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HASKELL LABORATORY

cc: [REDACTED]

January 13, 1984

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TEXTILE FIBERS DEPARTMENT
GENEVA

INDUSTRIAL HYGIENE SURVEY

[REDACTED] & [REDACTED]
IN THE U.K., NETHERLANDS AND GERMANY
(Haskell Laboratory Report No. 29-84; [REDACTED])

SUMMARY

Industrial hygiene surveys for [REDACTED] emission at heat-setting operation were done at three customer plants: Richards Ltd. in Aberdeen, Scotland; W & J Knox Fibers, Ltd. at Kilbirnie, Scotland; and at Ornatex at Kirkcraze, Netherlands. In addition, both [REDACTED] and [REDACTED] were monitored at Uentrop Werk in Germany.

The [REDACTED] samples at the three heat-setting plants cannot be considered to be "breathing zone" samples as defined in Du Pont Engineering Standard S-12-T; but, with the exception of two samples at Knox, they were placed at approximately nose level in locations where people normally worked. The [REDACTED] samples were in the range 0.3-1.5 mg/m³, which is well below the 5 mg/m³ AEL. These samples should approximate breathing zone concentrations.

At Uentrop Werk three types of samples were taken near a mist formed when fiber containing a newly applied finish [REDACTED] made a 90° bend over a guide: [REDACTED] fumes, total and respirable particulates, and fractions of particulates less than 10 µm. The [REDACTED] concentrations near the finish mist were in the range 0.8-1.0 mg/m³. Total particulates were approximately 24 mg/m³ of which about 4-6% were less than 5 µm.

The impactor sampling for fractions shows a mass median aerodynamic diameter of 8 μ m. None of the Uentrop Werk samples were considered to be in the breathing zone of workers. They are expected to give higher results than breathing zone samples due to close proximity to sources of emissions.

BACKGROUND

[REDACTED] is used in formulations of [REDACTED]. [REDACTED] is present as a contaminant in the [REDACTED] formulations and is also formed as a thermal degradation product when [REDACTED] are subjected to heat-setting.

It was desired to characterize the exposure of workers near the heat-setters to [REDACTED] in several customer plants in the United Kingdom and the Netherlands. Additionally, a request was made to monitor near a spinning machine which was applying a [REDACTED] to determine the total and respirable particulates concentrations, the [REDACTED] concentration and the particle size distribution.

SAMPLING AND ANALYSIS METHODS

[REDACTED] sampling and analysis were done per Haskell Laboratory Analytical Method [REDACTED]. [REDACTED] samples were taken using Du Pont Model P-200A sampling pumps. Sample tubes were SKC Tenax® 100/50 mg tubes with glass sealed ends (Cat#226-35-03, Lot 203). Flow rates were in the 100 mL/minute range. The pumps were calibrated before and after each sample (except samples -028 and -029) with a Kurz Flowmeter Model 541S, which itself was calibrated by bubble meter prior to the survey [REDACTED]. Pumps for samples -028 and -029 were calibrated directly with a bubble meter at Uentrop. Sampling details for all samples are on individual industrial hygiene data forms in the [REDACTED] file.

Analysis of the [REDACTED] samples was done at Haskell Laboratory using the solvent desorption/gas chromatography method described in [REDACTED]. A blank sample was submitted with each batch of samples from the various sites.

[REDACTED] Total & Respirable Particulates

Total and respirable [REDACTED] particulates were collected using filter cassettes (Millipore Gravimetric PVC 5-03700) and Du Pont P-2500A pumps. The P-2500A pumps were calibrated using the Kurz 541S flowmeter or directly with a bubble meter. Flow rates were in the 1.8 Lpm range. The filter cassettes were weighed before and after sampling at Haskell Laboratory and were desiccated 16-24 hours before each weighing.

