DAA/ MARSHALL

SPRINGBORN LABORATORIES INC.

6031.2

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

January 6 - March 1985

3. Date of Publication:

April 11, 1985

4. Type of Report and Contract Number:

10th Progress: NAS8-35818 (Amended work)

5. Author:

F. Mikes

6. Prepared For:

C. Marshall Space Flight Center, AL 35812

cc: AP-29-F - 1x
AS24D - 3x
AT01 - 1x
EM13B-18/Blevins - 1x
EH33/Morris - 10x
NASA Scientific & Technical Info. Facility - 1x + Repro.

LIST OF CONTENTS

TABLE I*

DC 1200 Primer Lots Used in Project Program

Springborn Labs ID #	DC Primer Lot #	Date Received at Springborn	Additional Description and Further Sample Labeling
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 MMSK34A025 83G529
6***	071620	3/30/84	red, S/L Exp 7/82, Temp 50 to 90F, RS1:705481, 08/81 MMSK343A025 81G464
7+	QL103753	1/10/85	red

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

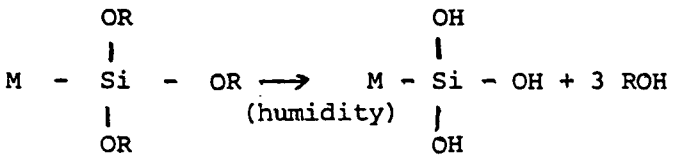
**Three (3) DC 1200 Primer Lots acquired by Springborn Laboratories for initial tests (see monthly reports 1, 2 and 3).

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

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

1. INTRODUCTION

From the series of analytical methods examined for DC 1200 silane primer testing, only one* Fourier transform infrared spectroscopy is presently a candidate for further tests. The FTIR method is currently the best technique for observing hydrolytic changes in the primers caused by moisture in the atmosphere. Hydrolytic changes are detected in the alcohol (OH-band) region of IR spectra of the silane coupling samples as the concentration of silanols (e.g., alcohol or hydroxyl groups) increases.



It has been found that this silane hydrolytic step progresses with time and the concentration of moisture in the sample atmosphere.

To further prove that FTIR can be used as a criterion test for acceptance of silane primer lots, intensities of the FTIR OH- band are being compared with primer adhesive bond strength using a mechanical test suggested by NASA. This is the objective of the amended work under Modification No. 1 of this contract ("changes" clause), a copy of which is enclosed with this report (see Appendix).

To establish a correlation between analytical findings and adhesive bond strength will be the subject of this and future reports.

2. VISIT AT NASA

One of our chemists, Carol A. Mowrey, spent three days at NASA-MSFC (Huntsville, AL) January 6-8, where she was instructed on the bonding/tensile strength test for DC 1200 primers by Bill White and Project Monitor, Don Morris.

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*The headspace GC method for alcohols content may also prove selective but requires further evaluation (see 9th and subsequent 11th reports).

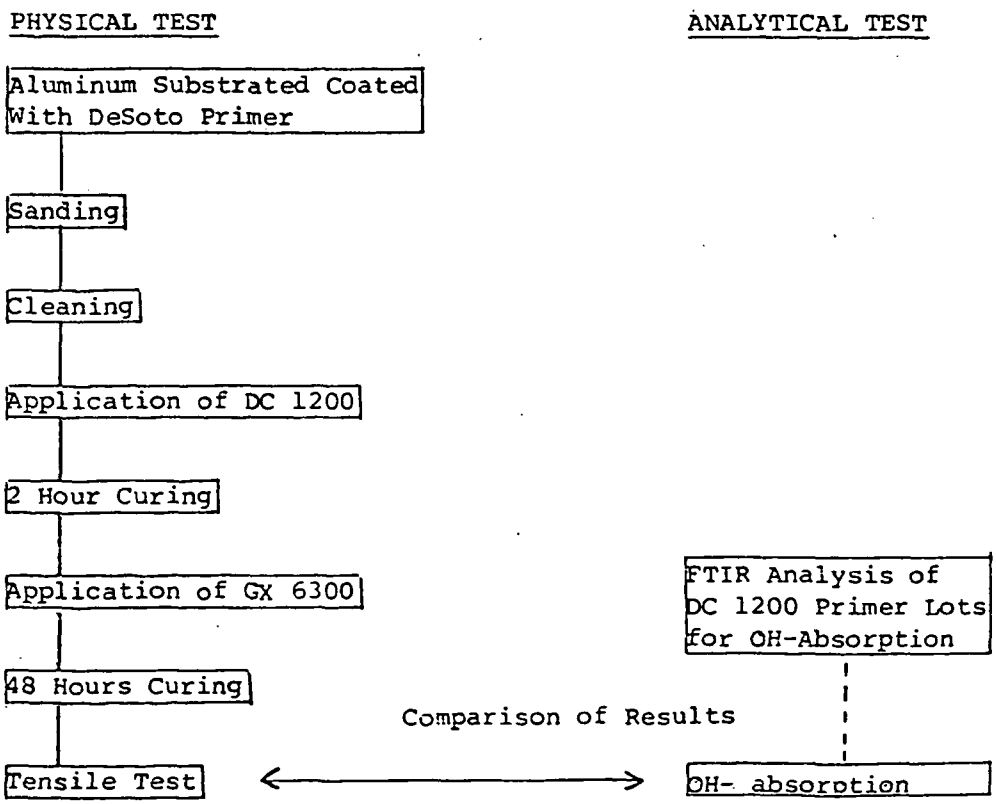
3. BONDING PROCEDURE USING DC 1200 PRIMERS

The following steps are involved in the bonding procedure using DC 1200 primer as coupling agent:

A series of panels, each with 7 metal strips (2.5 x 10 cm) were received for testing with the De Soto primer already applied. After sanding the 1/2 sq. in. overlapping area (where DC 1200 is to be applied), the surfaces were cleaned with toluene and acetone. The sanded areas were then coated with DC 1200 (i.e., wiped with a thin layer of silane using a cotton swab) and left 2 hours for curing at controlled humidity (e.g., 50%). After curing, the GX-6300 rubber mixture was prepared and applied as a thick layer (e.g., 0.5 mm) on the overlapping areas. Two panels were then placed on a metal frame so that their "overlapping areas" (total area 2 x 0.5 sq. in.) were fused through, ends 180° apart. The bonded panel was then removed from the frame and suitably placed between metal sheets with weight (3 lbs/panel) applied. The curing procedure was conducted during 48 hours at 50% humidity.

After curing, sheer tests were performed on the panels with a Tensile Tester Model 1125 (Instron Corp., Canton, MA).

The complete testing procedure is summarized in the scheme below:



4. RESULTS - INITIAL TESTS

After some initial adhesive tests to ensure reproducibility, the first series of DC 1200 primers were tested for sheer strength and OH-absorption. Good results were obtained as reported in the following table. An increase in OH-absorption substantiall decreased the break strength on the Instron Tensile Tester (see average PSI in table), thus confirming our previous studies.

A series of 9 DC 1200 primer FTIR spectra, taken at the same time as their sheer tests, are shown in Figs. 1 to 9.

An example of a lap sheer test results is given in Figs. 10 to 14. Fig. 10 indicates tests performed on samples #7, (a new DC 1200 received from NASA in January 1985 and #B - high OH- presence). Fig. 11 shows sheer test profiles of good sample #7 vs degraded sample #C (and one test with sample B). Fig. 12 is a continuation of test profiles for sample C (3x) and B (1x). Figure 13 indicates differences in the sheer test between samples #5 and #3, respectively. Fig. 14 shows similar patterns for samples #2 and B, plus three profiles of highly degraded sample #C.

An example of lap sheer test data collected from profiles in Fig. 10 is given in Fig. 15a. Fig. 15b shows in graphic form (a panel diagram) the sequence in which DC 1200 primers were applied.

Series of Preliminary Results
Summarizing Lap Sheer Tests
(see average PSI)
vs.
FTIR Absorption Intensities
in OH- Region

DC 1200 Lot #	A_U (Abs. units) = $\frac{Hmm \times Abs.\ unit/inch}{25\ mm/inch}$				Avg. PSI
	3360 cm^{-1}	3380 cm^{-1}	3420 cm^{-1}	3470 cm^{-1}	
1	0.4620	0.4620	0.4620	0.4788	
2	0.7176	0.6992	0.6532	0.5796	91.0
3	1.1252	1.0440	0.9744	0.7772	85.0
4	0.6636	0.6636	0.6636	0.5628	
5	0.5688	0.5544	0.5472	0.5328	152.0
6	0.3016	0.3068	0.3276	0.3484	
7	0.0288	0.0144	0.0048	0.0144	177.6
B	1.2204	1.1232	0.9720	0.7776	79.5
C	1.7716	1.6168	1.7200	1.3244	51.4

5. PLAN FOR FUTURE WORK - SHEER TESTS VS FTIR AND GC ANALYSES

5.1 Sheer Tests

A validation study will be completed and reported as a reproducibility factor (i.e., relative standard deviation of sheer strengths for two DC 1200 primers, coated onto 4 panels each, giving 14 test specimens for each primer, in duplicate. Lap sheer tests will be completed for all available DC 1200 primers.

