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MATERIAL - ADHESIVE - HEXCEL HP422-VOLATILE BY PRODUCTS - DETERMINATION OF

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MATERIAL - ADHESIVE - HEXCEL HP422 - VOLATILE BY PRODUCTS - DETERMINATION OF

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The tests described in this report were conducted between 22 June 1956 and 2 August 1957

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MATERIAL - ADHESIVE - HEXCEL HP422

VOLATILE BY PRODUCTS - DETERMINATION OF

PURPOSE

During the bonding cycle in which Hexcel HP422 adhesive is used, certain volatile or gaseous products are given off by the adhesive. These gases are caused by chemical reactions within the adhesive during the curing process and result in a bubbling or frothing of the adhesive while it is in the liquid stage. Also, in some cases, there may be a build-up of pressure within the panel due to these gases resulting in poor bonding. Therefore, this test is requested for the purpose of determining at what time and temperature venting of a bonded panel is necessary to release the volatile by-products.

SUMMARY

Samples from seven different rolls of tape were tested. The average total recovered amount of volatile material given off by 8 square inches of tape (having an average weight of 4.6690 grams) was 0.2274 grams or about 4 grams per square foot. This indicates that approximately 5% of the total weight of HP422 tape is given off as volatile material during the bonding process. Analysis of this volatile material showed 1.3% ammonia and 6.8% phenol. The remaining 91.9% was water given off as steam with a trace of formaldehyde.

The percent of the volatile material coming over during the first 15 minutes while heating up to 230°F ranged from 52.5% to 84.0% of the total amount obtained. During the next 30 minutes while the temperature rose from 230°F to 250°F the percent of volatiles coming over ranged from 13.2% to 43.8%. Volatiles coming over from 250°F to 350°F ranged from 2.7% to 6.5% of the total amount obtained.

OBJECT:

To determine the volatile by-products of Hexcel HP422 adhesive given off during bonding as a function of the time and temperature, and to determine the possible relationship between the volatile by-products and the flow value of Hexcel HP422 adhesive.

MATERIALS AND APPARATUS:

A: Materials

10 sq. yds, Hexcel HP422 adhesive	Hexcel Products Oakland, California
10 liters acetone	Convair Stock Technical grade
1 cylinder dry helium	U. S. Bureau of Mines Amarillo, Texas
25 lbs. dry ice	Liquid Carbonics Co. Fort Worth, Texas

B. Apparatus

Distillation apparatus	Engineering Chemistry Laboratory Stock
Variable heating bath	" "
Standard analytical equipment	" "
Bonding presses	Convair shop made

PROCEDURE:

A weighed amount of the adhesive was given the required time-temperature cure cycle in a distillation type apparatus surrounded by a controlled heating bath of Cerrobend metal as shown in Figure 1. During the heating cycle the system was flushed with a stream of dry helium to carry off the volatiles. These were caught in a cold trap composed of Blout tubes in a bath of acetone and dry ice. The heating cycle was divided into three steps as follows:

1. From room temperature to 230°F in 15 minutes.
2. From 230°F to 250°F in 30 minutes.
3. From 250°F to 350°F in 15 minutes.

The heating bath was controlled so that the tape sample

followed this heating cycle. The volatile material from each step was collected in a separate Blout tube. After the run, each tube was weighed and the weight of volatile material coming over during each step was determined. The total amount of volatiles during a run was determined by adding the individual amounts collected for each step. From this total and from the original weight of the tape sample, the percent of the original weight of the tape given off as volatile material was calculated. One sample was selected for composition analysis in order to determine the compounds present. The ammonia was determined by the Kjeldahl method. The phenol was determined by treating an aqueous solution of the recovered volatiles with bromine water which precipitated the tribrominated phenol compound. The precipitate was centrifuged, washed and weighed. The quantity of phenol present in the original solution was calculated.