The respirable particulate sample train had a cyclone separator ahead of the filter cassette. The cyclone/filter cassette assembly constitutes the Bendix BDX 99 Respirable Cyclone Dust Collector. The following table shows the percentages of particles of various aerodynamic diameters which pass through the cyclone and into the filter cassette. The particles passing through this cyclone are considered to be the respirable fraction.

<u>Aerodynamic Diameter in Micrometers</u>	<u>Percentage Passing Through Cyclone</u>
< 2.0	90
2.5	75
3.5	50
5.0	25
10.0	0

After the filter substrates for the total and respirable particulates were weighed, they were sent to Dr. Eric Kissa (Jackson Laboratory, Chambers Works) for fluorine analysis.

Particulate Fractions

A Model 2110 ten stage Sierra cascade impactor with a cyclone pre-separator (10 μ m cutoff) was used to determine the particle size distribution of the finish mist. Airflow through the impactor at 3.4 Lpm was generated by a vacuum pump. The Kurz 541S mass flowmeter was used in-line with the impactor and vacuum pump to measure the airflow. The Kurz flowmeter was calibrated before and after the impactor run with a bubble meter on site. The collection substrates for each of the ten stages were C-220 GF-Type A glass fiber filter papers which were desiccated and pre-weighed on site. After sampling the filters were again desiccated, weighed and sent directly to Dr. Eric Kissa for fluorine analysis.

RESULTS

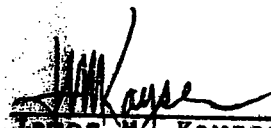
Tables 1, 2 and 3 show the monitoring results at Richards, Knox and Ornatex. All samples were well below the Acceptable Exposure Limit of 5 mg/m³.

Table 4 shows the location and type of sampling done at Ventrop Werk and Tables 5 & 6 show the results of the analyses of the samples at that site. Table 7 shows the method of determining the percent respirable particles in the Sierra impactor test. Table 8 shows the results of the fluorine analysis on the respirable and total particulate samples. The fluorine analyses on the impactor fractions are shown in Table 9.

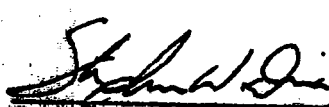
DISCUSSION & RECOMMENDATIONS

- The [redacted] emission at the customer plants seem to be under control. A spot check for emissions at the heat-setters should be performed approximately every two years or whenever conditions such as heat-setting temperature, throughput, number of machines or ventilation change so as to increase the [redacted] released.
- The test for particulates at Uentrop Werk using the BDX sampler resulted in an apparent respirable portion of 4-6%, while the respirable portion calculated from the Sierra impactor study was approximately 11%.
- Fluorine analysis by Dr. Eric Kissa on the total and respirable particulate samples (-022, -023, -030 and -031) indicate that the respirable portion is 62-66% [redacted] while the total particulates are approximately 44% [redacted]. Preliminary testing for fluorine on the impactor fractions show that the particles in the 0.5-6.4 μ m size range consist of approximately 20-40% [redacted].

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Date Issued: January 13, 1984

[redacted]
Haskell Laboratory Report Number 29-84

Attachments: Tables 1-9

Number of pages in this report: 13

TABLE 1

[REDACTED] MONITORING AT RICHARDS LTD., ABERDEEN, SCOTLAND

Nylon staple spun from Antron® Plus Type 309A heat-set on Cumerba equipment. Heat-setting temperature was 132°C and dryer temperature was 75°C. Sample date: October 4, 1983.

Sample Number	Location of Sample	Sample Time (min.)	Concentration [REDACTED] (mg/m ³)
H-83-38-001	Three feet from exit of presteamer, 12" above belt near control buttons, 2' from entry rollers, 6" from edge of yarn.	176	0.3
H-83-38-002		176	0.3
H-83-38-003	Two feet from exit of dryer, 3' above belt. Attached to yarn takeoff accumulator support stand.	167	0.4
H-83-38-004		167	0.4
H-83-38-005	Between dryer and setting chamber. Directly over and 2 1/2' above belt. Two feet horizontally from exit roller of setting chamber.	148	0.6
H-83-38-006		178	0.5
H-83-38-007	Blank		Non-detectable

TABLE 2

[REDACTED] MONITORING AT KNOX FIBERS, KILBIRNE, SCOTLAND

[REDACTED] heat-set on Suessen equipment at 200°C. Sampling date: October 5, 1983.