5.2 GC Analysis

Table of polar headspace volatiles released from DC 1200 primer lots will be presented as:

- qualitative profiles using mass spectral search following GC analysis, and
- quantitative table of components from GC response.

5.3 Correlation of Results

Data from FTIR, GC and stress strength analyses will be compared for degrees of correlation.

6. FINANCIAL STATUS

The total cumulative expenditure incurred as of report 1/6/85 is $6513 (one trip to NASA, $1833; work on project, $4680), equivalent to 26% of the budget.

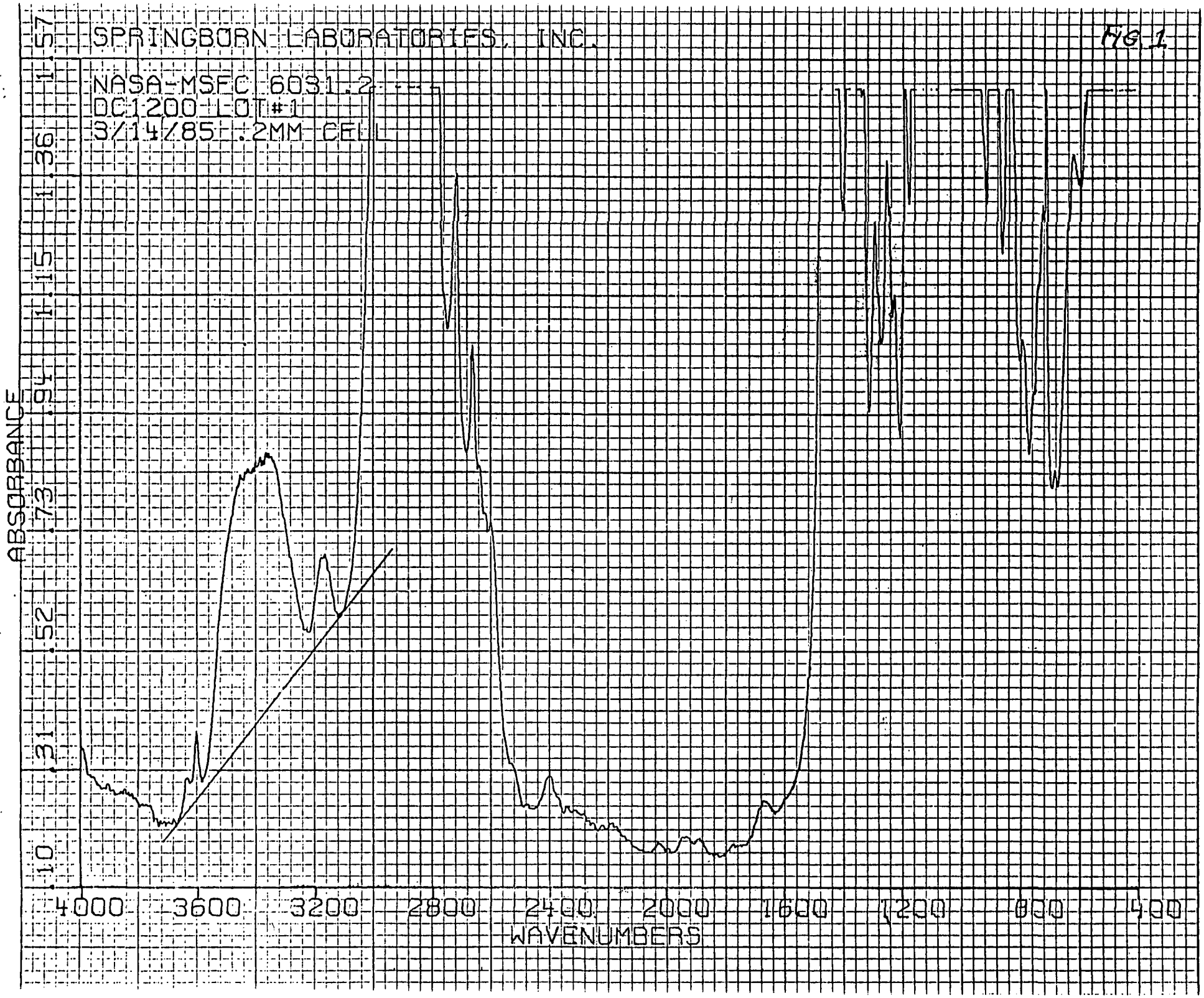

SPRINGBORN LABORATORIES, INC.
Fig. 1
NASA-MSFC 6031.2 ---
DC1200 LOT#1
3/14/85 .2MM CELL
ABSORBANCE
1.57
1.36
1.15
.94
.73
.52
.31
.10
4000
3600
3200
2800
2400
2000
1600
1200
800
400
WAVENUMBERS

FIG. 2

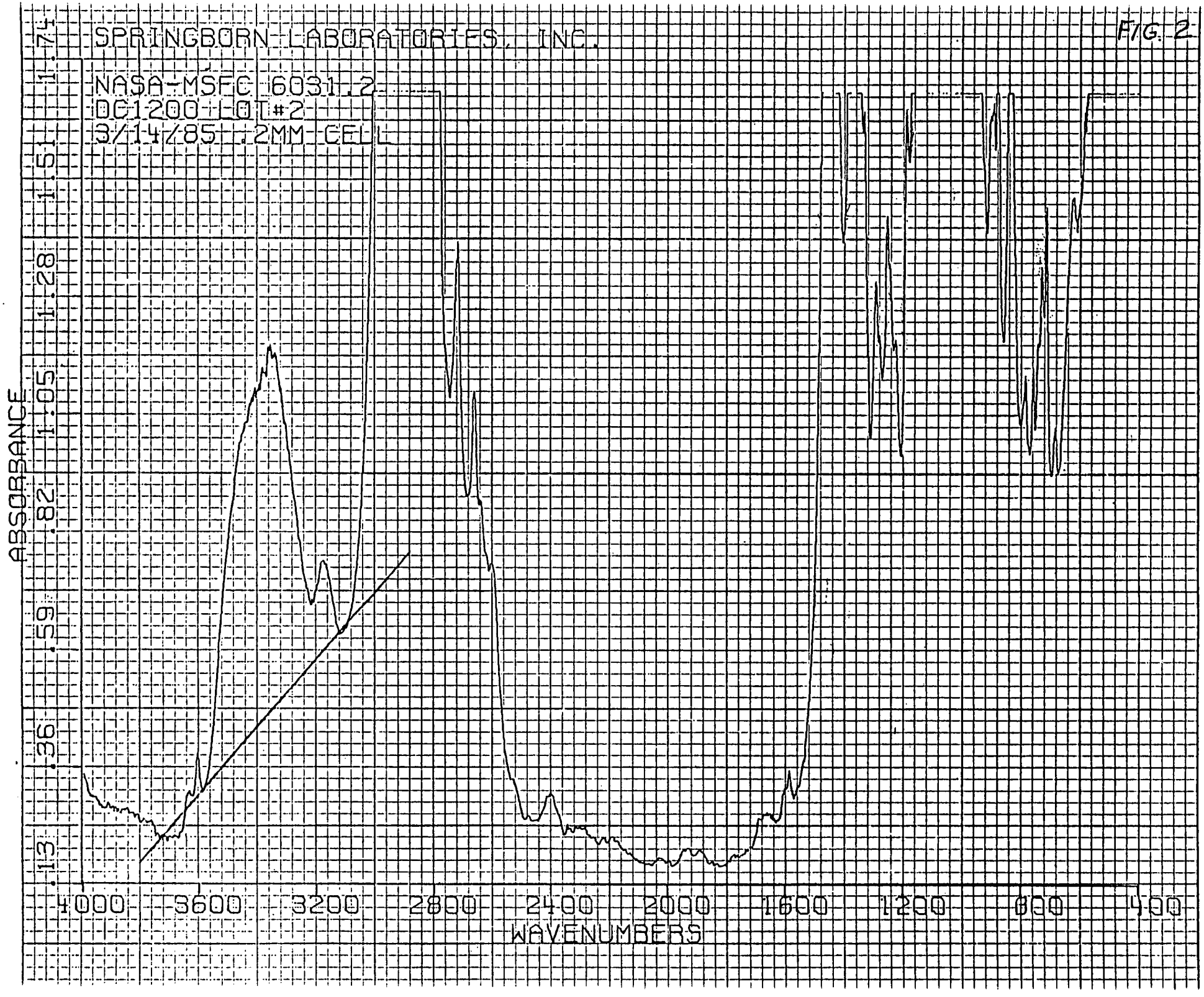

SPRINGBORN LABORATORIES, INC.
NASA-MSFC 6031.2
DC1200 LOT #2
3/14/85 .2MM CELL
ABSORBANCE
1.74
1.51
1.28
1.05
.82
.59
.36
.13
4000
3600
3200
2800
2400
2000
1600
1200
800
400
WAVENUMBERS

