water. It is evident from the data in Figure 4.10 that using humidified air at 100 °C does not change the wicking properties of the outer layer. The explanation may lie in the inner layer and an enhanced decontamination effect in the inner layer, or it may also be partly due to the outer layer. In order to investigate this, the inner layer has to be decontaminated and analysed with a non-treated outer layer and vice versa.

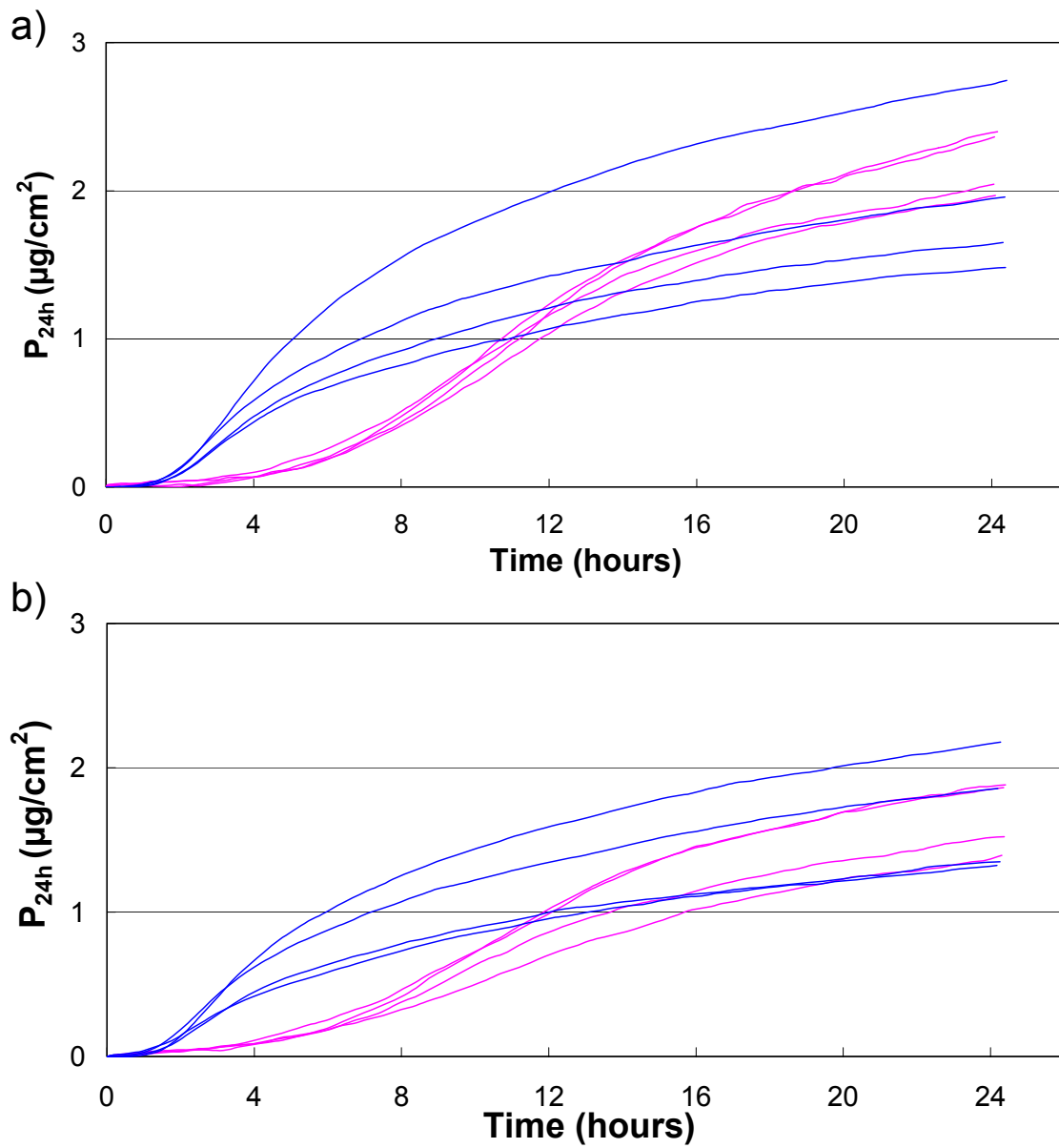


Figure 4.9 Accumulated penetrated amount of HD for NMI43 after heat treatment at 130 °C with dry (pink curves) and humid (blue curves) air for a) 1 hour and b) 4 hours

