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1. Contractor's Name and Address:

Springborn Laboratories, Inc.  
Department of Analytical Chemistry  
Ten Springborn Center  
Enfield, CT 06082

2. Title of Report:

Development of Acceptance Criteria for Batches of Silane  
Primer for External Tank Thermal Protection System  
Bonding Applications

December 1, 1984 - March 1, 1985

3. Date of Publication:

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CRITERIA FOR BATCHES OF SILANE PRIMER FOR  
EXTERNAL TANK THERMAL PROTECTION SYSTEM  
BONDING APPLICATIONS Progress Report, 1  
Dec. 1984 - 1 Mar. (Springborn Labs., Inc., G3/27 14678

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LIST OF CONTENTS

	<u>Page</u>
1. Table of DC 1200 Primer Lots Used in Project Program	1
2. Thermogravimetric and FTIR Analyses of DC 1200 Silane Primers - Concluding Tests	2
3. HPLC and GC Analysis of DC 1200 Silane Primers	3
4. Determination of Titanium and Silicon By Atomic Absorption Spectroscopy (Method Description)	4
5. Plan For Future Work	5
6. Financial Status	5
Table - Summary of Results Obtained for DC 1200 Silane Primers (Residue, Ash, Titanium, Silicone, Si/Ti Ratio, OH-Absorption, Lap Sheer Test, GC Headspace for Alcohols)	6

Figures - Nos. 1 through 13

Appendix - Pages A1 - A5

NOTE: In our 7-8 Report, please make a correction on page 9,  
5th line from bottom: Samples 7'''-6''', 13''', C''' -  
this should read: Samples 7'''-6''', 13''', C''', etc.

TABLE I\*

DC 1200 Primer Lots Used in Project Program

<u>Springborn Labs ID #</u>	<u>DC Primer Lot #</u>	<u>Date Received at Springborn</u>	<u>Additional Description and Further Sample Labeling</u>
A**	QL033703	5/11/83	red opaque
B**	QL071621	11/29/83	red, leaked
C**	QL093752	11/29/83	clear, sealed
1***	EN057367	3/30/84	clear
2***	QL033705	3/30/84	red
3***	063711	3/30/84	red, S/L Exp 6/84, Temp 50/90F RS3.900324, 7/83 MMSK343A025 83G382
4***	093713	3/30/84	red, S/L Exp. 09/84, Temp 50/90F, RS3.794481 09/83 MMSK343A025 83G530 (hold for J Mills)
5***	093733	3/30/84	red, S/L Exp 09/84, Temp 50/90F, RS3.794481, 09/83 MMSK343A025 83G529
6***	071620	3/30/84	red, S/L Exp 7/82, Temp 50 to 90F, RS1:705481, 08/81 MMSK343A025 81G464
7 <sup>+</sup>	QL103753	1/10/85	red

\*A sample identification table is included as the first page in each report.

\*\*Three (3) DC 1200 Primer Lots acquired by Springborn Laboratories for initial tests (see monthly reports 1, 2 and 3).

\*\*\*Six (6) DC 1200 Primer Lots received from NASA, Management Division Bldg. 4471, on March 30, 1984.

<sup>+</sup> DC 1200 Primer Lot received from NASA, Bldg. 4612, Mr. Don Morris, Project Monitor.

## 2. TGA and FTIR Analysis of DC 1200 Silane Primers - Concluding Tests

In order to compare TGA thermograms of all submitted silanes, additional TGA profiles of samples #B and #C were obtained (Fig. 1). Sample #C was also reexamined -- its TGA profile is compared to the one obtained earlier (10/8/84) in Fig. 2. (FTIR analysis of this sample did not indicate any significant increase in OH-absorption since the last TGA analysis.)

A new DC 1200 primer sample was obtained from NASA-MSFC on 1/6/85 (our ID #7). Its TGA and FTIR profiles were recorded and are enclosed as Figs. 3 (FTIR) and 4 (TGA).

New FTIR spectra of samples B and C were also obtained as shown in Figs. 5 (B) and 7 (C). A visual comparison of the three IR spectra (samples B, C, 7) indicate that sample #7 (new silane lot) does not contain any OH-absorption and thus has not undergone any hydrolysis. On the other hand, substantial OH-absorption is observed in primer sample #B (more than in the last FTIR scanning on 7/11/84). The largest OH-absorption is in sample #C as expected (see previous 7-8th report, 12/7/84).

TGA profiles of all silane primers received from NASA-MSFC to date are graphically presented in Fig. 7. Samples exhibiting similar thermograms are indicated in the table below. This table replaces a similar one given in the 7-8th report on page 5.

<u>Similarity Group</u>	<u>TGA* Profiles of DC 1200 Primer Lots #</u>	<u>See Fig. No.</u>
I	1, 2, 3, 5, ---(4, 6)	7
II	7	4
III	B, C	1, 2

\*TGA conditions are given in each TGA profile.

From these final tests, it can be concluded that the TGA method lacks the selectivity required for use as a criterion for silane primer batch evaluation.

### 3. HPLC and GC Analysis of DC 1200 Silane Primers

#### HPLC Method:

A series of DC 1200 silane primers were analyzed by HPLC using Sep Pak (silica 10  $\mu$ , 10 cm x 4.6 mm i.d.) cartridges with hexane chloroform (1:1) as mobile phase and refractive index detector. As the profiles in Fig. 8 (isopropanol\* and proponal standards) and Fig. 9 (DC 1200 primer samples) indicate, no conclusive pattern could be found correlating the FTIR increase in OH-absorption and an increase in the first HPLC peak of each silane sample.

#### GC Method:

Although previous GC studies had not proved conclusive, it was believed that there should be a GC method which could quickly determine the alcohol content of different DC 1200 silane batches. Analyses with a new GC method have been initiated, where only the headspace of the individual primer samples will be analyzed for alcohols. Initial correlations between samples with higher IR absorbance in the OH- region and alcohols content (by GC) seems to be good enough that all previously analyzed silanes will be examined by this method and reported in the 11th report.

Preliminary results for samples 6, 7 and C are given in the tables below. Their chromatographic profiles are shown in Figs. 10 to 13. It can be seen that the most degraded sample C (Fig. 11) contains the largest quantity of hydrolyzed products (some of which have to be further determined by GC/MS).<sup>t</sup> A GC profile of sample #6 in Fig. 12 indicates only a moderate release of an "alcoholic fraction" due to hydrolysis. The newest sample #7, recently received from NASA shows only a minimal presence of OH bonds by FTIR. This finding correlates with the GC profile of the polar fraction, where almost no decomposition products have been detected (Fig. 13). Fig. 10 gives a GC profile of a blank.

A preliminary calculation of total isopropanol and n-butanol content\*\* are indicated in the following table:

<u>% Isopropanol + n-butanol</u>	<u>Sample ID #</u>
3.2%	6
3.6%	7
9.9%	C

\*To be included in 11th report.

\*\*Detection based on retention times of standards only.

If some unidentified GC peaks are considered to be silane hydrolytic products, the percent decomposition in the silanes might be:

5.0% in sample #7

11.7% in sample #6

25.2% in sample #C

The GC conditions used were as follows: GC glass column (6' x 2mm i.d.) filled with 5% Carbowax 20M on Carbopack B, 100/120 mesh; temperature program started at 70°C, heated at 5°/min. to 170°C; helium as a carrier gas, flow rate 27 ml/min. Only 5-10  $\mu$ l of head-space was used for GC analysis. Further analyses are in progress.

4. Determination of Titanium and Silicon By Atomic Absorption Spectrometry (Method Description)

Six DC 1200 silane coupling agents received from NASA (our ID # 1-6) and two samples previously obtained (B and C) were first analyzed for their residue content. Ten ml of each sample was pipetted (in duplicate) into platinum crucibles, placed in an air-forced oven for 3 hours at 105°C, and then weighed for residue content. The platinum crucibles with residues were then placed in a muffle furnace overnight at 600°C. After cooling in a desiccator, the samples were weighed for ash content.

To each sample ash was added approximately 20X by weight of LiBO<sub>2</sub>. The powders were well mixed and then placed in a muffle furnace at 900°C for 15-30 minutes. Mixtures were swirled several times to insure complete fluxing. After removing the Pt crucibles from the muffle furnace, they were quenched in a beaker of cold water. The flux material was then dissolved in the presence of distilled water and HNO<sub>3</sub> (with heating), 5 drops of HF (HF was added into the cooled solution to prevent loss of Si as SiF<sub>6</sub>) and 20 drops of H<sub>2</sub>O<sub>2</sub>. The contents were then transferred to a 1000 ml volumetric flask. A completely clear yellow solution resulted. (The HNO<sub>3</sub> concentration of the final solutions were approximately 5% HNO<sub>3</sub>).

A blank with the same concentration of reagents was also prepared.

Instrumental analyses were conducted with an atomic absorption spectrometer (Model 450, Perkin Elmer). Silicon was measured at 251.7 nm; 0.2 nm slit width; N<sub>2</sub>O/acetylene flame (linearity to 150 ppm). Titanium was measured at 365.6 nm, 0.2 nm slit width, N<sub>2</sub>O/acetylene flame (linearity to 200 ppm).