FIG. 3

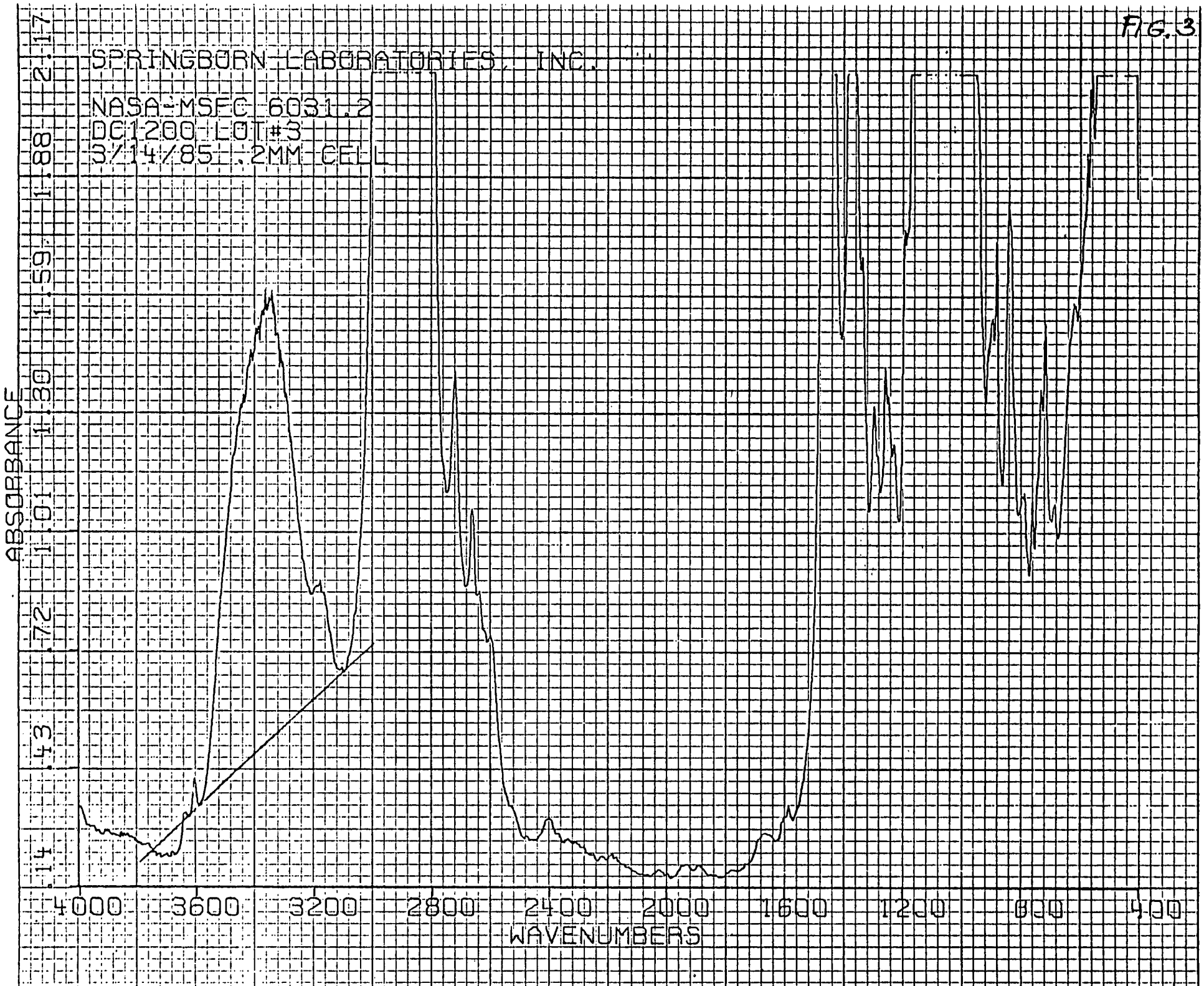

SPRINGBORN LABORATORIES, INC.
NASA-MSFC 6031.2
DC1200 LOT#3
3/14/85 .2MM CELL
ABSORBANCE
.14
.43
.72
1.01
1.30
1.59
1.88
2.17
4000
3600
3200
2800
2400
2000
1600
1200
800
400
WAVENUMBERS

FIG. 4

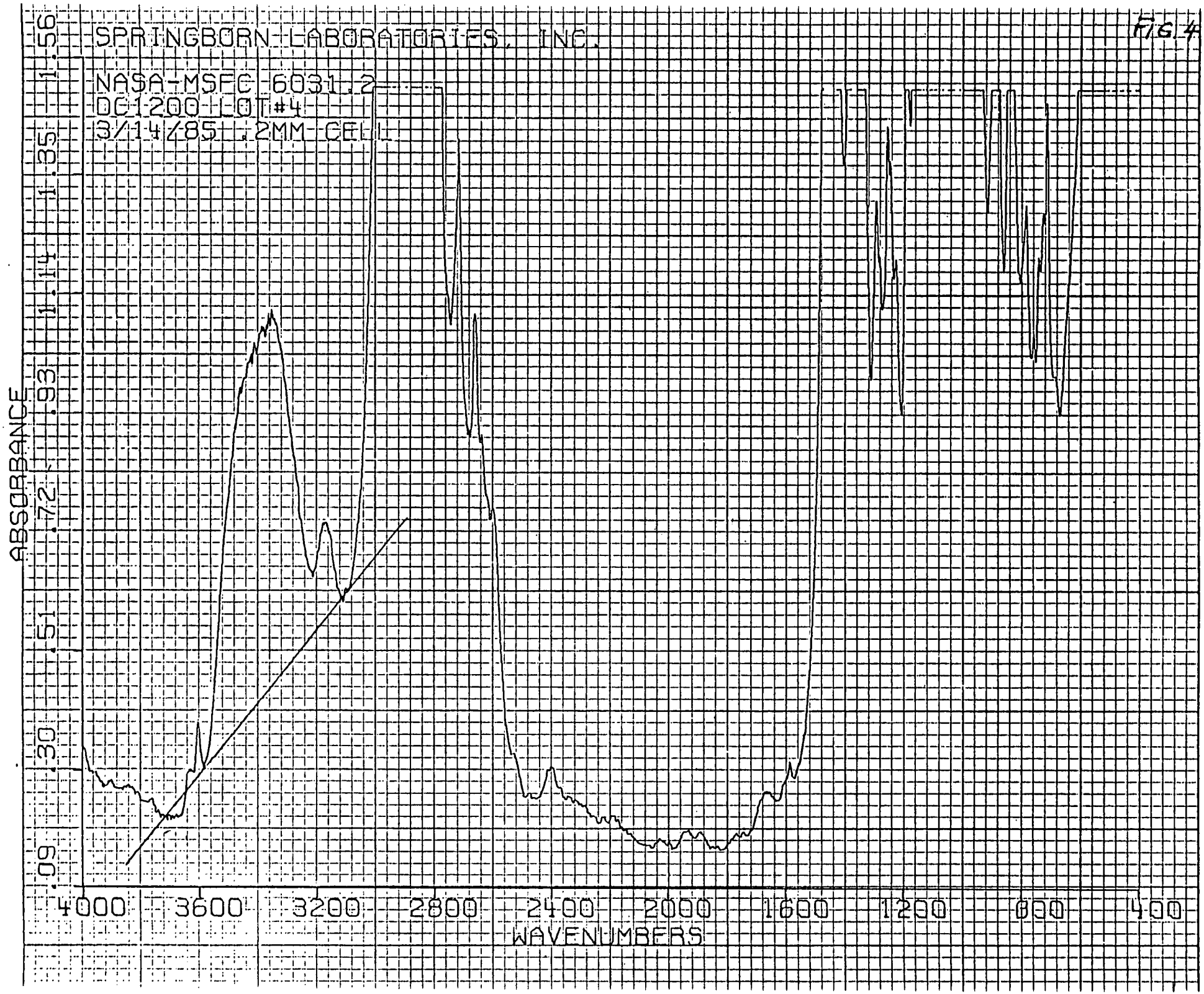

SPRINGBORN LABORATORIES, INC.
NASA-MSFC 6031.2
DC1200 LOT #4
3/14/85 .2MM CELL
ABSORBANCE
1.56
1.35
1.14
.93
.72
.51
.30
.09
4000
3600
3200
2800
2400
2000
1600
1200
800
400
WAVENUMBERS

FIG. 5.