The flow values were determined according to the following procedure:

A 4" x 4" sample is cut from a smooth, non-wrinkled section of the tape and weighed. The weight of the polyethylene cover is subtracted to obtain the actual weight of the adhesive and carrier cloth. After removal of the polyethylene covers, the adhesive is placed between two sheets of aluminum foil and cured between parallel aluminum plates in a heated press at 275°F and 25 psi pressure for 10 minutes. The sample is then removed from the press, cooled, and the total area covered by the adhesive flow is measured with a planimeter. The flow is calculated as follows:

$$Ft = \frac{At}{Wa - 0.73}$$

where Ft = Total flow

At = Total area covered by the adhesive and the flow out

Wa = Weight of the adhesive and cloth carrier

0.73 = Weight in grams of the cloth carrier

RESULTS

The total amount and percent of volatile material from various tape samples are listed in Table I. Table II shows what part of the total amount came over at three different time-temperature points in the cure cycle along with the percent of the total amount the particular part represents. The composition of the sample which was analyzed is given in Table III, and the flow values are listed in Table IV. A summary giving the maximum, minimum and average amounts of volatile material with regard to time and temperature are shown in Figure 2.

DISCUSSION

The calculation of the percent volatile material given off by the adhesive was based on the weight of the volatile material actually recovered. Consequently, this value might be slightly low since it was impossible to recover all of the volatile material given off. However, the weight of the material recovered was compared with the weight loss of the tape and approximately 90% recovery was found.

The amounts of phenol and ammonia were determined by standard analytical methods as outlined in the procedure. The remaining portion of the volatile material was assumed to be water based on the appearance of the condensate and the fact that the majority of the material distilled below 230°F. Although the odor of formaldehyde could definitely be detected, specific tests (semicarbazon test, 2-4 dinitrophenylhydrozone test, sodium bisulfite test) gave negative results. The odor of phenol and formaldehyde could be detected in the material coming over at the lower temperatures. The volatiles coming over after 350°F gave off the odor of ammonia.

It had been suggested that a sample of adhesive be heated from room temperature to 350°F in 15 minutes and the volatiles collected and analyzed. This could not be done with the apparatus used since a sufficiently rapid rate of heating could not be obtained.

There was no correlation between flow values and percent volatile material, although there was a considerable spread between the flow values.

CONCLUSION

The volatile products given off by HP422 adhesive during bonding were determined as a function of time and temperature. The data is presented in Tables I and II. In addition, the composition of the volatiles products was determined and found to be 91.9% water, 6.8% phenol, 1.3% ammonia and a trace of formaldehyde. The flow values for the adhesive, also determined and presented in Table IV, have no relationship to the volatile material given off during the heating cycle.

TABLE I

Weight and Percent of Volatile Material Obtained From Samples of Hexcel HP422 Adhesive Heated to 350°F Over a Period of 1 Hour.

TAPE ROLL NUMBER	GRAMS WEIGHT OF TAPE SAMPLE*	GRAMS WEIGHT OF VOLATILE MATERIAL OBTAINED	% OF TAPE WHICH IS VOLATILE MATERIAL
787	4.5847	.1767	3.85
272	4.7140	.2507	5.32
853	4.7966	.2467	5.14
515	4.4933	.2120	4.72
284	5.0117	.2667	5.32
71	4.4138	.2140	4.85

* Each sample represents 8 square inches of tape.

The weight listed includes that of the glass cloth carrier.

TABLE II

Weight and Percent of Volatile Material Obtained at Different Times During the Heat-up Cycle

TAPE ROLL NUMBER		15 MINUTES ROOM TEMP. 230°F	30 MINUTES 230°F 250°F	15 MINUTES 250° F 350°F
787	Weight obtained	0.1483 gm	0.0231 gm	0.0053 gm
	Percent of total	84.0	13.0	3.0
272	Weight obtained	0.1735 gm	0.0619 gm	0.0103 gm
	Percent of total	71.3	24.6	4.1
853	Weight obtained	0.1686 gm	0.0697 gm	0.0084 gm
	Percent of total	68.3	28.2	3.4
515	Weight obtained	0.1120 gm	0.0930 gm	0.0070 gm
	Percent of total	52.8	43.8	3.3
284	Weight obtained	0.1800 gm	0.0773 gm	0.0094 gm
	Percent of total	67.5	27.4	3.5
71	Weight obtained	0.1720 gm	0.0338 gm	0.0082 gm
	Percent of total	80.4	15.8	3.7

TABLE III

Composition of Volatile Materials Obtained From Samples of Hexcel HP422 Adhesive Heated to 350°F Over a Period of 1 Hour*

ROLL NUMBER ANALYZED	PERCENT PHENOL	PERCENT AMMONIA
515	6.8	1.3

* The remaining 91.9% of the volatile material is most likely a mixture of water and formaldehyde, as the greater portion comes over at temperatures below 230°F and the odor of formaldehyde is apparent.