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A protocol with the results obtained for Si and Ti contents in six DC 1200 silane samples is enclosed in the Appendix (pgs. A-1 to A-5).

The table on page 6 summarizes the DC 1200 primer results for:

- 1) residue content
- 2) ash analysis
- 3) analyses of titanium, silicon and their ratio (Si/Ti)
- 4) OH<sup>-</sup> absorptions (3360-3470 cm<sup>-1</sup>)
- 5) average PSI values from preliminary lap sheer tests  
(see more in the next report #10)

#### 5. Plan For Future Work

On January 6, 1985 the continuation work outlined in the Amendment No. 1 was initiated. A progress report covering the period 1/6/85 - 3/20/85 will be issued as the 10th report.

As agreed with the project monitor, Mr. Don Morris, the final report will be issued after concluding this amended work (concluding date June 1, 1985).

#### 6. Financial Status

The total cumulative expenditure incurred as of the report date, December 1, 1985, is \$45,627 (i.e., material and labor). The estimated percentage of contract completion (excluding Amendment No. 1) is 100%.

Summary of Results Obtained for DC 1200 Silane Primers

(Residue, Ash, Titanium, Silicone, Si/Ti Ratio,  
OH-Absorption, Lap Sheer Test,  
GC Headspace for Alcohols)

DC Primer Lot #	Residue Content @ 105°C	% Ash (800°C)		Titanium %		Silicone %		Ratio Si/Ti	Abs. Units = Abs. height (mm) x Abs. unit/inch 25 mm/inch				Lap Sheer Test Avg. psi**	Preliminary GC Analysis of Alcohols***
		Dupl.	Avg.	Dupl.	Avg.	Dupl.	Avg.		3360 cm <sup>-1</sup>	3300 cm <sup>-1</sup>	3420 cm <sup>-1</sup>	3470 cm <sup>-1</sup>		
		2.86 2.77	2.82	0.579 0.642	0.61	0.601 0.705	0.65	1.07						
A	4.86	2.75 2.69	2.72	0.604 0.558	0.58	0.747 0.815	0.78	1.34	1.2204	1.1232	0.972	0.776	79.55 (9 pts)	-
B	5.07	3.00 3.10	3.05	0.509 0.567	0.54	0.633 0.608	0.66	1.22	1.7716	1.6168	1.7200	1.3244	51.33 (9 pts)	25.2
C	4.15	2.96 3.07	3.02	0.66 0.64	0.65	0.88 0.91	0.90	1.38	0.4620	0.4620	0.4620	0.4788	*	-
1	3.51	3.14 3.07	3.10	0.62 0.61	0.62	0.89 0.87	0.88	1.42	0.7176	0.6992	0.6532	0.5796	91.0 (9 pts.)	-
2	3.84	3.17 3.09	3.14	0.62 0.62	0.62	0.95 0.91	0.93	1.50	1.1252	1.0440	0.9744	0.7772	84.66 (3 pts)	-
3	3.70	3.14 3.17	3.16	0.61 0.60	0.61	0.96 0.96	0.96	1.57	0.6636	0.6636	0.6636	0.5628	*	-
4	3.35	3.16 3.14	3.16	0.62 0.65	0.64	1.00 1.02	1.01	1.58	0.5688	0.5544	0.5472	0.5328	152.0 (5 pts)	-
5	3.56	3.15 3.14	3.16	0.62 0.61	0.62	0.93 0.93	0.93	1.50	0.3016	0.3068	0.3276	0.3484	*	11.7
6	3.78	3.15 3.17	3.16	0.62 0.61	0.62	0.93 0.93	0.93	1.50	0.0288	0.0144	0.0048	0.0144	177.33 (6 pts)	51
7	4.48	3.03 3.02	3.03	*	*	*	*	*						

\*Results will be included in report #11.

\*\*Preliminary results, see in detail report #10.

\*\*\*Total of alcohols found in headspace.

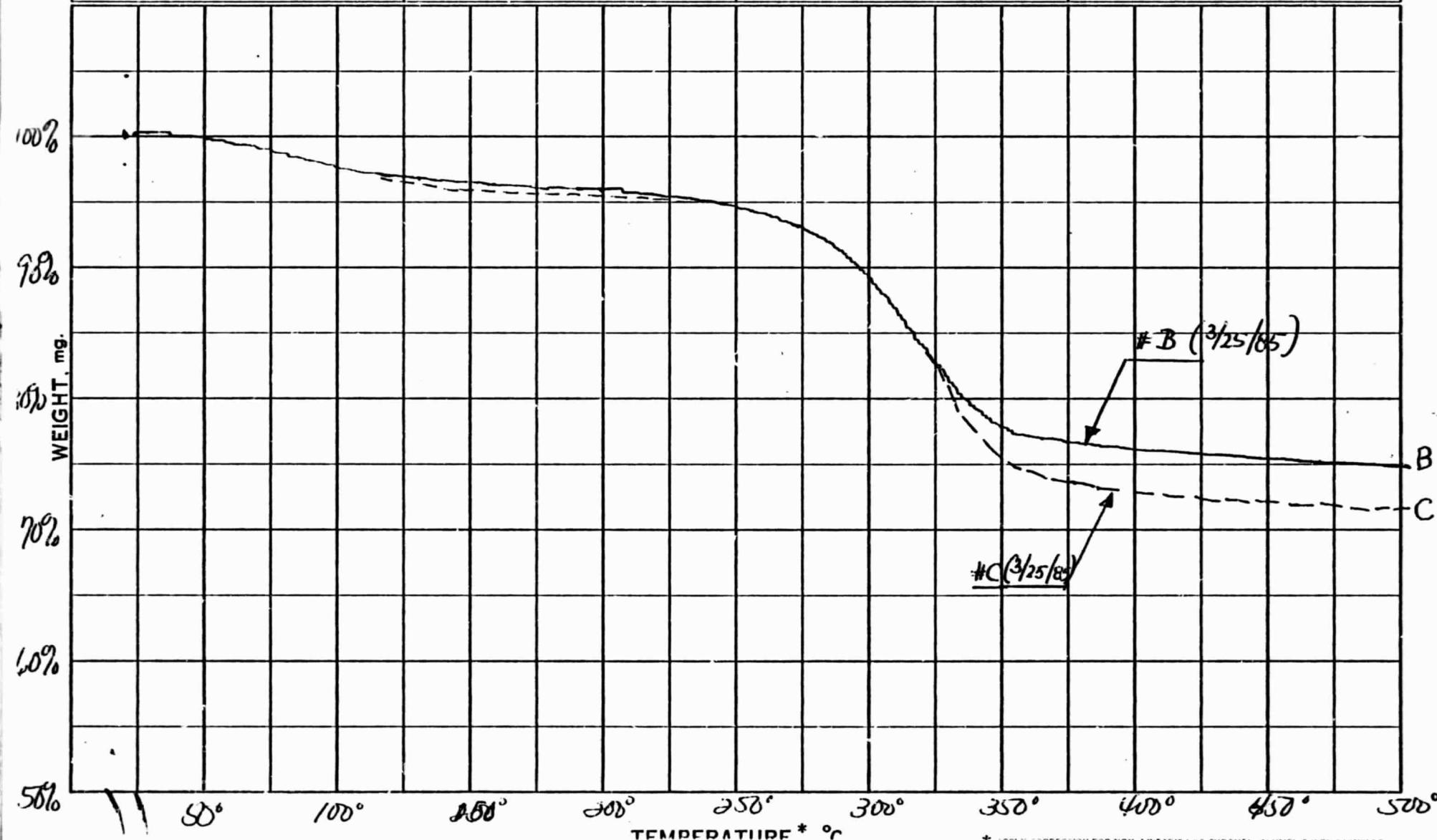
Nasa 6031.2



INSTRUMENTS

FIG.1

SAMPLE:	X-AXIS	Y-AXIS	RUN NO.
Sample B	TEMP. SCALE 50 °C SHIFT _____ inch	SCALE mg. inch (SCALE SETTING X 2)	2180 DATE 3-25-85
SIZE 21.8 mg.	TIME SCALE (ALT.)	SUPPRESSION 10.8 mg.	OPERATOR L. A. Mowrey
			HEATING RATE 20 °C min.
			ATM. N <sub>2</sub>
			TIME CONSTANT 2 sec.



