FINAL PROJECT REPORT
JUNE, 1992

PROJECT NUMBER: 9-XTI-W7962-1

TITLE: A NEW U.S. MANUFACTURING CAPABILITY OF GLASS PREFORMS FOR FIBER OPTICS IN DEFENSE PROGRAMS

SUBCONTRACTOR: POLYMICRO TECHNOLOGIES, INC.
PHOENIX, ARIZONA

PRINCIPAL INVESTIGATOR: DILIP K. NATH
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SUMMARY

The present project is a part of the program to develop "A New U.S. Manufacturing Capability of Fiber Optics in Defense Programs." The scope of the program extends beyond the limit of defense needs, impacting profoundly on important national issues such as, health industry vis-a-vis medical and insurance infrastructure as well as a great segment of commercial-industrial complex. At present, the glass preform - critical raw material to produce the optical fiber, is 100% imported from Germany and Japan. Objectively, to create a domestic source, a cooperative project participated by Los Alamos and Polymicro began in the summer of 1991 to develop the cladding part of the glass preform. The goal was achieved by developing 0·2NA preform, that was fabricated by Modified Chemical Vapor Deposition (MCVD) of born and fluorine doped silica glass as cladding layer on a silica substrate tube and collapsing the tube on a solid silica rod as the core. The preform was finally drawn into 200 micron core optical fiber and delivered to Los Alamos National Laboratory.

1·0 INTRODUCTION:

A key objective of this program was to create a competitive domestic source for 0·2 NA step-index multimode glass preforms and fibers now supplied by foreign companies currently manufacturing the fiber optics that is considered important as a critical raw material used in nuclear weapons test program, laser ordnance ignition, industrial process control, laser welding and cutting as well as a strongly emerging field of medical application of Minimally Invasive Surgery and Therapy. Heraeus of Germany and Mitsubishi of Japan are the two world suppliers for this type of preforms. Both companies have their own patented processes to manufacture these preforms. The purpose of this project was to develop a similar type of preform based on the patent docketed by D. K. Nath at Polymicro to

- reduce the cost of production of optical fiber by at least 50%;
- improve the performance of currently used fibers by enhancing the quality and design of preforms;
- market these preforms domestically and internationally; and,
- ultimately make new kinds of preforms for application in other fields.

The resultant preforms can then be specifically designed to produce radiation-hard, high laser power, UV and IR transmitting fiber.

2·0 TECHNICAL PROGRESS

The project progressed through four milestones as follows:

- Facility
- Equipments
- Patent
- Process Development

2·1 FACILITY:

Polymicro had no facility to make glass preform. A warehouse-type facility was converted to build

- preform room,
- glass cleaning and storage room,
- computer interface room,
- scrubber room, and
- hydrogen gas storage facility.

2·2 EQUIPMENTS:

A complete set of Modified Chemical Vapor Deposition (MCVD) preform making unit costs about $350K. Because of budget restriction, old equipments were bought and refurbished. The only new equipment bought was a scrubber. The following equipment-related tasks were performed.

- Refurbished - lathe, control consoles, chemical delivery system and computer interface
- Bought/Installed - scrubber, torch system, exhaust, gas lines
- Debugged - lathe system, chemical delivery system, srubber, glass cleaning

2·3 PATENT:

A patent has been docketed by D. K. Nath at Polymicro to fabricate preform of the present project. Because other foreign companies have patents on similar type of glass preforms, a thorough patent search was made. One German and two Japanese companies were identified. The results are shown below.

HERAEUS

- US Patent 4,082,420, April 4, 1978
- RF Plasma Processed
- Highest NA Preform With F Doping
- Used By Most Manufacturers Internationally
- Usable in UV
- Radiation Hard
- Patent Expires April 4, 1995

MITSUBISHI

- US Patent 4,733,939, March 29, 1988
- CVD Processed Preform
- B and F Doping
- OH Content 0.1 to 300 ppm
- 60% More Expensive Than Heraeus
- Radiation Hard
- Not Usable in UV
- Patent Expires March 29, 2005

SUMITOMO

- Jap. Patent J56036606, April 9, 1981
- CVD Processed Preform
- B and F Doping
- OH or OD Content At Least 40 ppm
- Claims on X-rays, Gamma Rays and Radioactive Environment
- Not in UV
- Patent Expires April 9, 1998

RECOMMENDATION BY ATTORNEY

"Limited Patent Protection to a Conventional Optical Fiber Having a High OH Content May Be Available."