TABLE IV

FLOW VALUES FOR VARIOUS SAMPLES OF

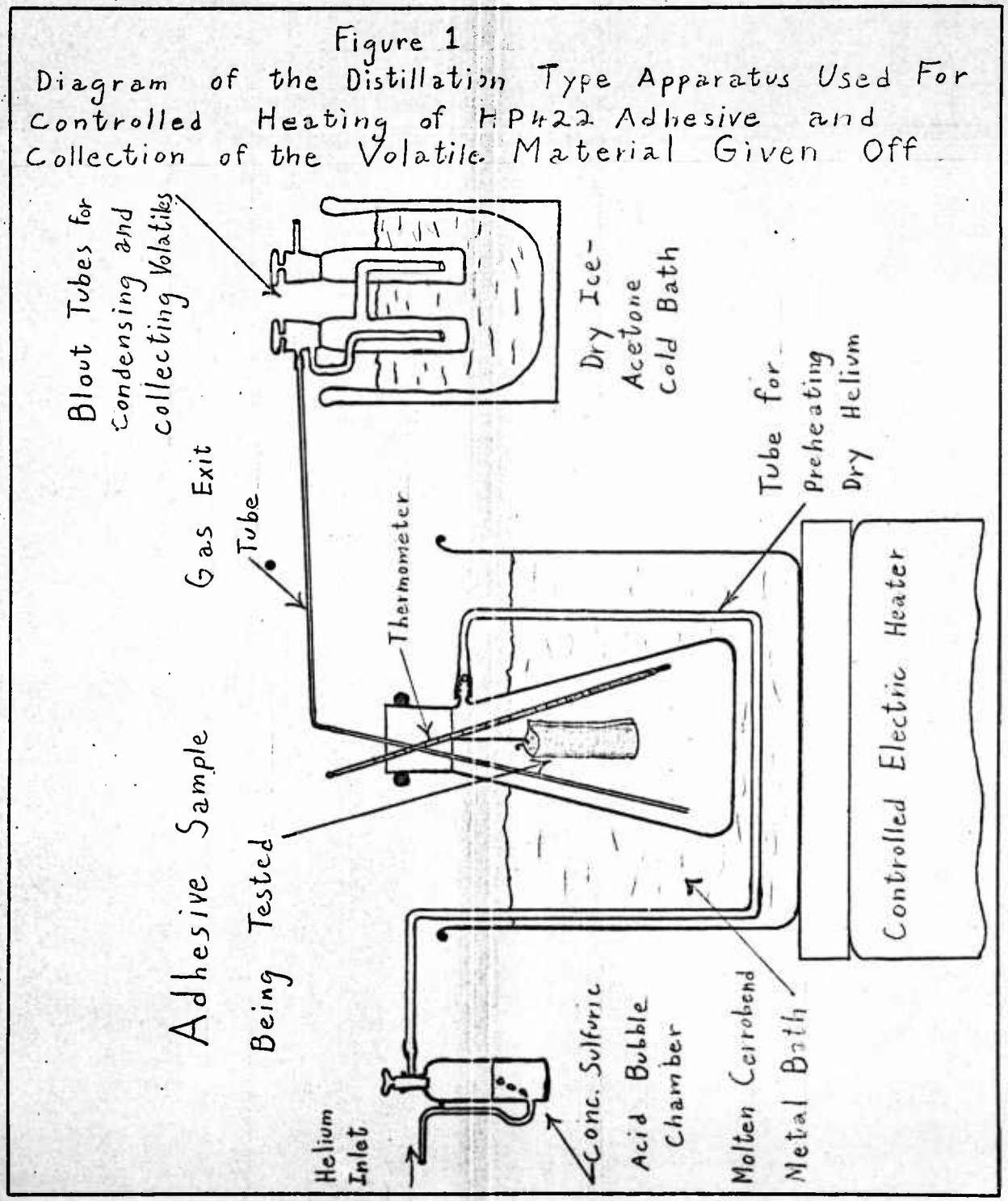
HEXCEL HP422 ADHESIVE

TAPE ROLL NUMBER	FLOW VALUE
787	3.9
272	6.4
853	4.0
515	4.1
284	6.5
71	2.7

Determined by method outlined under procedure.