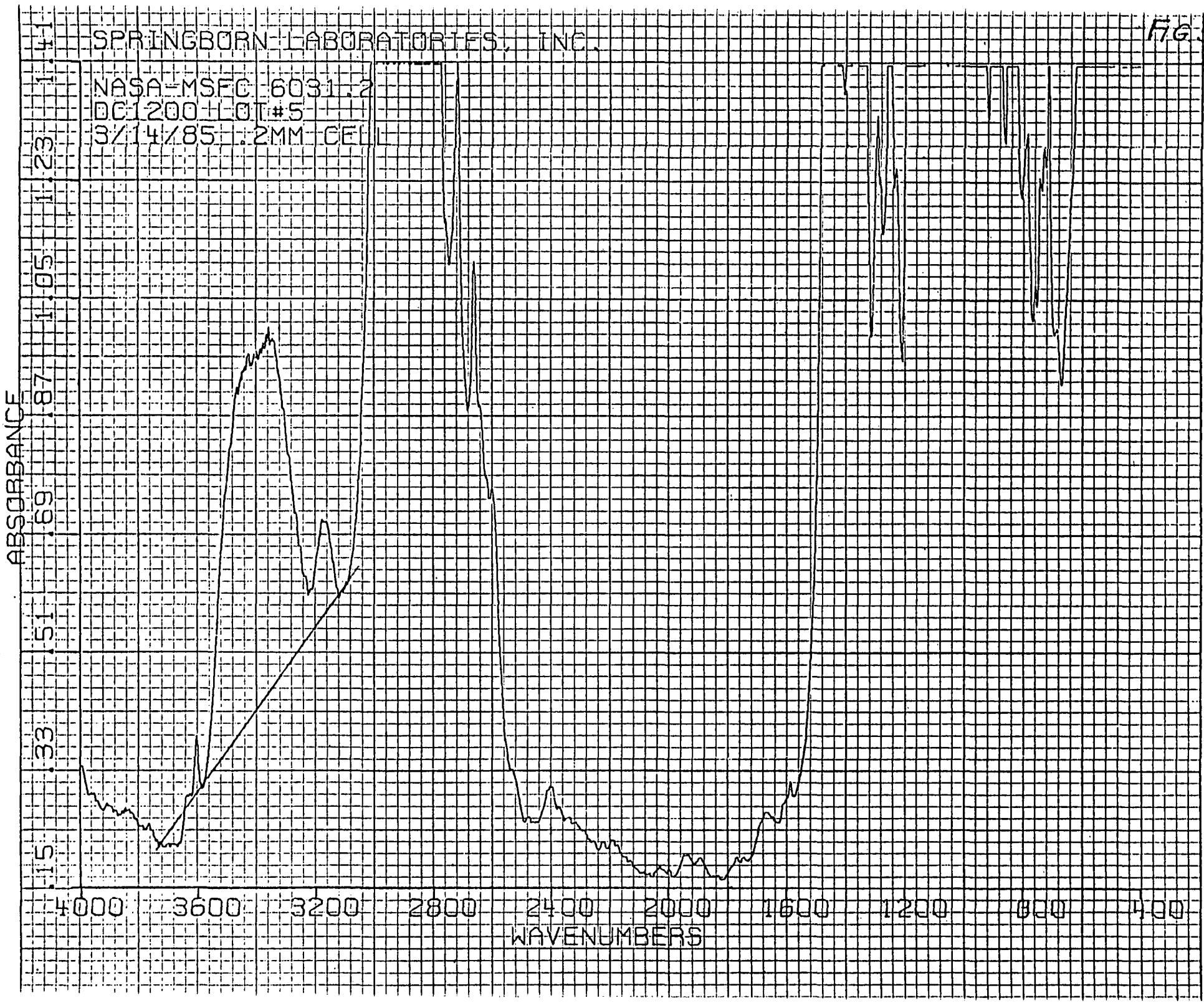

SPRINGBORN LABORATORIES, INC.
NASA-MSFC 6031.2
DC1200 LOT#5
3/14/85 .2MM CELL
ABSORBANCE
.15
.33
.51
.69
.87
1.05
1.23
1.41
4000
3600
3200
2800
2400
2000
1600
1200
800
400
WAVENUMBERS

FIG. 6

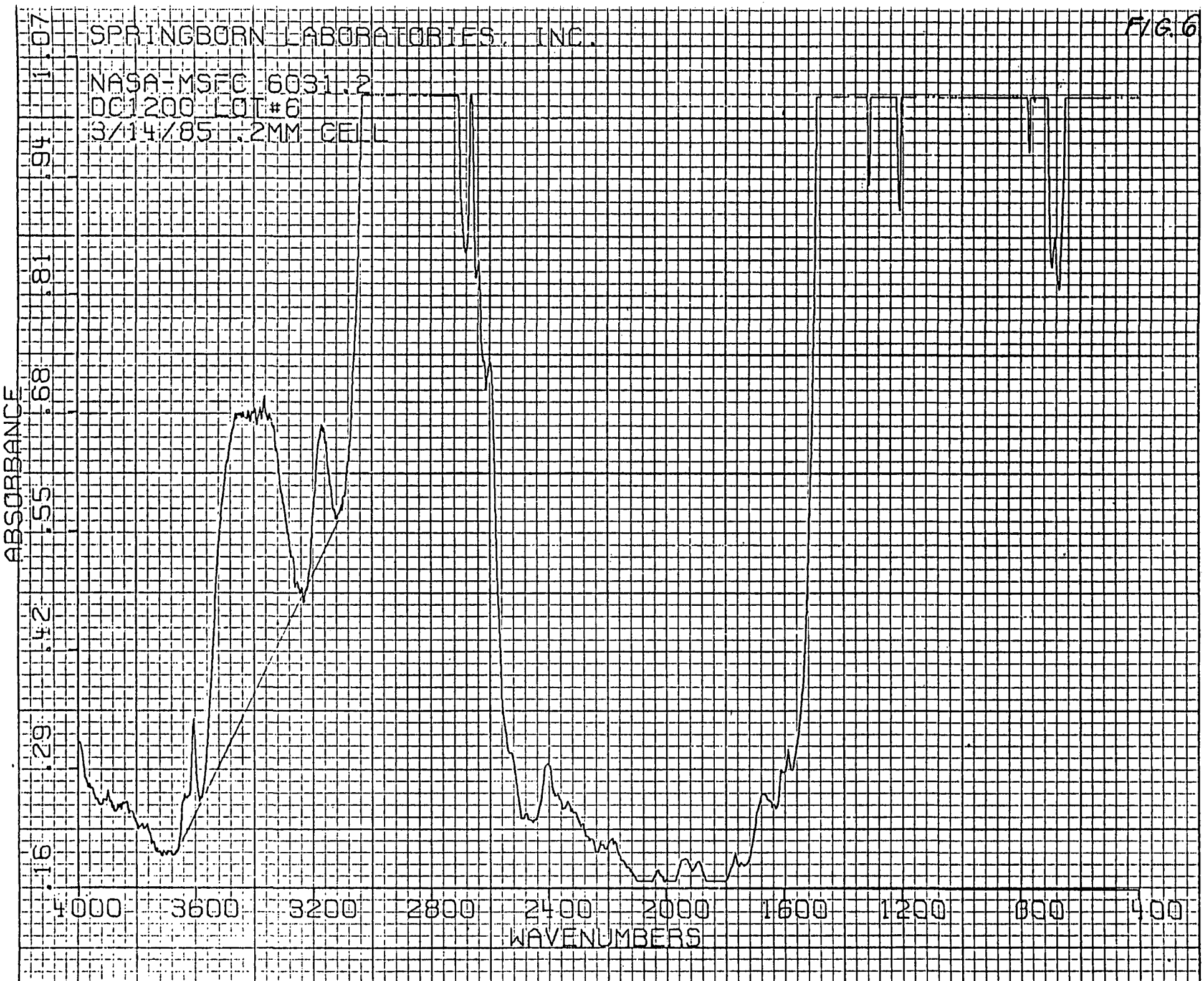
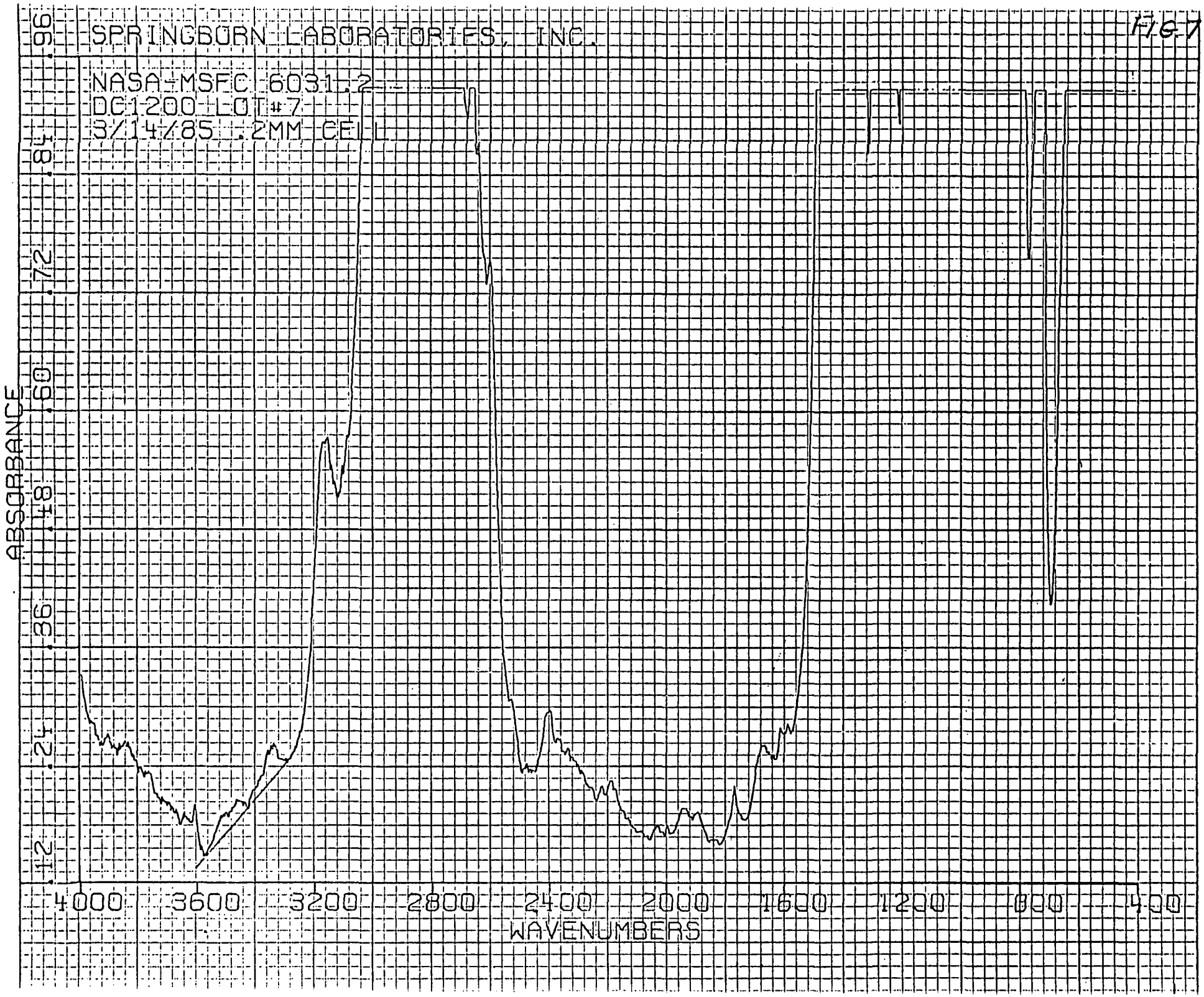