<u>Sample Number</u>	<u>Location of Sample</u>	<u>Sample Time (min.)</u>	<u>Concentration { [REDACTED] } (mg/m³)</u>
H-83-38-008	Blank		Non-detectable
H-83-38-009	Over wrapper near	147	1.1
H-83-38-010	entrance to heat-setter. Eighteen inches from wrapper and 14" from bottom lip of exhaust hood.	148	1.0
H-83-38-011	On posts either side of	146	0.9
H-83-38-012	exit from heat-setter. Twelve to fifteen inches from either side of hood at level of bottom lip of hood.	145	1.0
H-83-38-013	Immediately over (outside)	67	1.0
H-83-38-014	yarn masts at exit of heat-setter (under exhaust hood).	65	1.5

TABLE 3

[REDACTED] SAMPLING AT ORNATEX, KIRKRADE, NETHERLANDS

[REDACTED] heat-set at 132°C on Superba equipment.
Sampling date: October 10, 1983.

Sample Number	Location of Sample	Sample Time (min.)	Concentration (mg/m ³)
H-83-38-015	Blank		Non-detectable
H-83-38-016	Above conveyor belt at exit of heat-setter. Samples were 22" above the conveyor and 30" horizontally from the exit.	163	1.0
H-83-38-017		161	0.9
H-83-38-018	Directly above the entrance to the heat-setter. Samples were 15" above and either side of the conveyor.	154	1.0
H-83-38-019		151	1.0
H-83-38-020	On the machine (not running) adjacent to the one running P185. Samples were opposite the exit of the heat-setter. Fumes from machine running P185 were wafting in the direction of the samples, which were 4 1/2" from the floor.	147	1.4
H-83-38-021		147	1.4

TABLE 4

[REDACTED] AND PARTICULATE SAMPLING AT UENTROP WERK
LOCATION AND TYPES OF SAMPLES

Location of Samples

Sampling was done at BCF Spinning Machines 31 & 32. [REDACTED] was being finished with [REDACTED] at a throughput of 34 kg/hr. The yarn ran vertically to machine 32 position 01, then horizontally 152 cm to machine 31 position 10, then vertically 46 cm down to the doffing gun. A visible mist was formed as the yarn passed over the guide at 31-10. The Sierra impactor preselector orifice was 36 cm diagonally above and to the right of the 31-10 guide. The samplers for [REDACTED] and particulates were in a zone which was approximately 24 cm above, 15 cm to the right, and 18 cm behind the guide.

<u>Sample Number</u>	<u>Type Sample</u>
H-83-38-022	Respirable particulate with BDX 99.
H-83-38-023	Total particulate - filter cassette.
H-83-38-024	[REDACTED] Tenax tube.
H-83-38-025	[REDACTED] Tenax tube.
H-83-38-026	Respirable fractions - Sierra impactor.
H-83-38-027	Blank Tenax tube.
H-83-38-028	[REDACTED] Tenax tube.
H-83-38-029	[REDACTED] Tenax tube.
H-83-38-030	Total particulates - filter cassette.
H-83-38-031	Respirable particulates with BDX 99.