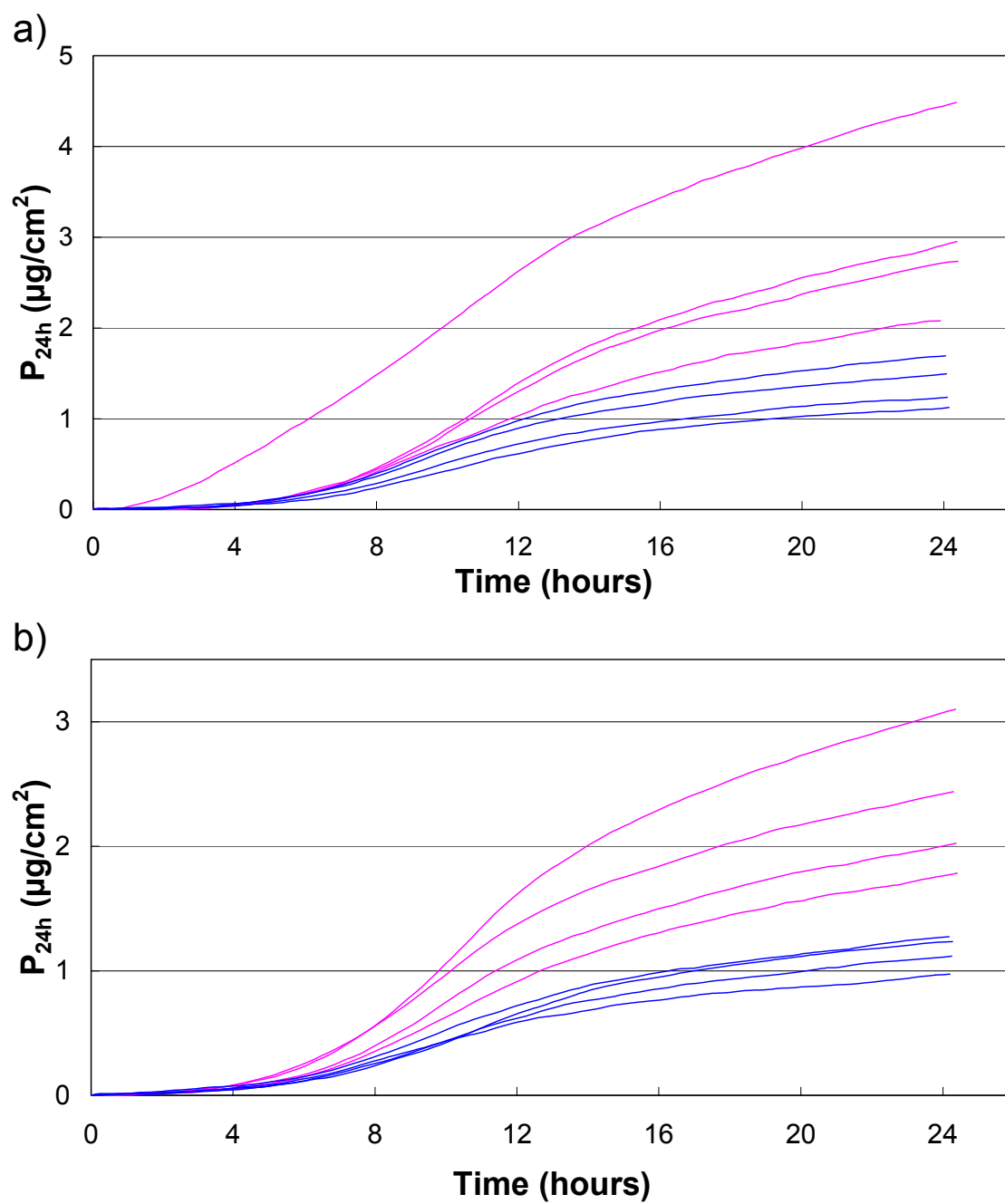


Figure 4.10 Accumulated penetrated amount of HD for NM 143 after heat treatment at 100 °C with dry (pink curves) and humid (blue curves) air for a) 1 hour and b) 4 hours

4.6 Comparison between decontamination by hot air and laundering

For the comparative study of hot air and laundering decontamination, NM143 F cloth from a suit was used instead of the yard cloth. The suit had been stored in the original sealed packaging from its production date (1990). After opening (22.11.2000) the suit was stored in the laboratory on a cloth hanger at stable room temperature (20-22 °C). Due to seasonal changes the relative humidity (RH) varied. The protection data were analysed before decontamination (12.03.01) and after decontamination (14.03.01). The RH these days was between 20 – 30%. Before decontamination the suit did not satisfy the protection criterion (see chapter 4.1).