Nasa 6031.2

SAMPLE: Sample C  SIZE $22 \pm 6$ mg.	X-AXIS TEMP. SCALE $50$ °C SHIFT _____ inch TIME SCALE (ALT.)	Y-AXIS SCALE _____ mg. inch (SCALE SETTING X 2) SUPPRESSION $12.6$ mg.	RUN NO. 2181 DATE 3-25-85 OPERATOR C. A. Monroe HEATING RATE $20$ °C min. ATM. $N_2$ TIME CONSTANT $\frac{1}{2}$ sec.
--	--	--	--

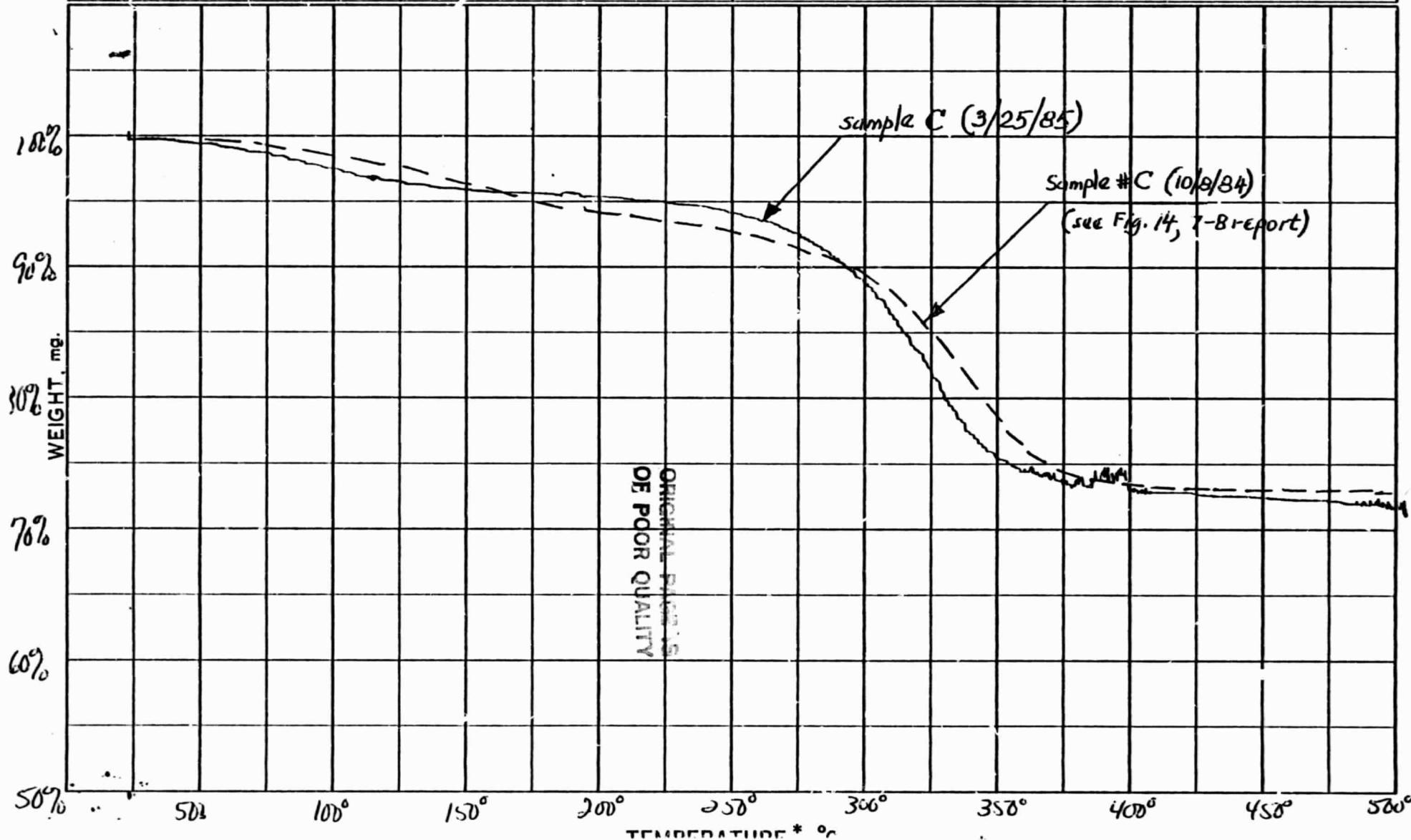
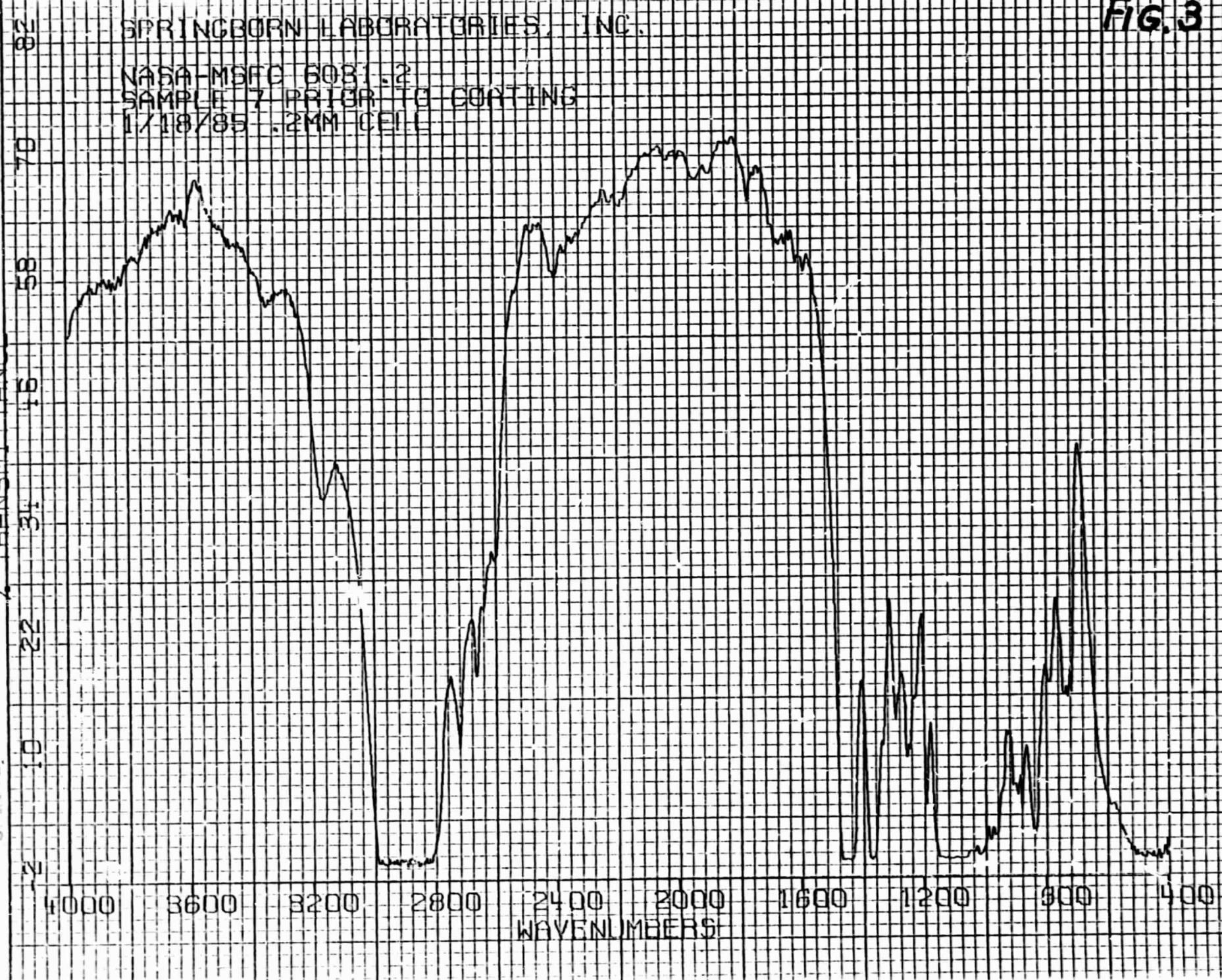


FIG. 3

SPRINGBORN LABORATORIES INC.

NASA-MSFC 6031.2  
SAMPLE 7 PRIOR TO COATING  
1/18/85 .2MM C.E.I.

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100-1000

Nasa 4031.2



INSTRUMENTS

FIG. 4

SAMPLE: Sample 7 SIZE 20.2 mg.	X-AXIS TEMP. SCALE 50 °C SHIFT _____ inch TIME SCALE (ALT.)	Y-AXIS SCALE 4 mg. (SCALE SETTING X 2) SUPPRESSION 10.2 mg.	RUN NO. 2182 DATE 3-26-85 OPERATOR C.A. Mowery HEATING RATE 20 °C ATM. 1/2 min. TIME CONSTANT 2 sec.
--------------------------------------	--	--	--