SPRINGBORN LABORATORIES, INC.
NASA-MSFC 6031.2
DC1200 LOT#6
3/14/85 .2MM CELL
ABSORBANCE
1.07
.94
.81
.68
.55
.42
.29
.16
4000
3600
3200
2800
2400
2000
1600
1200
800
400
WAVENUMBERS

FIG 7


SPRINGBORN LABORATORIES, INC.
NASA-MSFC 6031.2
DC1200 LOT#7
3/14/85 .2MM CELL
ABSORBANCE
.96
.84
.72
.60
.48
.36
.24
.12
4000
3600
3200
2800
2400
2000
1600
1200
800
400
WAVENUMBERS

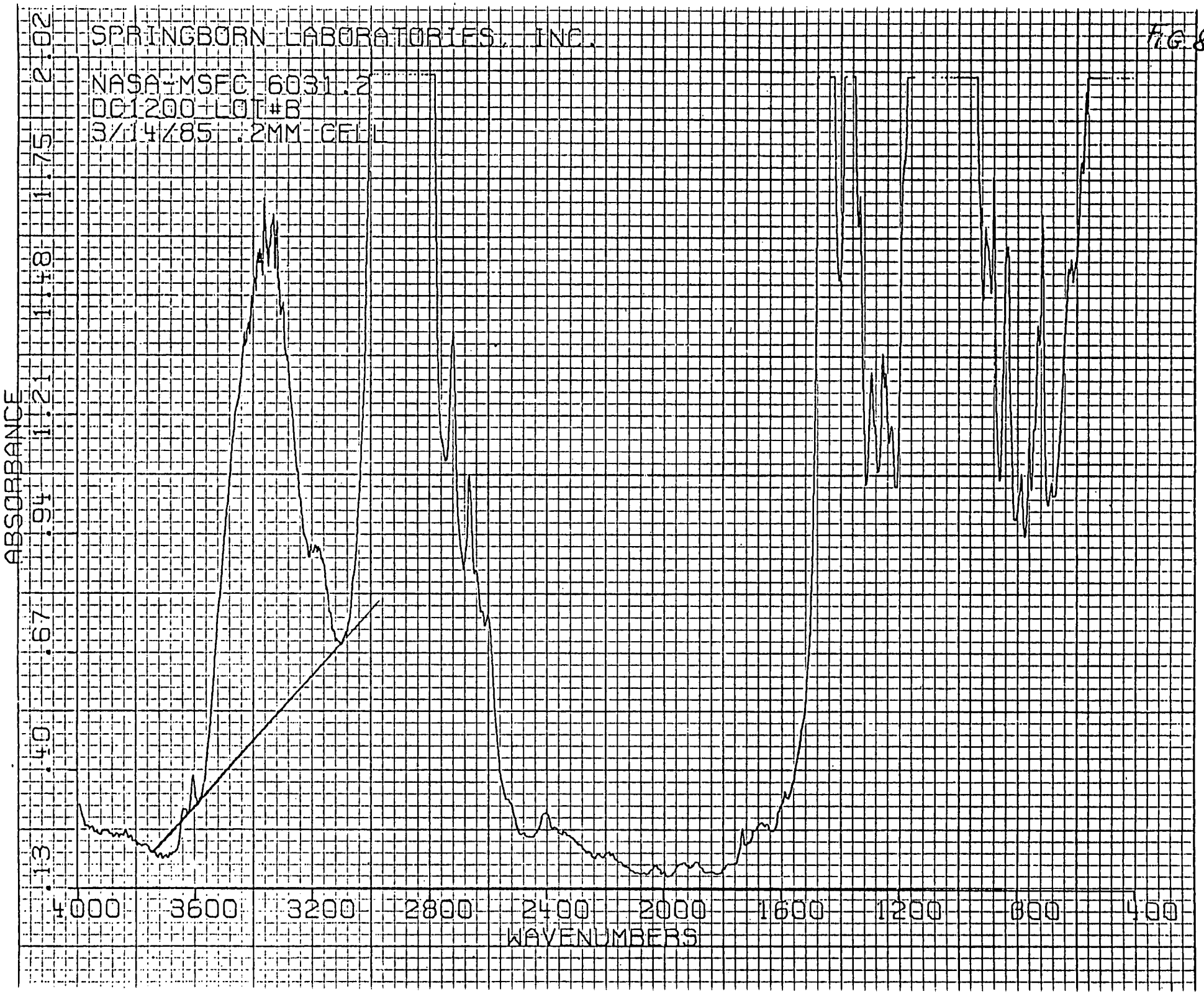

SPRINGBORN LABORATORIES INC.
NASA-MSFC 6031.2
DC1200 LOT #B
3/14/85 .2MM CELL
ABSORBANCE
2.02
1.75
1.48
1.21
.94
.67
.40
.13
4000
3600
3200
2800
2400
2000
1600
1200
800
400
WAVENUMBERS

FIG 8

FIG 9.

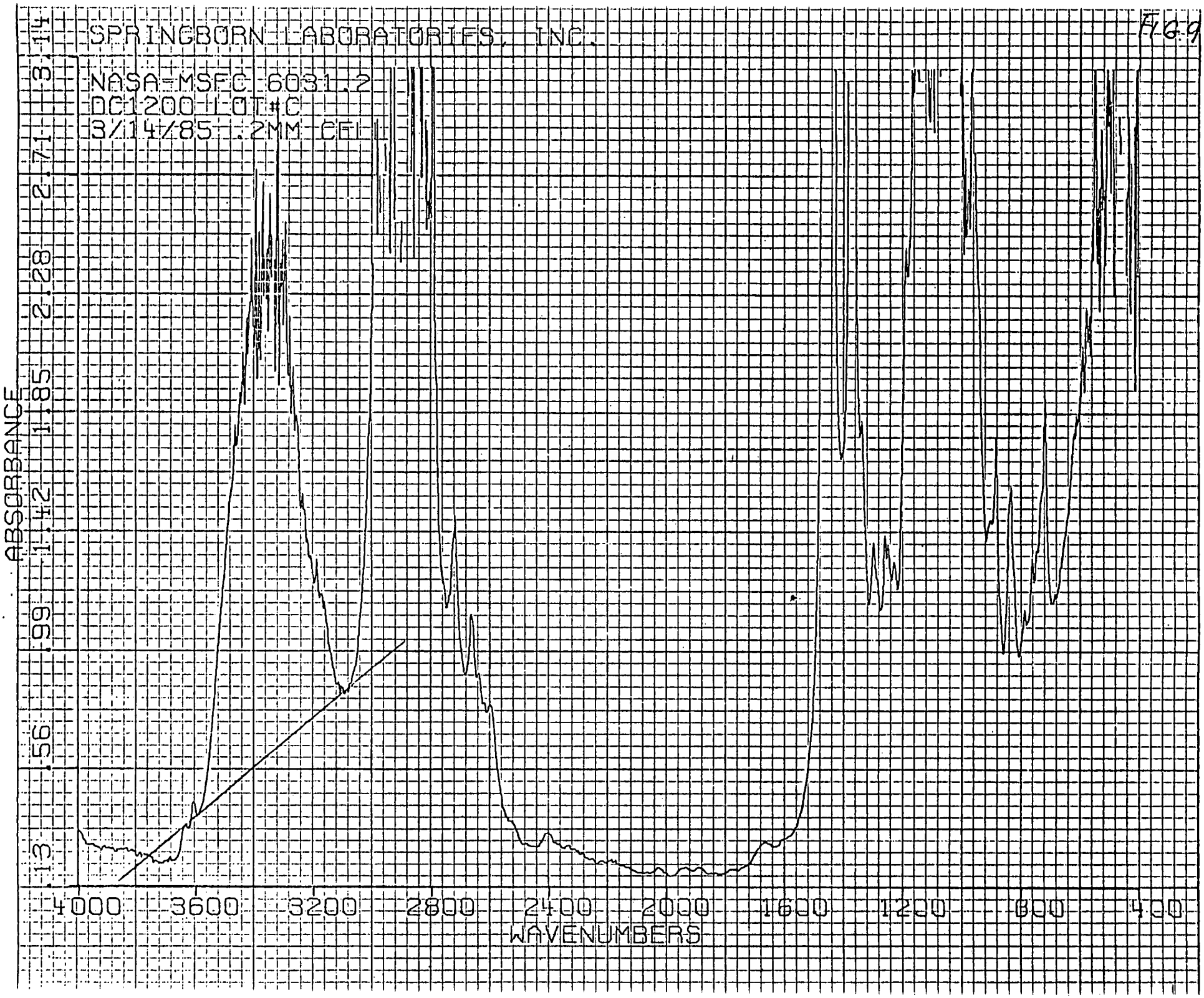

SPRINGBORN LABORATORIES, INC.
NASA-MSFC 6031.2
DC1200 I 0T#C
3/14/85 .2MM CEL I
ABSORBANCE
3.14
2.71
2.28
1.85
1.42
.99
.56
.13
4000
3600
3200
2800
2400
2000
1600
1200
800
400
WAVENUMBERS