TABLE 5

**[REDACTED] AND PARTICULATE SAMPLING AT UENTROP WERK
RESULTS OF ANALYSIS**

<u>PARTICULATES</u>			
<u>Sample Number</u>	<u>Time (min.)</u>	<u>Concentration (mg/m³)</u>	<u>Particulate Type</u>
H-83-38-022	365	1.34	Respirable
H-83-38-023	363	22.32	Total
H-83-38-030	174	25.55	Total
H-83-38-031	174	.97	Respirable

[REDACTED]

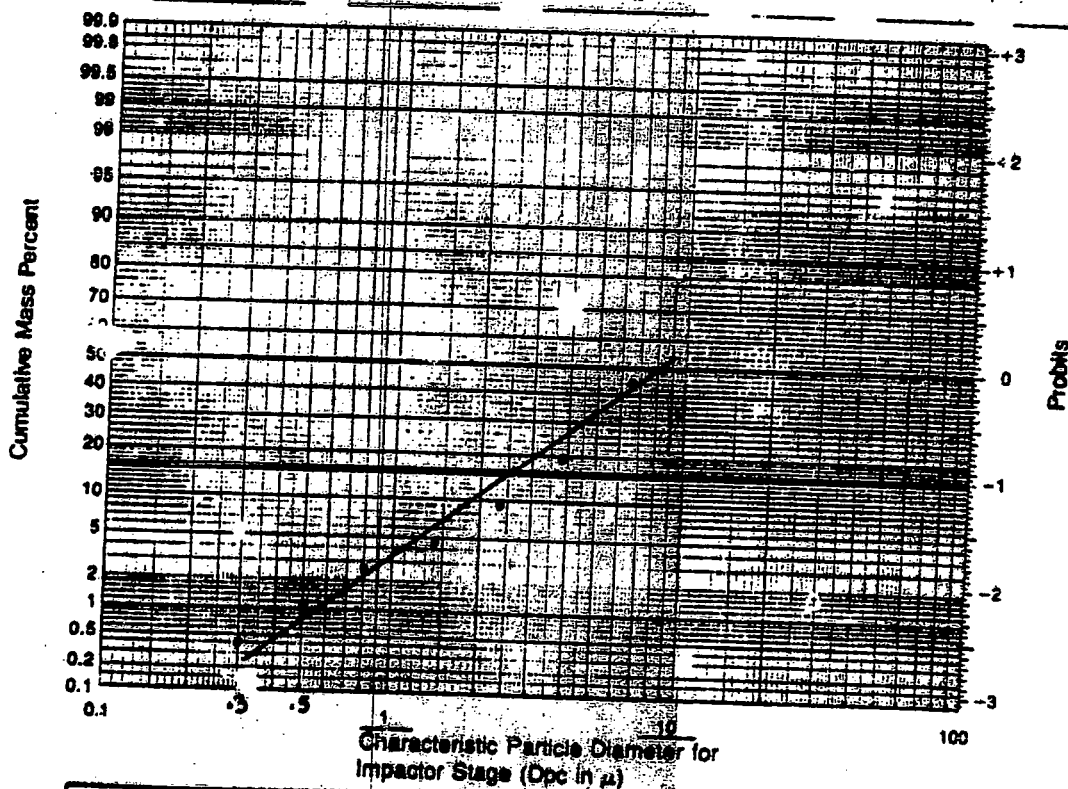
<u>Sample Number</u>	<u>Time (min.)</u>	<u>Concentration (mg/m³)</u>
H-83-38-024	366	0.9
H-83-38-025	367	0.9
H-83-38-027	Blank	Non-detectable
H-83-38-028	175	0.8
H-83-38-029	175	1.0

TABLE 6

SIERRA IMPACTOR SAMPLING - UENTROP WORK

PARTICLE SIZE DETERMINATION

N.B.	P.	MR	H. No.	H-83-38	Date	10/26/83
Sample No.	-026	Sample Time	1248	(min.)	Sample Vol.	4243 (Liters)
Stage	DIAMETER	Pre Wt. (mg)	Wt. Gain (mg)	%	Cum. %	Dpc
Precep.						
1			3.0	X		
2			6.1	X	99.9	
3	6.4 μ m		14.1	54.8	45.1	
4	3.8 μ m		6.6	25.7	19.4	
5	2.4 μ m		2.5	9.7	9.7	
6	1.4 μ m		1.3	5.0	4.7	
7	.8 μ m		.5	1.9	2.8	
8	.5 μ m		.4	1.6	1.2	
9	.3 μ m		.2	.8	.4	
10	.05 μ m		.1	.4	0	



No Writing Underneath

NB.