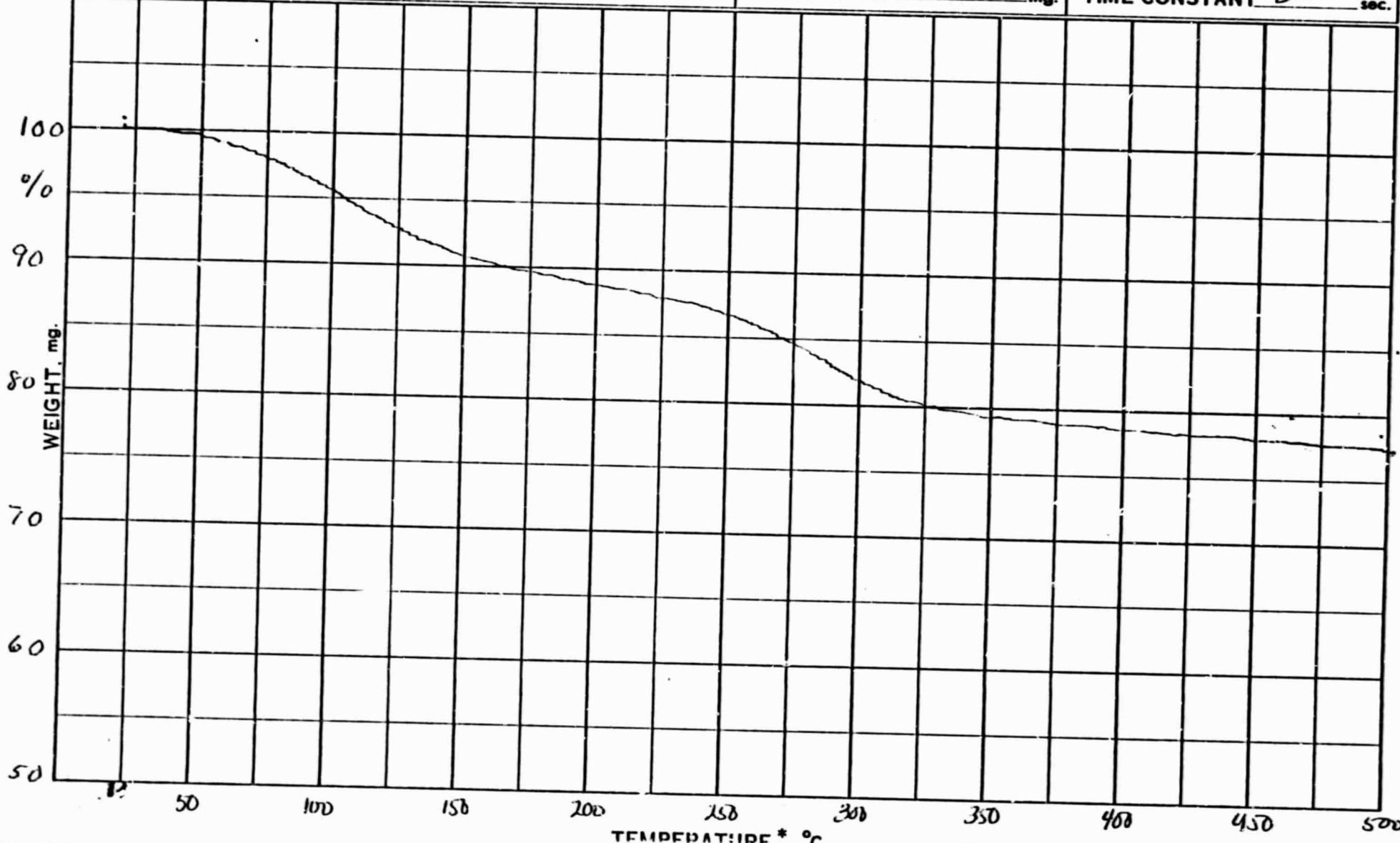
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FIG. 5

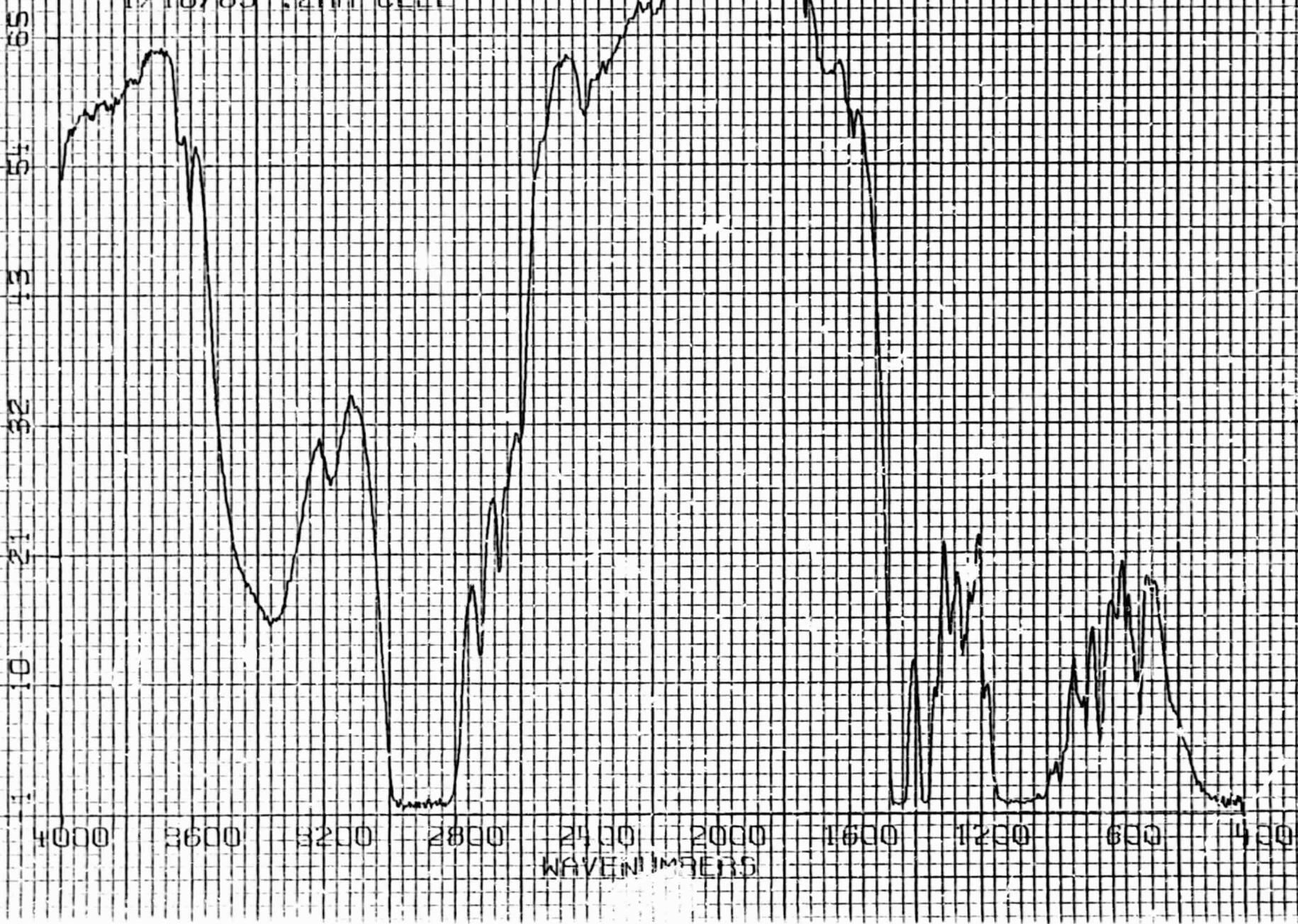
SPRINGBORN LABORATORIES, INC.

NASA MSFC 6081.2

SAMPLE B PRIOR TO COATING

1/18/85, 2MM CELL

% TRANSMITTANCE



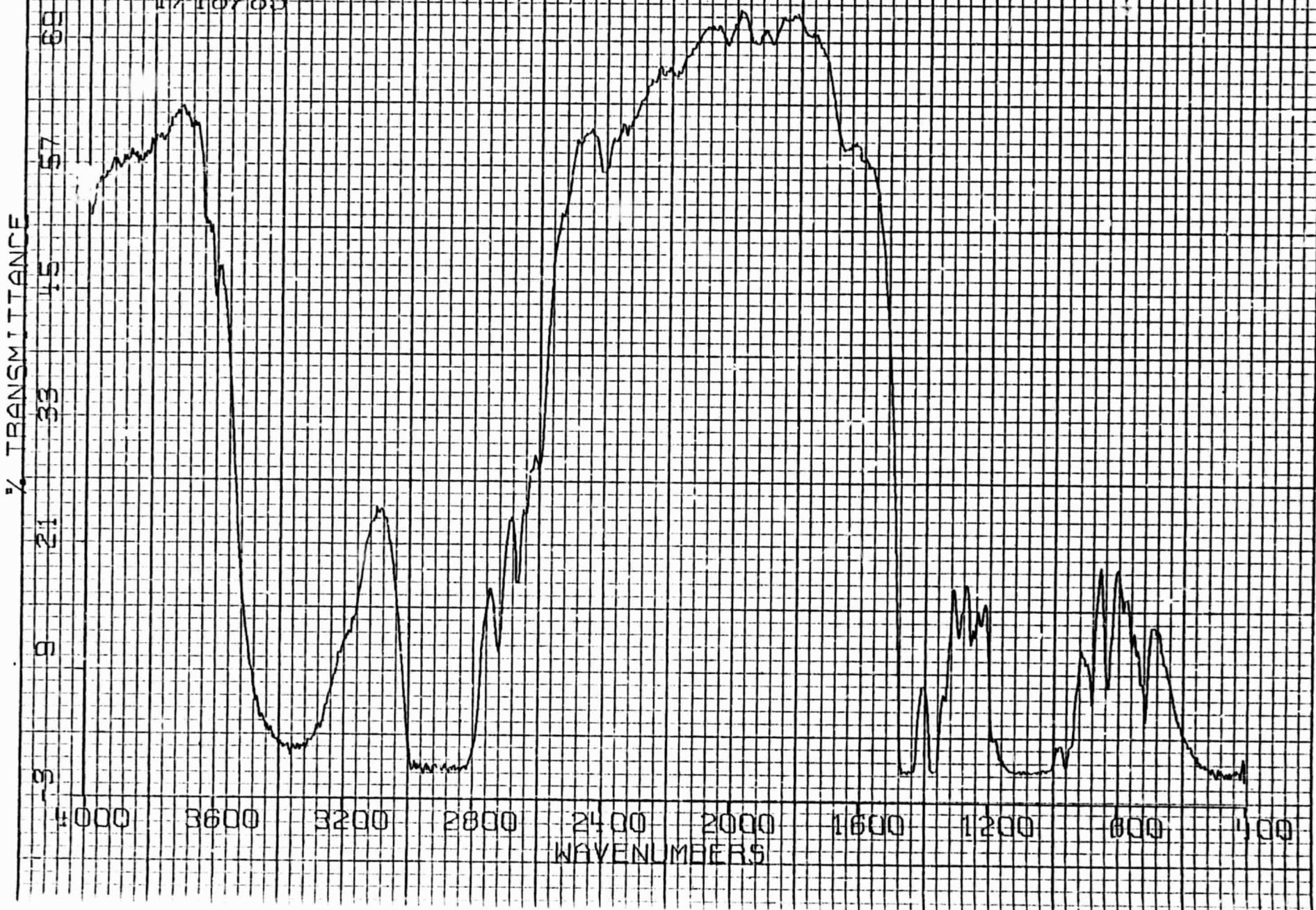
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FIG.6