FIG. 10

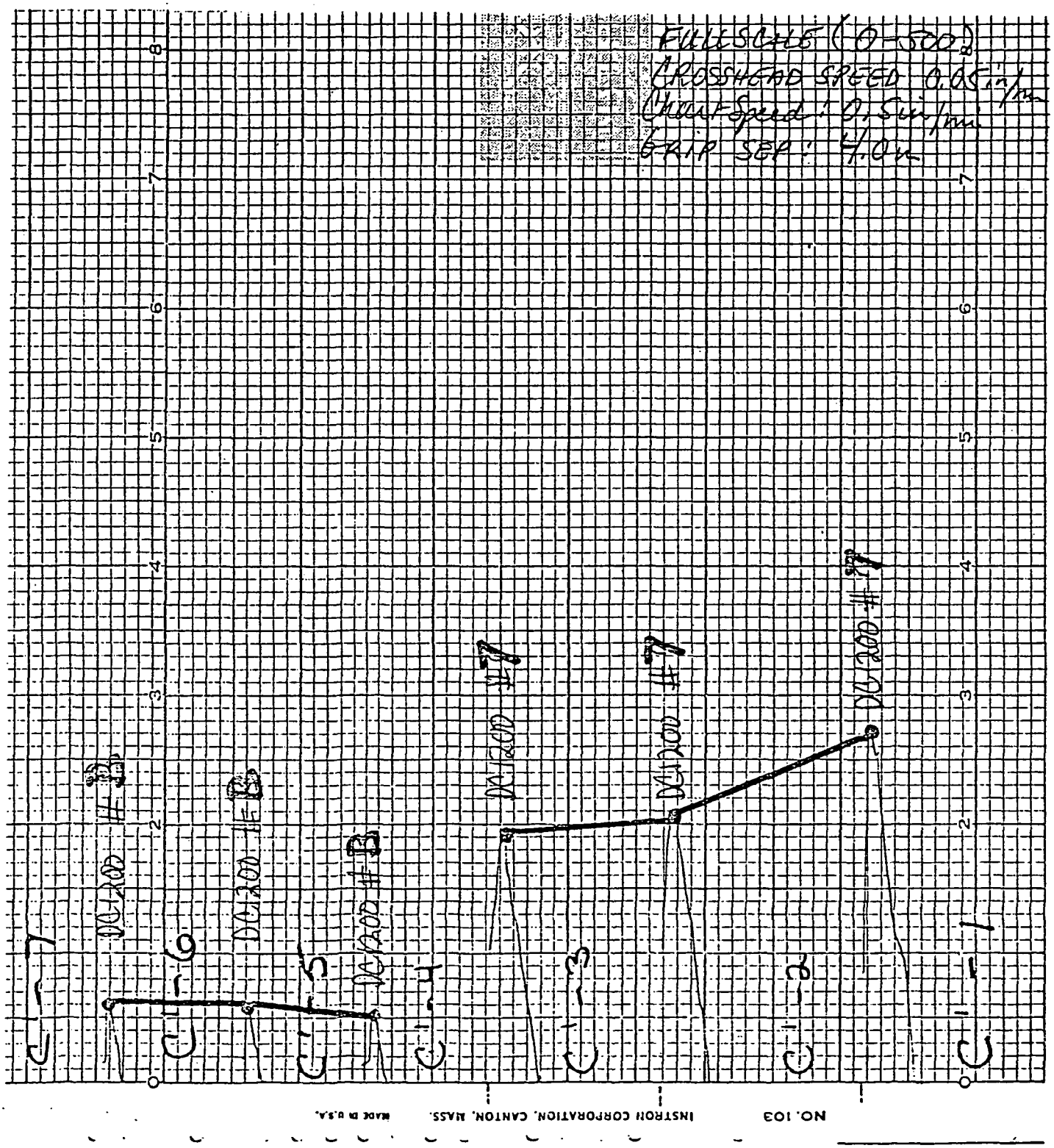

FULLSCALE (0-500 g)
CROSSHEAD SPEED 0.05 in/min
CHART SPEED: 0.5 cm/min
GRIP SEP: 4.0 in
DURAD #17
DURAD #17
DURAD #17
DURAD #17
DURAD #17
DURAD #17
INSTRON CORPORATION, CANTON, MASS.
NO. 103

FIG. 11

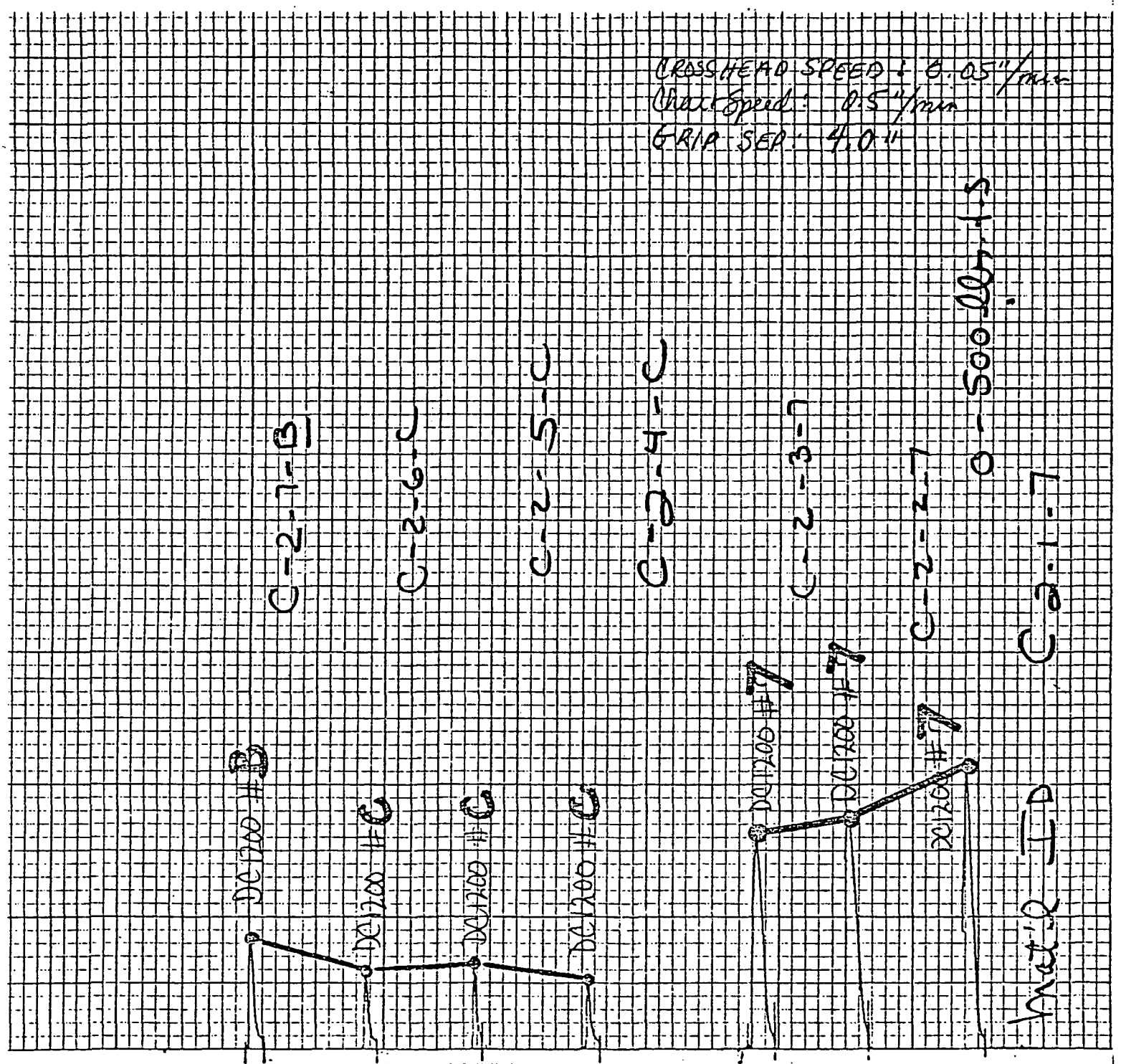

CROSSHEAD SPEED : 0.05"/min
Chart Speed : 0.5"/min
GRIP SEP. : 4.0"
0-500-1047-2
C-2-7-B1
C-2-6-C
C-2-5-C
C-2-4-C
C-2-3-C
C-2-2-C
C-2-1-C
Mat'l-D
DC1200 #B
DC1200 #C
DC1200 #C
DC1200 #C
DC1200 #C
DC1200 #C
DC1200 #C
No. INS 3710-022
PRINTED IN U.S.A.