Figure 1

Diagram of the Distillation Type Apparatus Used For Controlled Heating of HP422 Adhesive and Collection of the Volatile Material Given Off



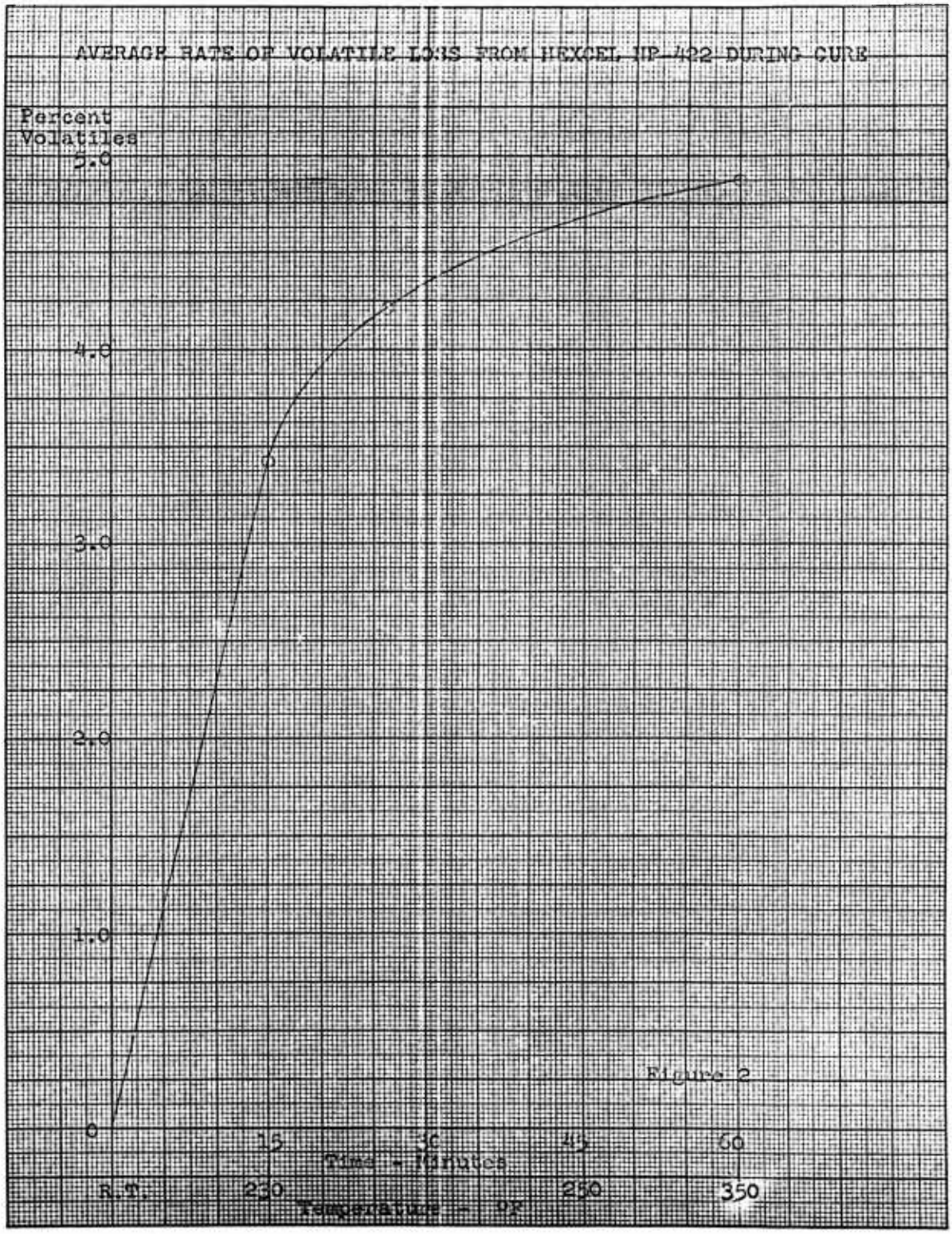


Figure 2

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