The hot air decontamination was performed at 100 °C for 4 hours with humid air. Decontamination by laundering was performed at 60 °C. The results are summarised in Figures 4.11 and 4.12. After decontamination by washing and heat treatment the protective properties were better than before any treatment of the fabric. All parallels are below the protection criterion after decontamination. There is not a statistically significant difference between hot air and washing decontamination results. Both procedures re-establish the protection criterion, and are thus equally good.

The TDG contents of the laundering water and first time rinsing water were analysed by GC. The TDG content in the laundering- and rinsing waters corresponded to 9.9% and 6.1% of the initial HD contamination, respectively. The fact that only a small amount of the HD can be accounted for in the laundering and –rinsing waters needs further investigations. Some of the HD has evaporated and some is still adsorbed on the activated charcoal surface, or absorbed in the binders or the polyurethane foam. It is recommended to investigate the fate and location of the remaining HD after laundering, especially to identify whether it remains in the fabric as HD or whether it has been hydrolysed to TDG or oxidised to other compounds. TG-MS analysis would be a helpful technique to analyse the remaining components after laundering and hot air decontamination experiments.

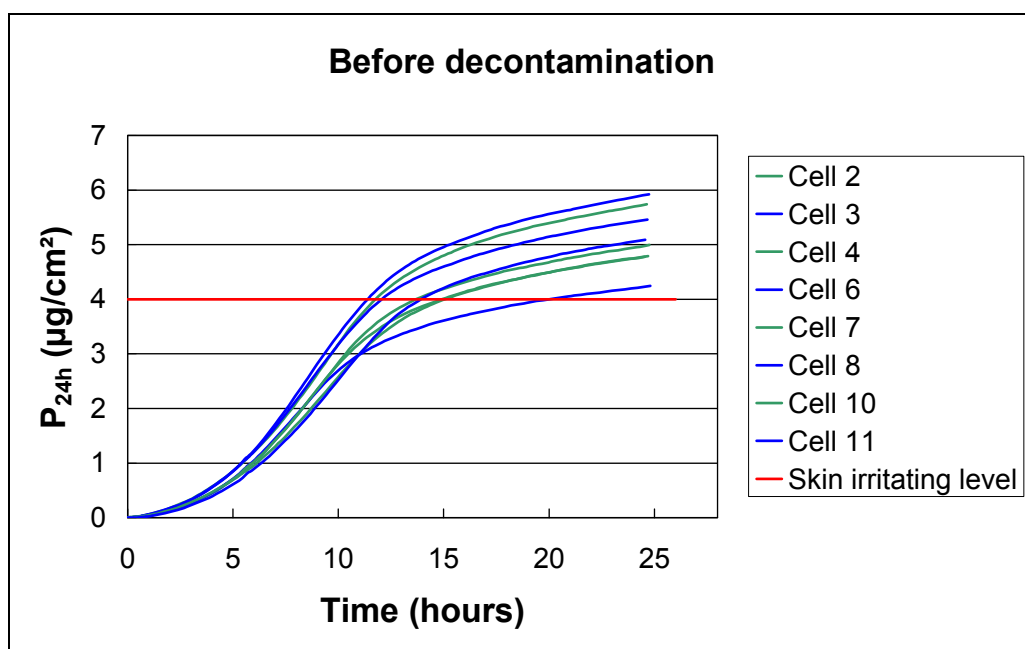


Figure 4.11 Accumulated penetrated amount of HD through NMI43 F, before hot air decontamination (blue curves) and before laundering (green curves)