2·4 Process Development:

The experimental work started on January 6, 1992. The detailed activities are shown in program schedule (Appendix 1). In essence, the process consists of chemical vapor deposition of fluorine-and-boron-doped silica glass inside a silica tube as cladding layer and then collapsing the tube onto a pure silica rod, which acts as core for guiding light. The resultant glass rod, called preform, is drawn into hair-thin optical fiber to be used as waveguide. The success of the process development effort can be best described by the following achievements.

A. A worldwide search of core rods was completed and samples from Heraeus, Nippon, Mitsubishi, Thermal American, and General Electric were evaluated by Los Alamos National Laboratories. Heraeus and Nippon core rods were found to be the most suitable materials for radiation hardness. Heraeus core rods was used for the deliverable.

B. Boron and fluorine doped silica glass deposition process has been fully developed. Data sheet for the fabrication of the preform that was used for the deliverable is given in Table I.

C. The collapsing technique of the doped silica tube on silica core rods, which act as waveguide, has been developed and optimized via four techniques as given in Appendix 1, Activity No. 15.

D. The fabricated glass preform was drawn into 200 micron core optical fiber. The properties of the fibers are as follows:

	(MICRON)					Attn. Loss (dB/km) at	
Lot No.	Core	Clad	Glass O.D.	Polyimide	NA	308μm	800μm
DBS-01&02	199·2	214·9	265·7	280·7	0·20	90	15·8
DEQ-01	201·4	216·2	268·1	291·1	0·20	98	15·3

The spectral attenuation curves of these fibers in the range 250 - 1120 μm are shown in Figures 1 - 4.

3.0 BUDGET STATUS

ITEM	TOTAL COST ($)	FUNDED BY: PTI ($)	LOS ALAMOS ($)
Capital	41,142	23,355	17,787
Material	19,357	10,645	8,712
Labor	16,652	10,729	5,923
Overhead	41,355	27,359	13,996
Facility	25,000	15,947	9,053
TOTAL(S)	143,506	88,035	55,471

LANL subcontracted this project to PTI for $55,471.

TABLE I.

PREFORM I.D. E-1025
OPERATOR
START/FINISH
START DATE (5.28.92)
START 08:00 FINISH 12:30
COMPLETED? Y ✓ N ___ WHY? ___

RECIPE NO. # BF3/1012
CORE ROD NO. ___
CORE OD 14.82
LITION 9665-1 FILE # 3 (600)
SPINDLE POT:
DEPOSITION 43.5 % COLLAPSE ___ %
SUBSTRATE O.D. 25.62 WALL 1.53 CSA 115.65

BUBBLER LEVEL BEFORE START — TRANSFILLED?

$SiCl_4$	21.3 cm	Y ✓	N ___
$GeCl_4$ I	___ cm	Y ___	N ___
$GeCl_4$ II	___ cm	Y ___	N ___
$POCl_3$	___ cm	Y ___	N ___

OPER	TIME	H_2O		FLAME H_2 LPM	FLAME POT	FLAME O_2 POT	BY PASS O_2 LPM	BY PASS O_2 POT	"IN" O.D.	MD OD	EX OD
	8:48	.14		42.0	21.0	80%	1.21	24.0			

DEPOSITION

OPER	TIME	TYCOM STEP #	CAB. TEMP	O_2 LPM	He SCCM	$SiCl_4$ GPM	$SiCl_4$ CARR	$SiCl_4$ %	$POCl_3$ GPM	$POCl_3$ CARR	$POCl_3$ %	$GeCl_4$ I GPM	$GeCl_4$ I CARR	$GeCl_4$ I %	BCl_3 SCCM	FLAME H_2	FLAME O_2	AUT/MAN
	08 56	005	34.8	3.70	681											43	100%	MAN ✓
	09 06	15	35.0	1.11	—	2.50	396	84.4							11.7	43.0	"	"
	09 36	50	35.1	1.11	—	2.51	426	78.5							11.8	46.0	"	"
	10 21	105	35.0	1.11	—	2.49	457	73.6							11.8	48.0	"	"
	11 05	160	34.9	1.39	—	2.49	470	71.1		grade out					6.4	51.0	"	"
	11 12	165	34.9	1.79	—	2.50	471	70.8		"					3.5	52.0	"	"
	11	170	34.8	4.03	--	1.54	308	66.8							out	80.0	"	"