SPRINGBORN LABORATORIES, INC.

NASA-MSFC 6081-2

SAMPLE C PRIOR TO COATING  
1/18/85



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INSTRUMENTS

FIG. 7

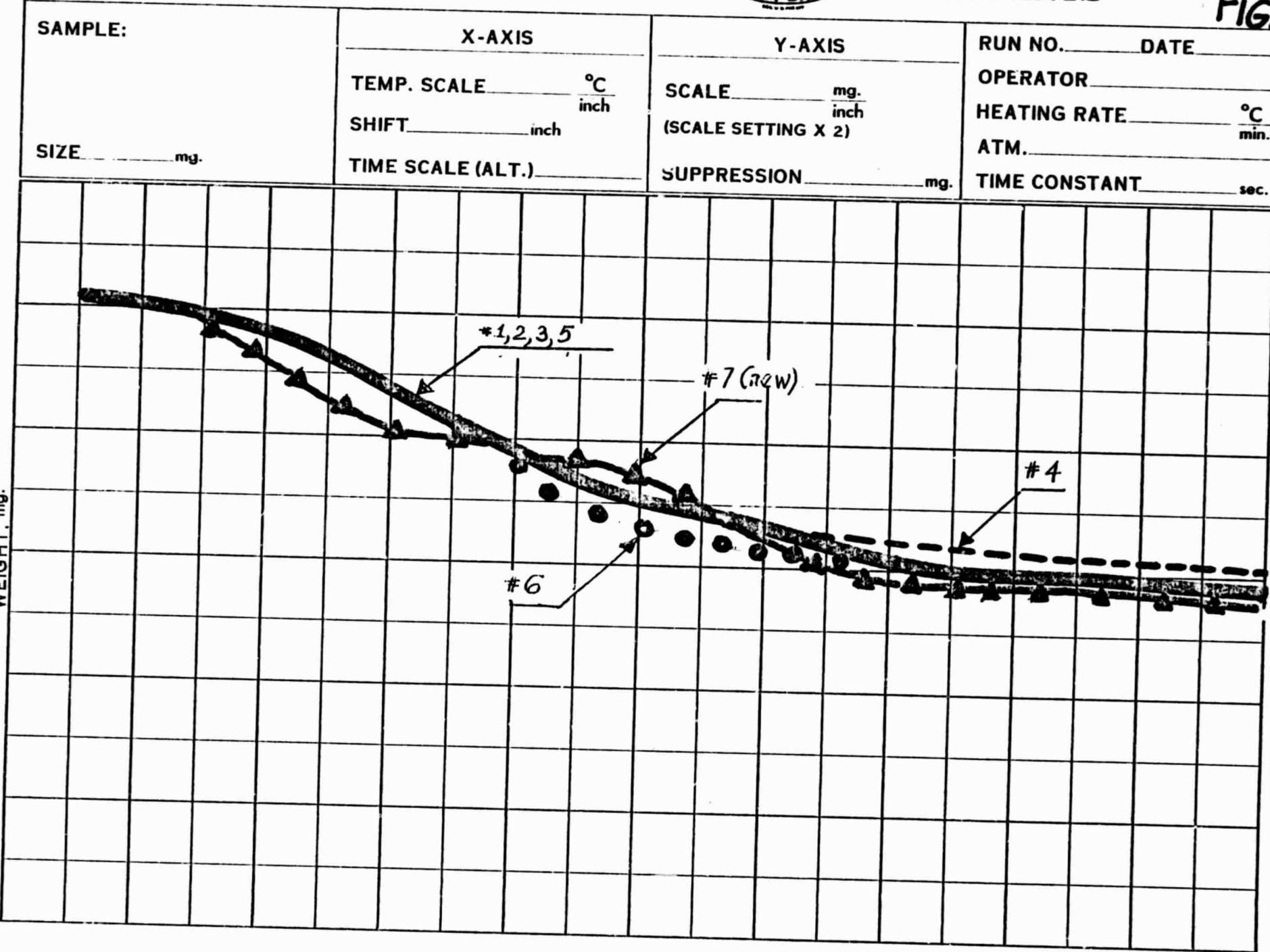
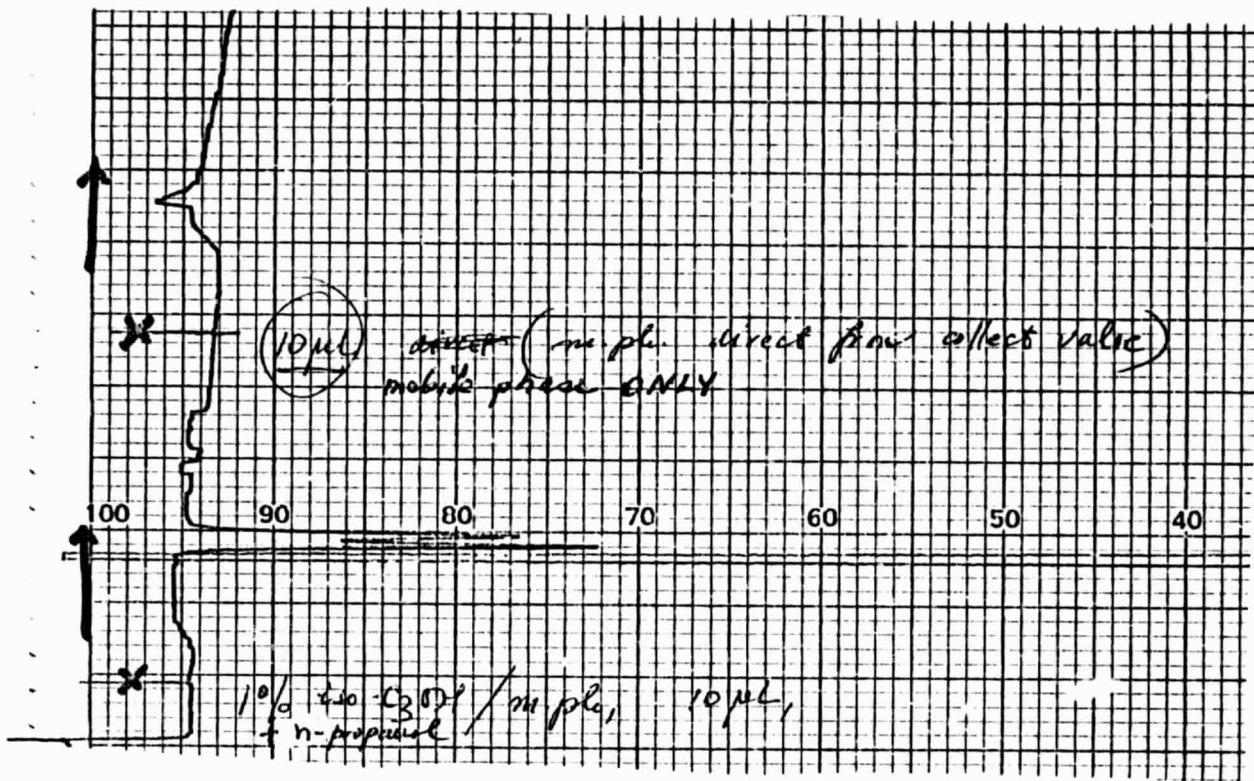
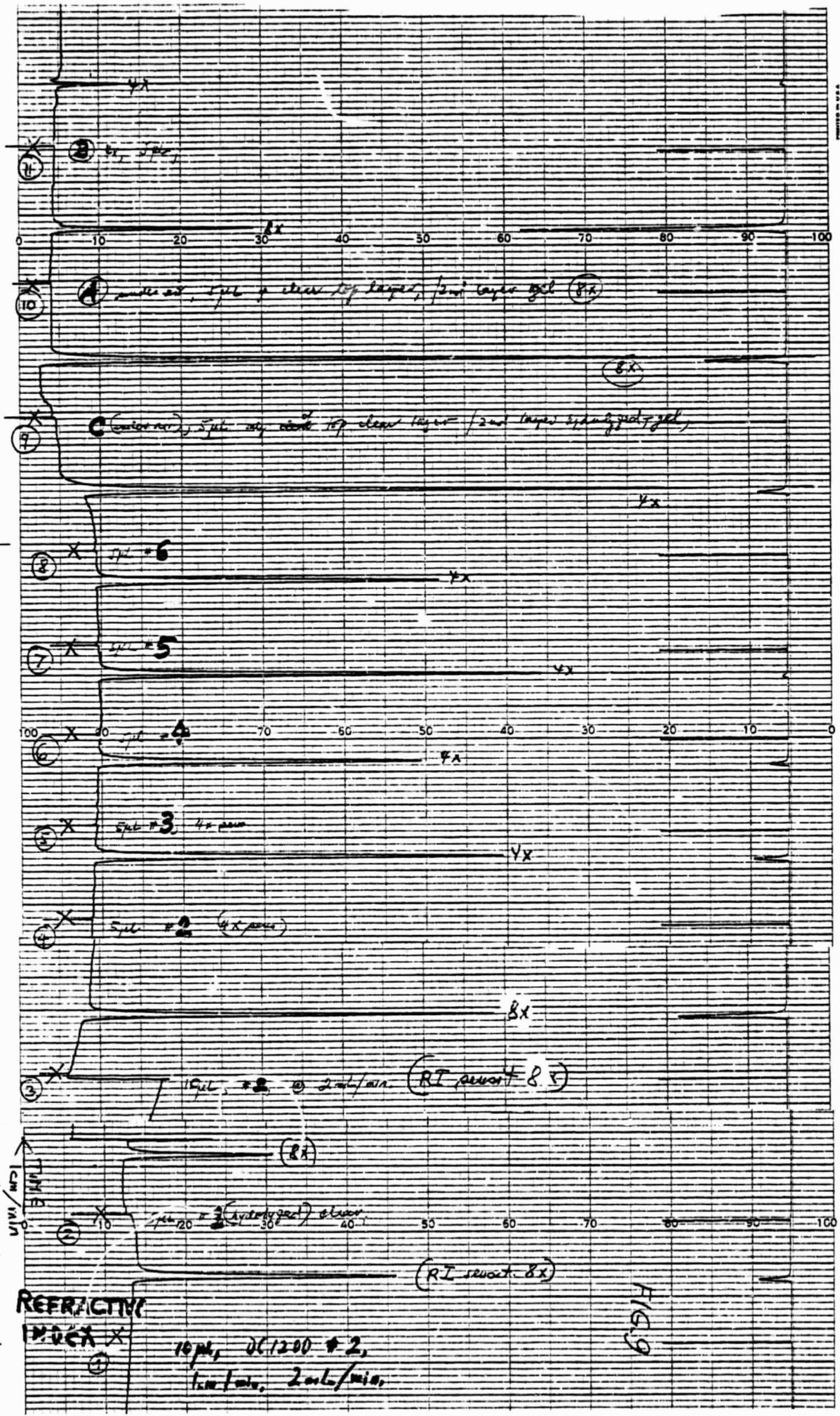