FIG. 12

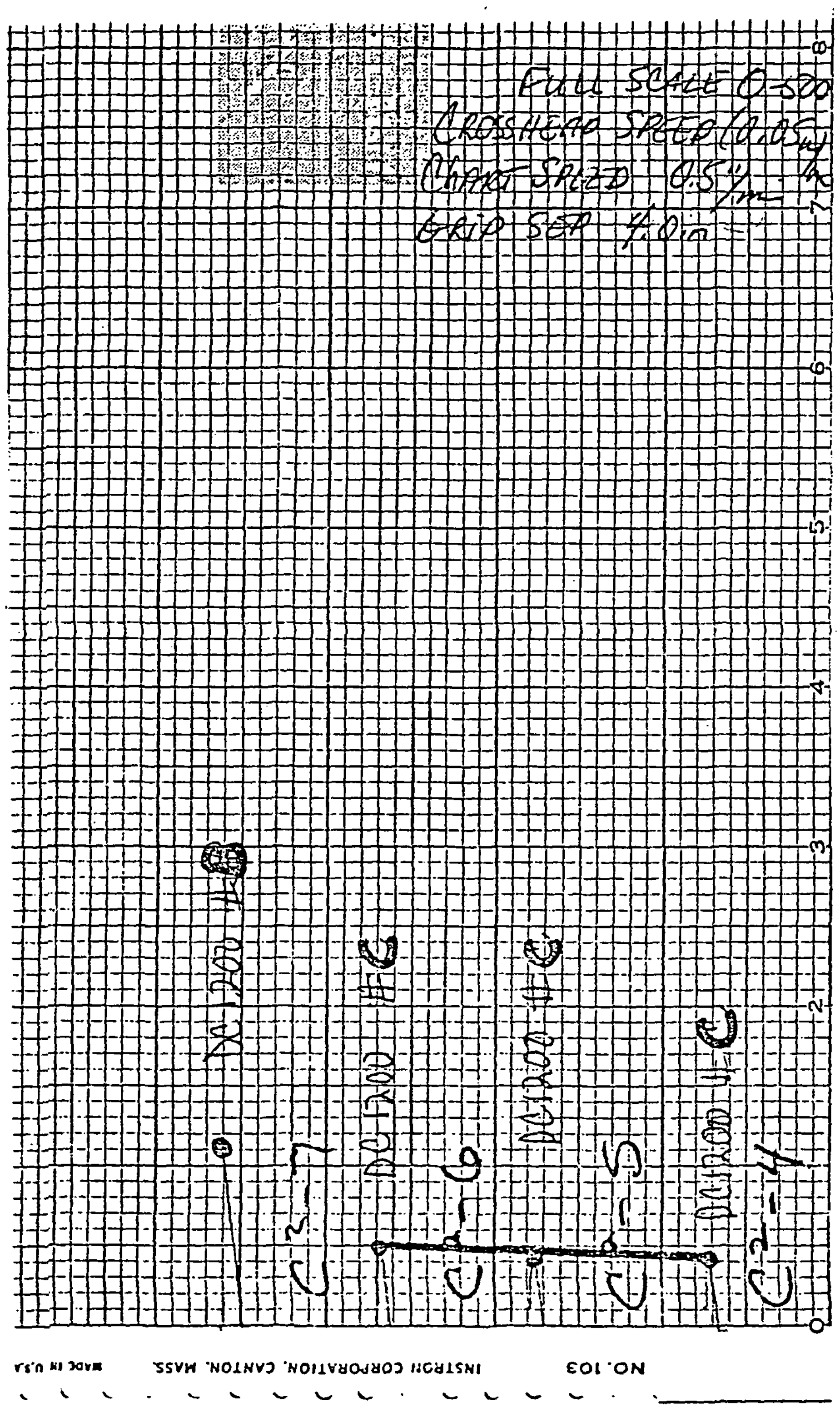

FULL SCALE 0-500
CROSSHEAD SPEED 0.05 in
CHART SPEED 0.5 in/m
GRIP SEP 4.0 in

FIG. 13

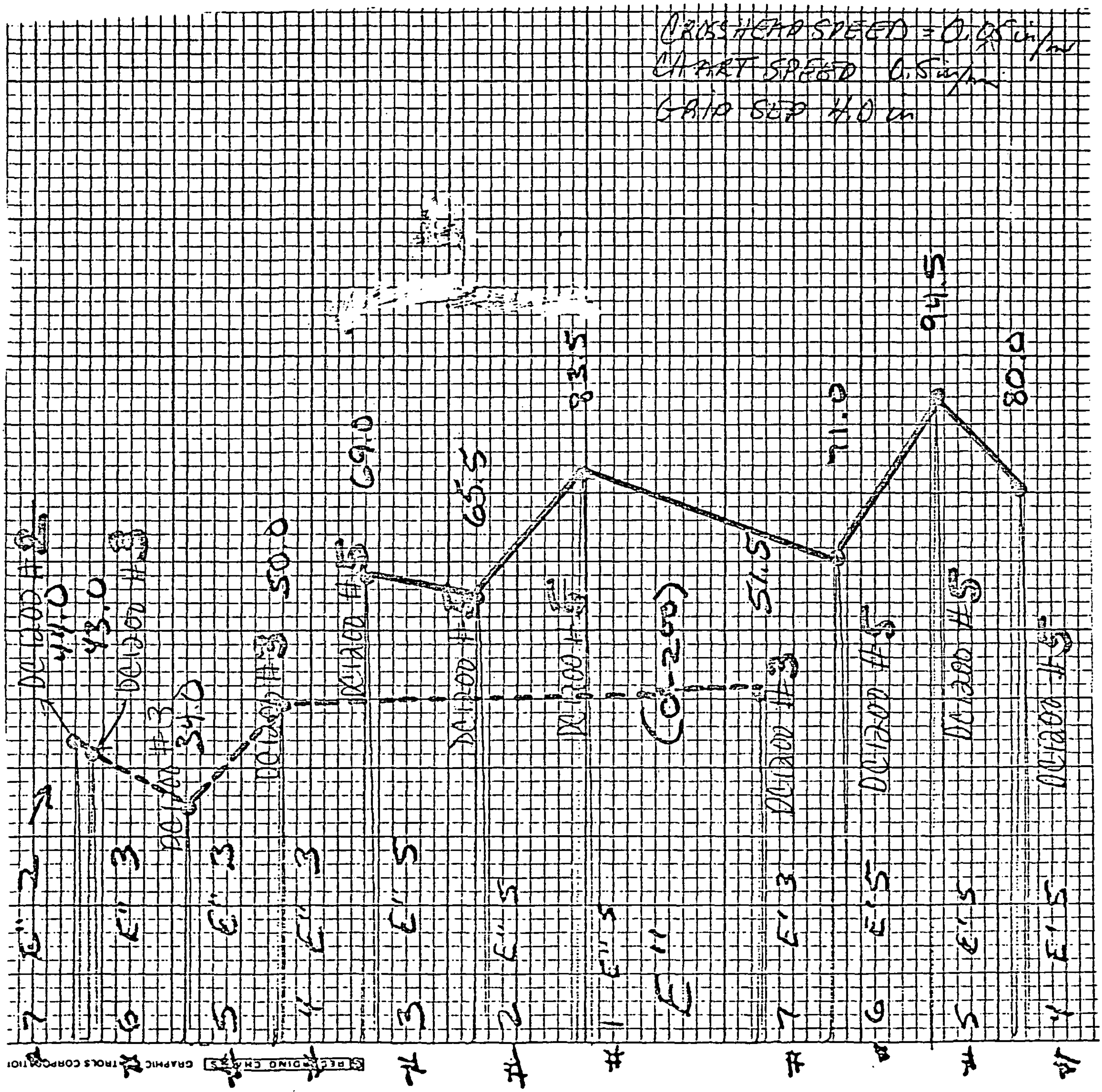

CROSSHEAD SPEED = 0.05 in/min
CHART SPEED 0.5 in/min
GRIP SEP 4.0 in

FIG. 14

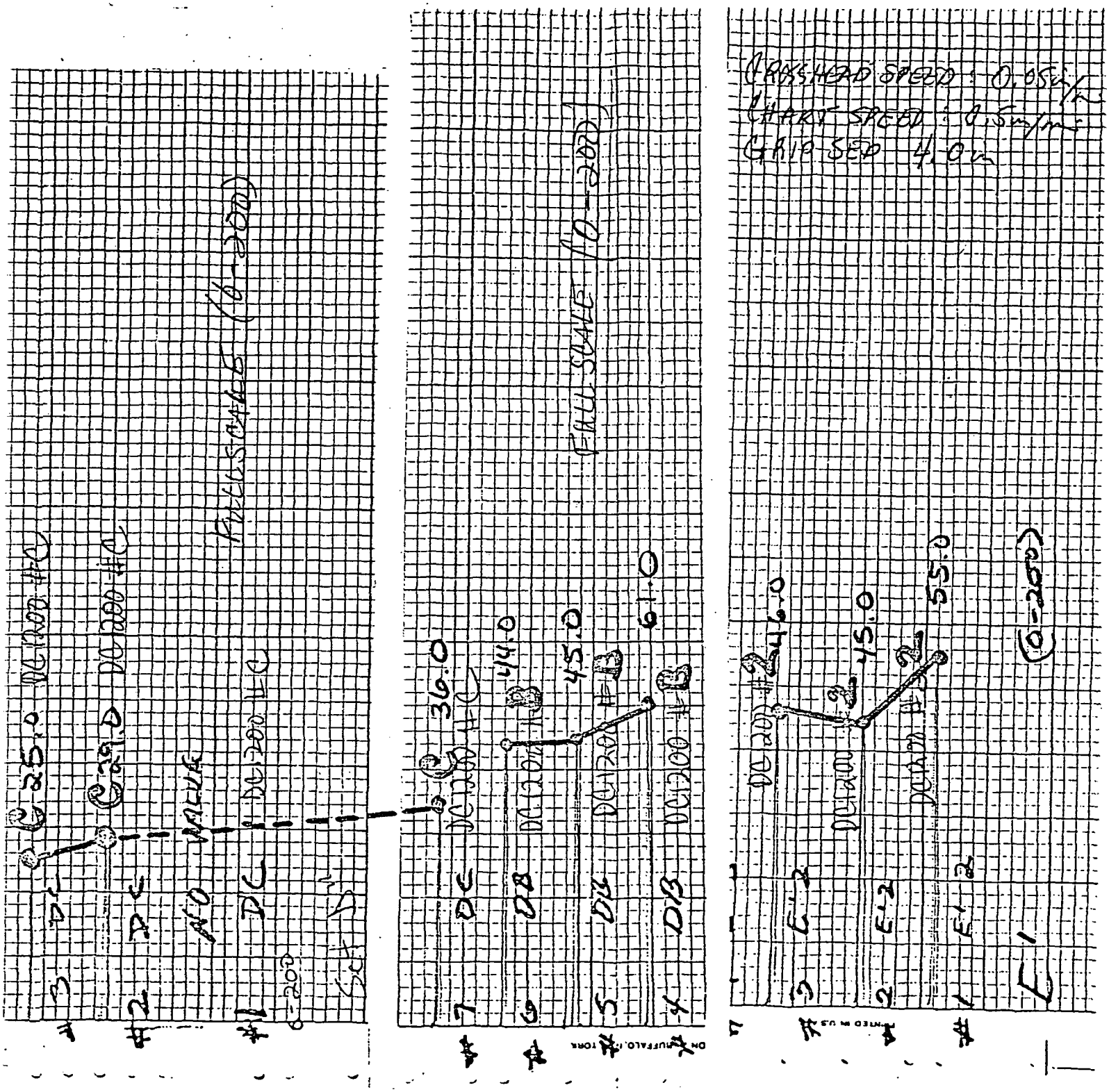

CROSSHEAD SPEED : 0.05 in/min
CHART SPEED : 0.5 in/min
GRIP SEP 4.0 in
(0-250)
(0-200)
(0-200)
FULL SCALE LOAD

FIG. 152

SPRINGBORN LABORATORIES INC

NASA-MSFC 6031.2 LAP SHEAR TESTS

TEST SERIES D'''				
Panel # (ID)	DC1200 ID #	lbs	PSI	% Adhesive Failure
D'''-1	7	58.5	117	95%
D'''-2	7	77.0	154	100%
D'''-3	7	51.0	102	95%
D'''-4	B	61.0	122	95%
D'''-5	B	45.0	90	100%
D'''-6	B	44.0	88	95%
D'''-7	C	36.6	72	100%

COMMENTS: Bubbles Silicone Rubber,

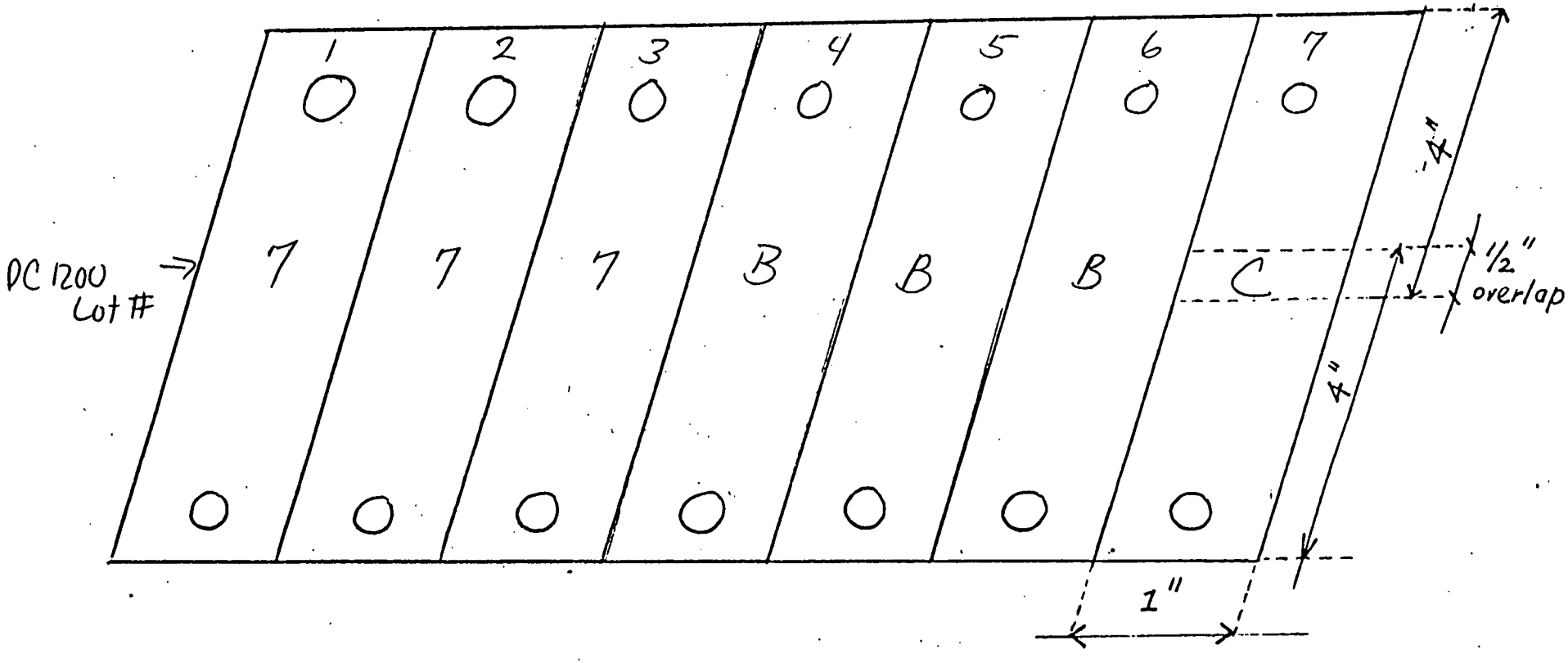
DC1200 #7 -- Trace of acetone in Container (transfer) caused solidification
Data believed not applicable to this study

[redacted] - End panels -- disregard data

FIG. 15b

SERIES PANEL D'''

NASA - MSFC
6031.2



APPENDIX

Modification No. 1 of Contract NAS8-35818

SCOPE OF WORK

EXHIBIT "A"

Delete: B. Phase 2

Add: B. Phase 2

The contractor shall then proceed to utilize the selected instrumentation/ procedure to develop specific methodology and typical acceptance levels, ranges or minimum/maximum values for the DC-1200 primer system. The methodology is to be defined such that it is sensitive to modest changes in primer composition and reactivity. In order to encompass and assess the normal variability of the primer on a lot-to-lot basis, the contractor shall obtain quantities from a minimum of 5 different manufacturer's lots. It is expected that a portion of this lot requirement can be supplied by the Government, depending on available stock at the time. The acceptance ranges or levels shall be developed from a minimum of 5 primer lots.

The contractor shall perform, using appropriate test substrates and adhesive supplied by the Contracting Agency, such adhesive bond testing as required to establish correlations between adhesive bond strength and variations in primer acceptance parameters. This shall include deliberate alteration of each acceptance parameter to the point that its effect on adhesive bond strength is observed.

The appropriate quantity of substrate and GX-6300 silicone adhesive will be furnished (GFE) to the contractor to complete this study. The contractor shall send appropriate bonding personnel to the Contracting Agency, Materials and Processes Laboratory, for at least 2 working days to observe lay-up and bonding techniques involving the GX-6300 adhesive with lap shear test panels.

The contractor will provide the Contracting Agency with ten test panels for adhesive bond tests prepared from lots of primer representing variations in acceptance parameters. The lots of primer to be use for these panels will be determined by the Contracting Agency in a later phase of the project.