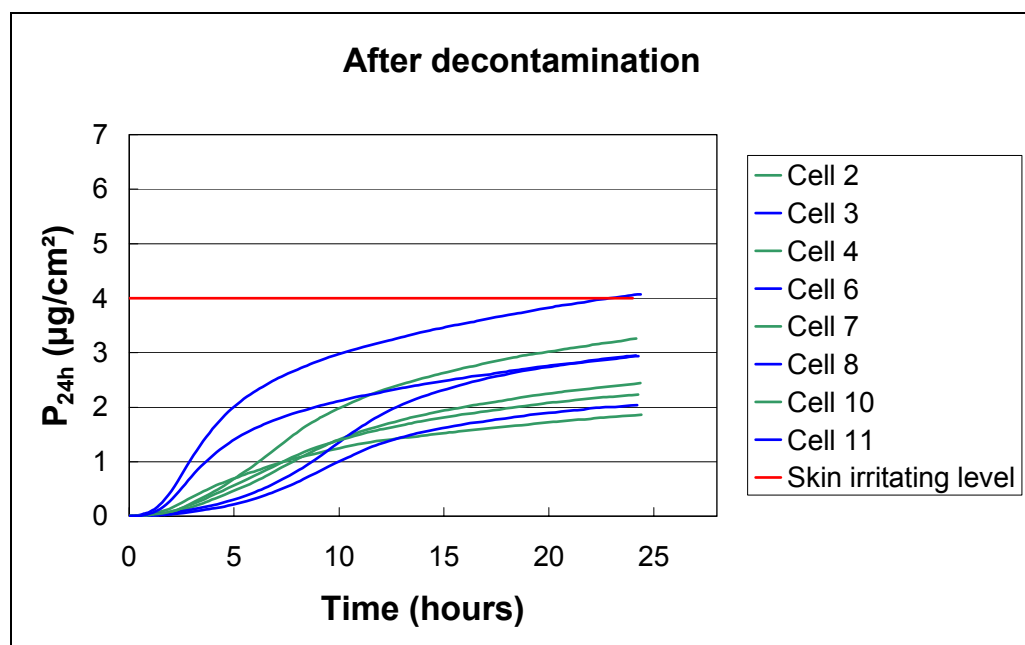


Figure 4.12 Accumulated penetrated amount of HD through NMI43 F, after hot air decontamination (blue curves) and after laundering (green curves)

5 CONCLUSIONS

The thermal decontamination of NM143 suits has been studied at selected temperature from 80 and to 150°C. Circular pieces of cloths were challenged with 10 g/m² sulphur mustard (HD) and subsequently decontaminated with hot air. The protective properties of the fabric against liquid HD drops were measured before and after decontamination.

The present results are very promising as the basis for a decontamination procedure based on thermal treatment of garments. Small-scale experiments show that efficient decontamination can be obtained in a wide temperature range between 80 to 150 °C, with a time for the heat treatment of 1 hour and airflow of 2 l/min. Introducing humidity into the airflow at 100 °C gave improved results. The NM143 fabric is thermally resistant to these decontamination conditions.

Heat-treatment according to procedures from this study was compared with the recommended laundering procedure from previous studies (5,6). This was heat-treatment for 4 hours at 100 °C in humid air (airflow 2 l/min) and laundering at 60 °C for 45 min, and rinsing three times in cold water. Equally good results were obtained, and both procedures restored the adsorption capacity of the fabric.

If a thermal decontamination procedure is chosen and procurement of a container based system initiated, full-scale experiments are necessary to validate the effectiveness of the procedure, both for garments and other types of gear such as respirators, boots, gloves, weapons etc. The arrangement of the suits and other items in such a container is probably very important in order to ensure an optimum temperature and sufficient airflow.

APPENDIX

A EFFECT OF DROP SIZE AND POSITION ON PENETRATION DATA

Datafile 1 st time	Cell nr.	Br / hours	P _{24h} / µg/cm ²	Average	Comment
010109	7	*	0.208	0.099	Effect of drop size. 0.5 x 20µl
	8	*	0.258		
	10	*	0.333		
	11	*	0.097		
			*	0.802	Effect of drop size. 10 x 1µl, Average for 76 first time penetrations.
	2	10.83	8.567	9.107	Effect of drop size. 2 x 5µl
	3	10.46	8.565		
	4	11.28	7.584		
	6	9.73	11.713		
	010108	7	12.17	6.419	5.257
8		14.20	4.591		
10		13.85	5.141		
11		13.87	4.879		
001114	10	4.65	7.793	7.936	Effect of location of HD. Super-imposed drops
	11	4.67	7.695		
	12	3.90	8.319		
001114	14	7.55	3.901	3.211	Effect of location of HD. Placed "beside"
	15	7.97	3.194		
	16	8.80	2.539		

Table A.5.1 Penetration data for investigation of parameters. P_{24h} is the accumulated amount of penetrated HD 24 hours after exposure. Br is the breakthrough time defined as the time when the penetrated amount of HD exceeds $0.1 \mu\text{g}/\text{cm}^2$