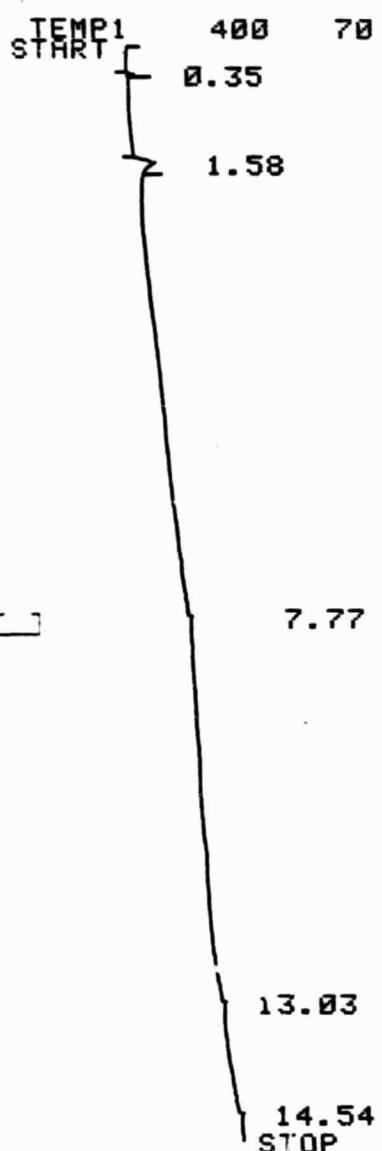
FIG. 8

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## GC of blank



HP RUN # 152      APR/05/85      TIME 15:31:18  
AREA %

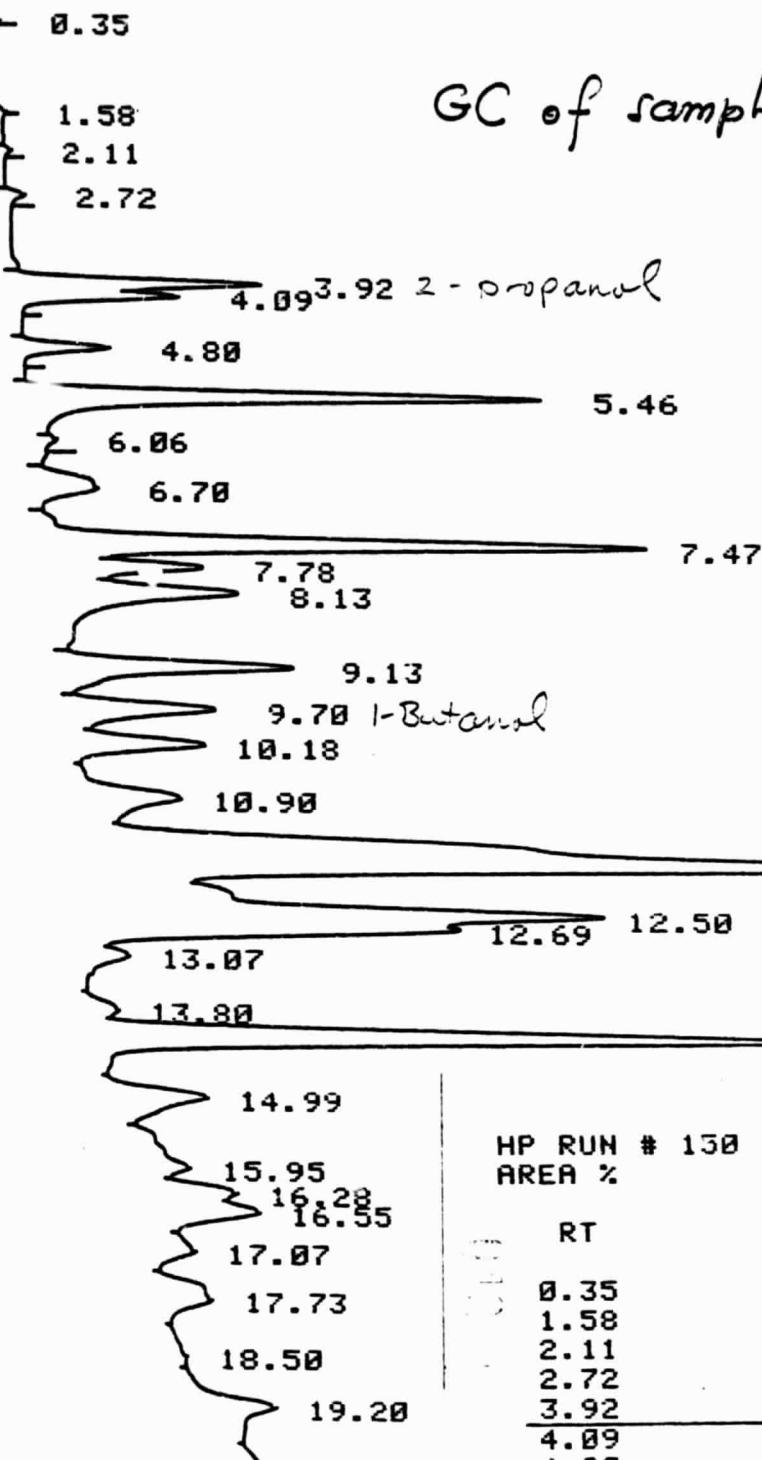
RT	AREA	AREA %
0.35	11	5.556
1.58	187	94.444