TUBE: 021/21877

	AVG	STD	MIN	MAX
OUTER DIAMETER :	25.62mm	0.04mm	25.55mm	25.73mm
INNER DIAMETER :	22.57mm	0.05mm	22.44mm	22.69mm
SIDING :	0.13mm	0.00mm	0.13mm	0.14mm
BOW :				0.33mm

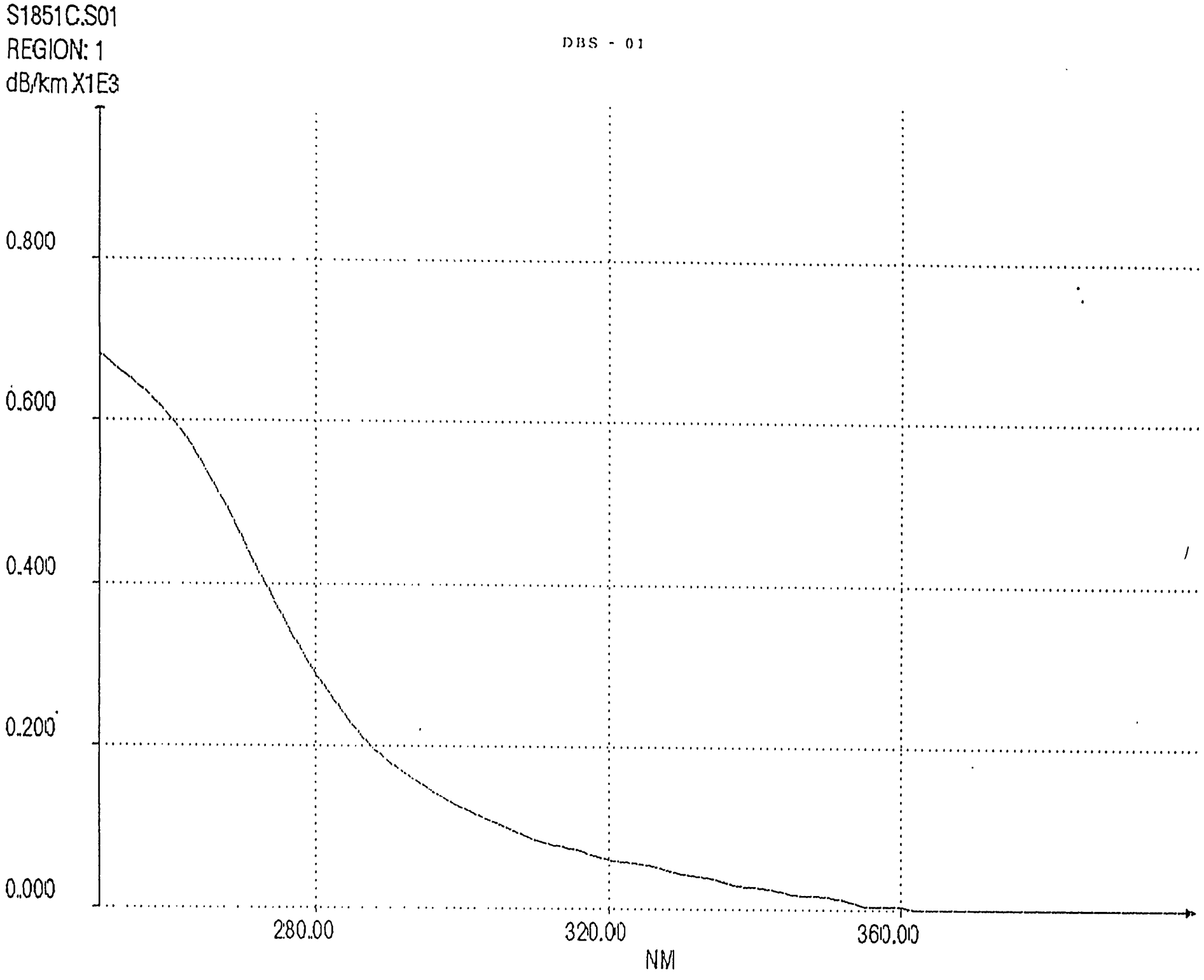