Pg.

T.I.

8-22734

Mass median diameter = 8 μ m
 G.S.D. = 3

HL 29-84

Company Sanitized. Does not contain TSCA CB1

TABLE 7
RESPIRABLE PARTICLES IN SIERRA IMPACTOR

<u>Impactor Stage</u>	<u>Particle Diameter</u>	<u>Mass, mg</u>	<u>% Comparable to BDX-99*</u>	<u>Net Respirable, mg</u>
1	--	3.0	0	0.0
2	--	6.1	0	0.0
3	6.4	14.1	25	3.5
4	3.8	6.6	50	3.3
5	2.4	2.5	75	1.9
6	1.4	1.3	100	1.3
7	0.8	0.5	100	0.5
8	0.5	0.4	100	0.4
9	0.3	0.2	100	0.2
10		0.1	100	0.1
Total				11.2 mg

Total Particulates = 101 mg (from separate total dust samplers)
 % Respirable = $11.2/101 \times 100 = 11\%$

*The cyclone pre-selection for the Sierra impactor passes larger particles than does the BDX-99 whose selection characteristics are shown in the table on page 3. The particles which pass through the BDX-99 are considered to be the respirable fraction. This column estimates the percentage of particulates on each stage that would have passed the BDX-99 and thus would have been respirable. The total particulates are calculated as an average of the two total particulate samples.

TABLE 8
FLUORINE ANALYSIS OF PARTICULATE
UENTROP WERE

	A	B	C	D
<u>Sample Number</u>	<u>Particulates</u> <u>Weight, mg</u>	<u>Fluorine</u> <u>Weight, mg</u>	<u>Weight, mg</u> <u>(B ÷ 0.4)</u>	<u>Percentage</u> <u>of Total</u> <u>(C ÷ A)</u>
H-83-38-022	.83	.22	.55	66%
Respirable				
H-83-38-023	15.22	2.70	6.75	44%
Total				
H-83-38-030	8.61	1.51	3.78	44%
Total				
H-83-38-031	.29	.07	.18	62%
Respirable				
E-65		.0016		
Blank				
C56		.0028		
Blank				

The respirable particulates are 62-66% [REDACTED] while the total particulates are 44% [REDACTED]

Fluorine analyses by Dr. Eric Kissa, Jackson Laboratory, 11/15/83,
[REDACTED]

TABLE 9
FLUORINE ANALYSIS OF SIERRA IMPACTOR FRACTIONS
UENTROP WERK

Sample Number	A	B	C	D
H-83-38-026	Particulates	Fluorine (1)	Weight, mg	Percentage
	Weight, mg	Weight, mg	(B ÷ 0.4)	of Total
				(C ÷ A)
Stage 1	3.2	.449	1.123	35
Stage 2	6.1	.999	2.498	41
Stage 3	14.1	1.479	3.698	26
Stage 4	6.6	.919	2.298	35
Stage 5	2.5	.404	1.010	40
Stage 6	1.3	.111	.275	21
Stage 7	0.5	.110	See footnote 2.	See footnote 2.
Stage 8	0.4	.108	See footnote 2.	See footnote 2.
Stage 9	0.2	.170	See footnote 2.	See footnote 2.
Stage 10	0.1	.075	See footnote 2.	See footnote 2.
Blank		.219		
Blank		.145		
Blank		.149		
		$\bar{X} = .171 \text{ mg}$		

¹The average blank value of 0.171 mg was subtracted from the fluorine wt. of the samples.

²Weight of fluorine in blanks w. s too large (0.171 mg) compared to total particulate weight to make an accurate calculation of $\frac{C}{A}$