DIL FACTOR: 1.0000 E+ 0

TEMP1      400      70      70

FIG. 11

## GC of sample C headspace

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AREA %

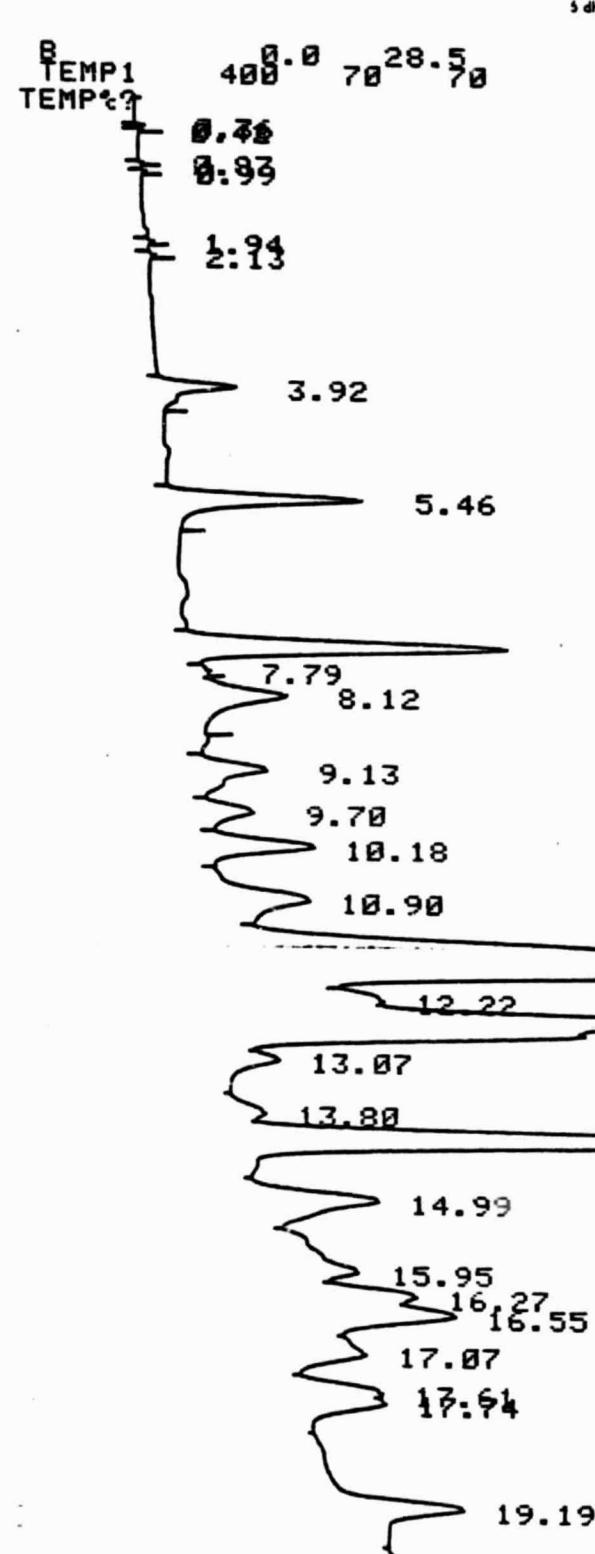
RT	AREA	AREA %
0.35	26	0.012
1.58	18	0.008
2.11	54	0.024
2.72	193	0.087
3.92	3802	1.708
4.09	2481	1.078
4.80	1289	0.579
5.46	10330	4.640
6.06	240	0.108
6.70	1890	0.849
7.47	13230	5.942
7.78	3639	1.634
8.13	8282	3.720
9.13	7214	3.240
9.70	5424	2.436
10.18	5173	2.323
10.90	7856	3.528
11.80	40350	18.123
12.50	21180	9.513
12.69	9140	4.105
13.07	3854	1.731
13.80	3659	1.643
14.17	22830	10.254
ESCAPE		

2 - Propanol

1 - butanol

TIME 14:33:46

## GC of sample #6 headspace

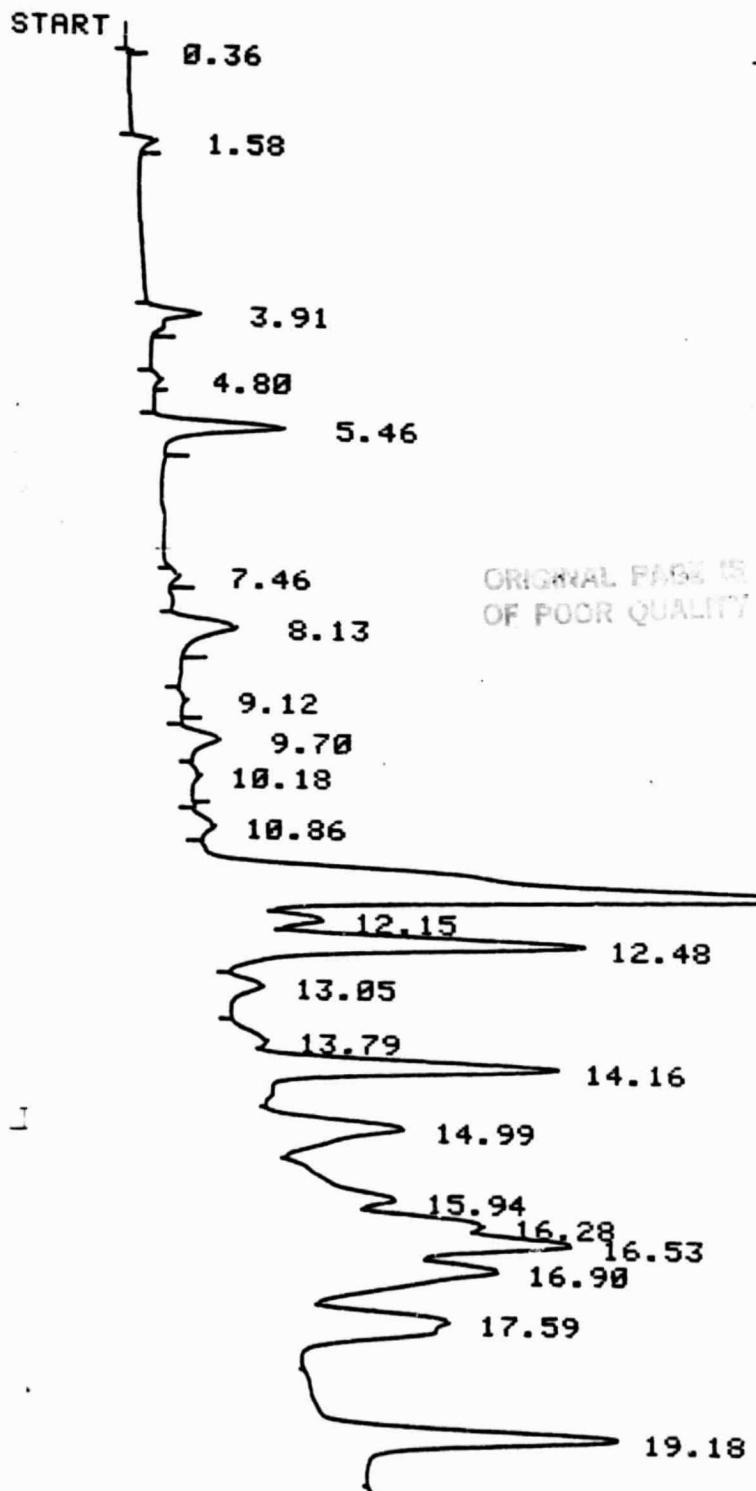


GRAPHIC CONTROLS CORPORATION BUFFALO NEW YORK GC GC-WAI 74703/HP 5

RT	AREA	AREA %
0.36	17	0.010
0.42	7	0.004
0.87	3	0.002
0.99	9	0.005
1.94	17	0.010
2.13	20	0.012
3.92	1284	0.749
5.46	3769	2.198
7.47	6095	3.554
7.79	39	0.023
8.12	2103	1.226
9.13	1774	1.035
9.70	1402	0.818
10.18	3040	1.773
10.90	5038	2.938
11.79	37320	21.763
12.22	4611	2.689
12.49	16900	9.855
12.67	7486	4.365
13.07	3161	1.843
13.80	2618	1.527
14.17	22390	13.057
ESCAPE		1- butanol
		11.79
		12.49
		14.17

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## GC of sample #7 headspace



RT	AREA	AREA %
0.36	12	0.009
1.58	230	0.163
3.91	954	0.677
4.80	103	0.073
5.46	2403	1.706
7.46	145	0.103
8.13	1634	1.160
9.12	103	0.073
9.70	878	0.623
10.18	230	0.163
10.86	507	0.360
11.78	25160	17.860
12.15	3186	2.262
12.48	11820	8.390
13.05	2802	1.989
13.79	3686	2.616
14.16	13510	9.590
ESCAPE		

MOLDS CORPORATION BUFFALO, NEW YORK GC GC-WA17203/HP 9270-0625

HP RUN # 153 APR/05/85

TIME 15:53:32

6031.2

APPENDIX

PROTOCOL:           Atomic Absorption Spectrometry

SAMPLE(s)           Six DC 1200 Silane Primers

ANALYZED FOR:       Si and Ti

ANALYST:           Connie C. Cantor

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36408

NASA

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6/7/64

Instrumental Analysis

Silicon 251.7 nm

0.2 mm slit width

 $\text{N}_2\text{O}/\text{C}_2\text{H}_2$  flame  
linear to 150 ppm

<u>Standards</u>	ABSORBANCE						<u>AIGAB<sub>S</sub></u> : <u>AB<sub>S</sub>/ppm</u>
10 ppm	0.021	0.020	0.019	0.021	0.022	0.021	0.0208 0.00208
25 ppm	0.040	0.041	0.041	0.042	0.040	0.042	0.0411 0.00164
50 ppm	0.075	0.077	0.076	0.075	0.076	0.075	0.0756 0.00151
75 ppm	0.114	0.112	0.115	0.113	0.114	0.113	0.1135 0.00151