S1851C.S01
REGION: 1
dB/km X1E3
DBS - 01
0.800
0.600
0.400
0.200
0.000
280.00
320.00
360.00
NM

Figure 1

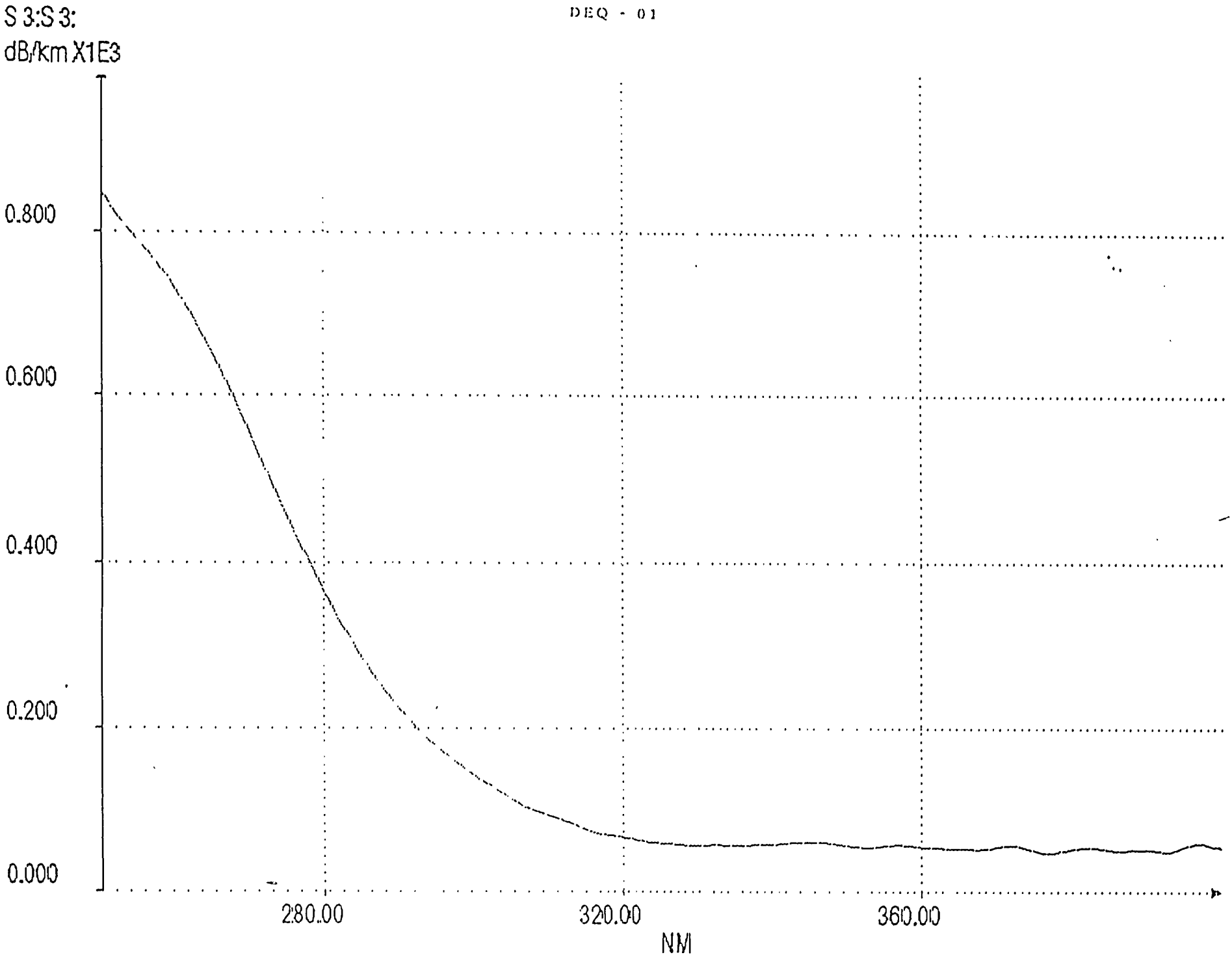

S1852C.S00+ REGION: 1
S 3:S 3:
dB/km X1E3
DEQ - 01
0.800