B DECONTAMINATION DATA FOR DRY AIR THERMAL TREATMENT

Data file before decon.	Data file after decon.	Cell nr.	T / °C	Time / hours	Br / hours	P _{24h} / µg/cm ² before decon.	P _{24h} / µg/cm ² after decon.	Average after decon.
010219	010221	2	25	24	6.05	0.815	6.123	5.557
		3			6.07	0.804	5.732	
		4			6.90	0.704	4.743	
		6			6.15	0.761	5.613	
001211	001214	2	80	24	10.45	1.029	2.325	2.187
		3			10.48	0.950	2.162	
		4			10.90	0.948	2.249	
		6			10.95	0.602	2.012	
001205	001207	12	100	24	9.48	1.323	2.463	1.983
		14			10.73	1.282	2.075	
		15			12.36	0.649	1.682	
		16			11.58	1.041	1.712	
001127	001130	2	130	24	8.05	0.469	2.758	4.364
		3			6.47	0.542	4.242	
		4			5.70	0.455	4.790	
		6			5.75	0.498	5.067	
010102	010104	12			7.10	0.963	4.169	
		14			6.35	0.962	5.479	
		15			7.57	0.610	4.238	
		16			7.60	0.692	4.171	
010115	010122	12	150	24	4.70	0.646	8.670	7.940
		14			4.75	0.806	9.052	
		15			5.57	0.794	5.948	
		16			4.80	0.548	8.091	
010115	010122	2	25	4	8.50	0.746	3.294	4.619
		3			7.30	0.633	4.348	
		4			8.10	0.709	3.411	
		6			2.95	0.741	7.425	
010212	010215	7	80	4	10.57	0.705	2.350	2.664
		8			9.80	0.721	2.476	
		10			10.25	1.398	2.710	
		11			9.47	0.773	3.122	
010212	010215	12	100	4	10.30	0.919	2.438	2.336
		14			9.95	1.188	3.098	
		15			11.57	0.632	2.025	
		16			12.80	0.755	1.784	
001120	001122	12	130	4	15.90	0.470	1.394	1.665
		14			12.40	0.502	1.863	
		15			14.00	0.432	1.522	
		16			12.00	0.425	1.882	

Data file before decon.	Data file after decon.	Cell nr.	T / °C	Time / hours	Br / hours	P _{24h} / µg/cm ² before decon.	P _{24h} / µg/cm ² after decon.	Average after decon.
001128	001130	12	150	4	7.90	0.889	3.713	4.221
		14			7.15	0.907	4.055	
		15			6.77	1.069	5.047	
		16			7.20	0.826	4.072	
001113	001114	10	25	1	4.65	*	7.793	7.936
		11			4.67	*	7.695	
		12			3.90	*	8.319	
010108	010109	2	80	1	12.90	0.717	1.975	2.713
		3			8.90	0.563	3.171	
		4			9.30	0.789	2.925	
		6			10.20	0.625	2.782	
010108	010109	12	100	1	12.3	0.819	2.072	3.037
		14			6.4	1.127	4.442	
		15			10.8	0.759	2.913	
		16			10.8	0.609	2.721	
010102	010104	2	130	1	11.30	0.947	2.045	2.194
		3			10.90	0.741	2.365	
		4			12.10	0.593	1.968	
		6			11.40	0.920	2.399	
001130	001201	7	150	1	10.60	0.935	1.979	2.548
		8			9.80	0.875	2.291	
		10			9.10	1.005	2.902	
		11			8.30	1.026	3.021	

Table B.1 Penetration data before and after thermal decontamination of NMI43 at various temperatures. Br is the breakthrough time defined as the time when the penetration amount of HD exceeds 0.1 µg/cm². The accumulated penetrated amount of HD, 24 hours after exposure to 10 g/cm² HD (P_{24h}) is given before and after decontamination

C DECONTAMINATION DATA FOR THERMAL TREATMENT IN HUMID AIR

Data file before decon.	Data file after decon.	Cell nr.	T / °C	Time / hours	Br / hours	P _{24h} / µg/cm ² before decon.	P _{24h} / µg/cm ² after decon.	Average after decon.
010219	010221	7	130	4	13.37	0.672	1.324	1.676
		8			7.40	0.593	1.855	
		10			12.25	0.733	1.348	
		11			6.27	0.918	2.176	
010219	010221	12	130	1	9.10	0.838	1.650	1.959
		14			7.15	1.249	1.958	
		15			11.17	0.747	1.482	
		16			5.20	0.753	2.746	
010306	010309	7	100	4	16.57	0.869	1.273	1.150
		8			*	0.733	0.974	
		10			20.25	1.077	1.117	
		11			17.47	0.977	1.235	
010306	010309	2	100	1	12.45	0.907	1.693	1.388
		3			13.27	0.935	1.494	
		4			16.90	0.715	1.239	
		6			19.35	0.875	1.124	

Table C.1 Penetration data before and after thermal decontamination of NM143 at various temperatures. Br is the breakthrough time defined as the time when the penetration amount of HD exceeds 0.1 µg/cm². The accumulated penetrated amount of HD, 24 hours after exposure to 10 g/cm² HD (P_{24h}) is given before and after decontamination.

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