Samples

Blank	-0.001	0.000	-0.000	0.001	-0.002	0.001	0.000
3-2	0.112	0.110	0.111	0.107	0.112	0.112	0.111
4-2	0.111	0.110	0.112	0.110	0.112	0.109	0.111
5-2	0.116	0.117	0.118	0.116	0.115	0.117	0.116
6-2	0.119	0.121	0.118	0.120	0.121	0.119	0.119
8-2	0.108	0.107	0.107	0.108	0.109	0.109	0.108
9-2	0.107	0.108	0.109	0.110	0.109	0.107	0.108
3-3	0.101	0.100	0.099	0.101	0.100	0.102	0.101
4-3	0.102	0.105	0.104	0.105	0.106	0.103	0.104
5-3	0.102	0.103	0.101	0.103	0.104	0.102	0.103
6-3	0.099	0.101	0.102	0.103	0.100	0.103	0.101
8-3	0.110	0.111	0.110	0.109	0.111	0.112	0.110
7-3	0.107	0.105	0.106	0.107	0.105	0.106	0.106

Calculations

$$3-3 \quad \frac{0.101 \text{ AB}_S}{0.0015 \frac{\text{AB}_S}{\text{ppm}}} = 67.33 \text{ ppm} \quad 1000 \cdot \omega = \frac{6733.3 \text{ ug}}{7.63 \text{ gm}} = 8825 \frac{\text{ug}}{\text{gm}}$$

$$4-3 \quad \frac{0.104}{0.0015} = 69.33 \times 1000 = \frac{69333}{7.63} = 9087 \frac{\text{ug}}{\text{gm}}$$

SL TESTING INSTITUTE  
SPLASH-OCEAN LABORATORIES, INC.

A-2  
TI 36409

RECEIVED FROM NASA

PROJECT NO. 6031.2  
DATE 6/7/84

$$5-3 \quad \frac{0.103}{0.0015} = 68.66 \times 1000_{\text{ml}} = \frac{68666}{7.73 \text{ gm}} = 8883 \mu\text{g/gm}$$

$$6-3 \quad \frac{0.101}{0.0015} = 67.33 \times 1000 = \frac{67333}{7.73 \text{ gm}} = 8710.$$

$$8-3 \quad \frac{0.110}{0.0015} = 73.33 \times 1000 = \frac{73333}{7.73 \text{ gm}} = 9487$$

$$9-3 \quad \frac{0.106}{0.0015} = 70.66 \times 1000 = \frac{70666}{7.73 \text{ gm}} = 9142$$

$$3-2 \quad \frac{0.111}{0.0015} = 74.0 \times 1000 = \frac{74000}{7.73} = 9573$$

$$4-2 \quad \frac{0.111}{0.0015} = 74.0 \times 1000 = \frac{74000}{7.73} = 9573$$

$$5-2 \quad \frac{0.116}{0.0015} = 77.33 \times 1000 = \frac{77333}{7.73} = 10004$$

$$6-2 \quad \frac{0.019}{0.0015} = 79.33 \times 1000 = \frac{79333}{7.73} = 10263$$

$$8-2 \quad \frac{0.108}{0.0015} = 72.0 \times 1000 = \frac{72000}{7.73} = 9314$$

$$9-2 \quad \frac{0.108}{0.0015} = 72.0 \times 1000 = \frac{72000}{7.73} = 9314$$

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SPACELABORATORY INC.

TI 36410

PROJECT NAME NASA

PROJECT NO 6031.2  
DATE 6

Ti 365.6nm  
0.2 nm slit width

N<sub>2</sub>O/C<sub>2</sub>H<sub>2</sub> flame  
linear to 200 ppm

Standards

	AVG ABS							ABS/PPM
10 ppm	0.039	0.038	0.039	0.039	0.037	0.040	0.0386	0.00386
25 ppm	0.076	0.075	0.074	0.075	0.076	0.075	0.075	0.0030
50 ppm	0.129	0.132	0.132	0.130	0.129	0.128	0.130	0.0026
100 ppm	0.234	0.235	0.235	0.238	0.235	0.238	0.236	0.0024

Samples

	AVG ABS							PPM
Blank	-0.001	0.001	-0.002	0.001	0.000	0.001	0.000	0.0027
3-3	0.136	0.135	0.136	0.138	0.137	0.135	0.136	
4-3	0.128	0.132	0.130	0.132	0.134	0.136	0.132	
5-3	0.129	0.129	0.132	0.130	0.129	0.128	0.130	
6-3	0.128	0.129	0.127	0.129	0.128	0.129	0.128	
8-3	0.133	0.126	0.132	0.127	0.133	0.129	0.130	
9-3	0.136	0.131	0.129	0.131	0.128	0.127	0.129	
3-2	0.125	0.127	0.128	0.126	0.129	0.125	0.127	
4-2	0.126	0.127	0.125	0.126	0.127	0.123	0.126	
5-2	0.128	0.132	0.129	0.129	0.128	0.127	0.129	
6-2	0.135	0.136	0.135	0.134	0.136	0.133	0.135	
8-2	0.130	0.129	0.131	0.130	0.129	0.129	0.130	
9-2	0.129	0.129	0.128	0.129	0.127	0.126	0.128	

CalculationsORIGINAL PAGE IS  
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$$\frac{3-3}{\#1} \frac{0.136 - 50.37 \times 1000}{0.0027} = \frac{50370}{7.63} = 6602 \mu\text{g/gm}$$

$$\frac{4-3}{\#1} \frac{0.132 - 48.88 \times 1000}{0.0027} = \frac{48886}{7.63} = 6407 \mu\text{g/gm}$$

$$\frac{5-3}{\#2} \frac{0.130 - 48.14 \times 1000}{0.0027} = \frac{48148}{7.73} = 6229 \mu\text{g/gm}$$

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A-4

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TEST DATE NASA

PROJECT

DATE

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$$6-3 \frac{0.128}{\#2 \quad 0.0027} = 47.4 \times 1000 = \frac{47407}{7.73} = 6133.$$

$$8-3 \frac{0.130}{\#3 \quad 0.0027} = 48.14 \times 1000 = \frac{48148}{7.73} = 6229$$

$$9-3 \frac{0.129}{\#3 \quad 0.0027} = 47.77 \times 1000 = \frac{47777}{7.73} = 6181$$

$$3-2 \frac{0.127}{\#4 \quad 0.0027} = 47.04 \times 1000 = \frac{47037}{7.73} = 6085$$

$$4-2 \frac{0.126}{\#4 \quad 0.0027} = 46.66 \times 1000 = \frac{46666}{7.73} = 6037$$

$$5-2 \frac{0.129}{\#5 \quad 0.0027} = 47.77 \times 1000 = \frac{47777}{7.73} = 6181$$

$$6-2 \frac{0.135}{\#5 \quad 0.0027} = 50.0 \times 1000 = \frac{50000}{7.73} = 6468$$

$$8-2 \frac{0.130}{\#6 \quad 0.0027} = 48.14 \times 1000 = \frac{48148}{7.73} = 6229$$

$$9-2 \frac{0.128}{\#6 \quad 0.0027} = 47.407 \times 1000 = \frac{47407}{7.73} = 6133$$

Residues at 105°C - 5mls each sample

Sample	wt al pan	wt w/residue(1 hr)	wt w/residue(2 hr)	overnight wt w/residue	% residue
1	1.4338	1.5810	1.5757	1.5677	3.51
2	1.4369	1.6004	1.5951	1.5855	3.84
3	1.3751	1.5331	1.5258	1.5180	3.70
4	1.4351	1.5745	1.5701	1.5649	3.35
5	1.3829	1.5358	1.5290	1.5204	3.56
6	1.4264	1.5902		1--	--

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A-5

36412

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SAMPLE NO.

DATE 6/9/84

## Summary

	Residue @ 105°C	% Ash Dapl	% Ash Avg	% Ti	2Ti: avg	2Si:	% Si: Avg	Ratio Si/Ti
1	3.51	<del>2.9</del> <del>3.0</del>	3.02	0.66 0.64	0.65	0.88 0.91	0.72	1.38
2	3.84	<u>3.34</u> <u>3.07</u>	3.10	0.62 0.41	0.62	0.89 0.87	0.88	1.42
3	3.70	<u>3.19</u> <u>3.09</u>	3.14	0.62 0.62	0.62	0.95 0.91	0.93	1.50
4	3.35	<u>3.14</u> <u>3.17</u>	3.16	0.61 0.60	0.61	0.96 0.96	0.96	1.57
5	3.56	<u>3.36</u> <u>3.44</u>	3.40	0.62 0.65	0.64	1.00 1.02	1.01	1.58
6	3.78	<u>3.15</u> <u>3.17</u>	3.16	0.62 0.61	0.62	0.93 0.93	0.93	1.50

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