0.600
0.400
0.200
0.000
280.00
320.00
360.00
NM

Figure 2

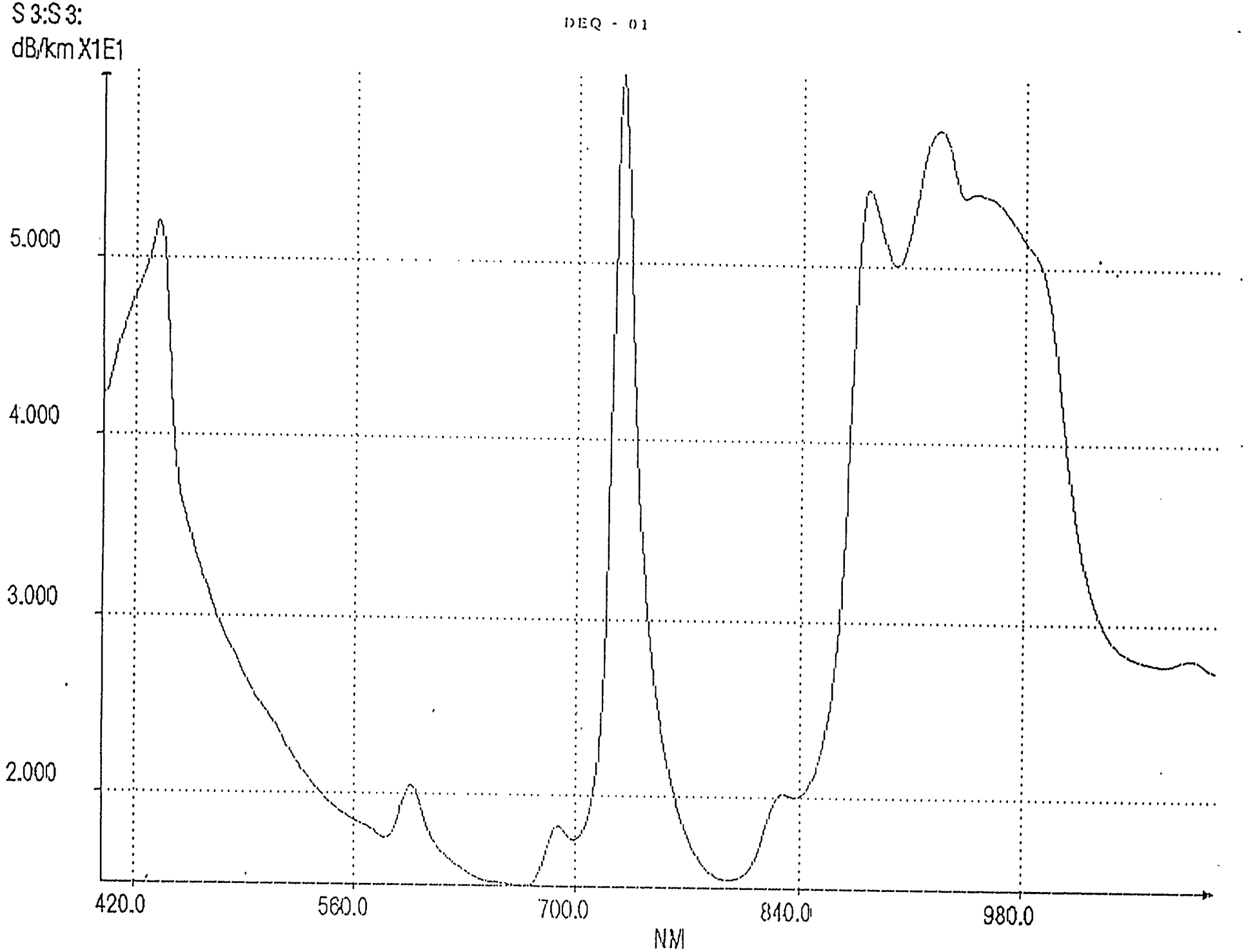

S1853C.S00+ REGION: 1
S 3:S 3:
dB/km X1E1
DEQ - 01
5.000
4.000
3.000
2.000
420.0
560.0
700.0
840.0
980.0
NM

Figure 3

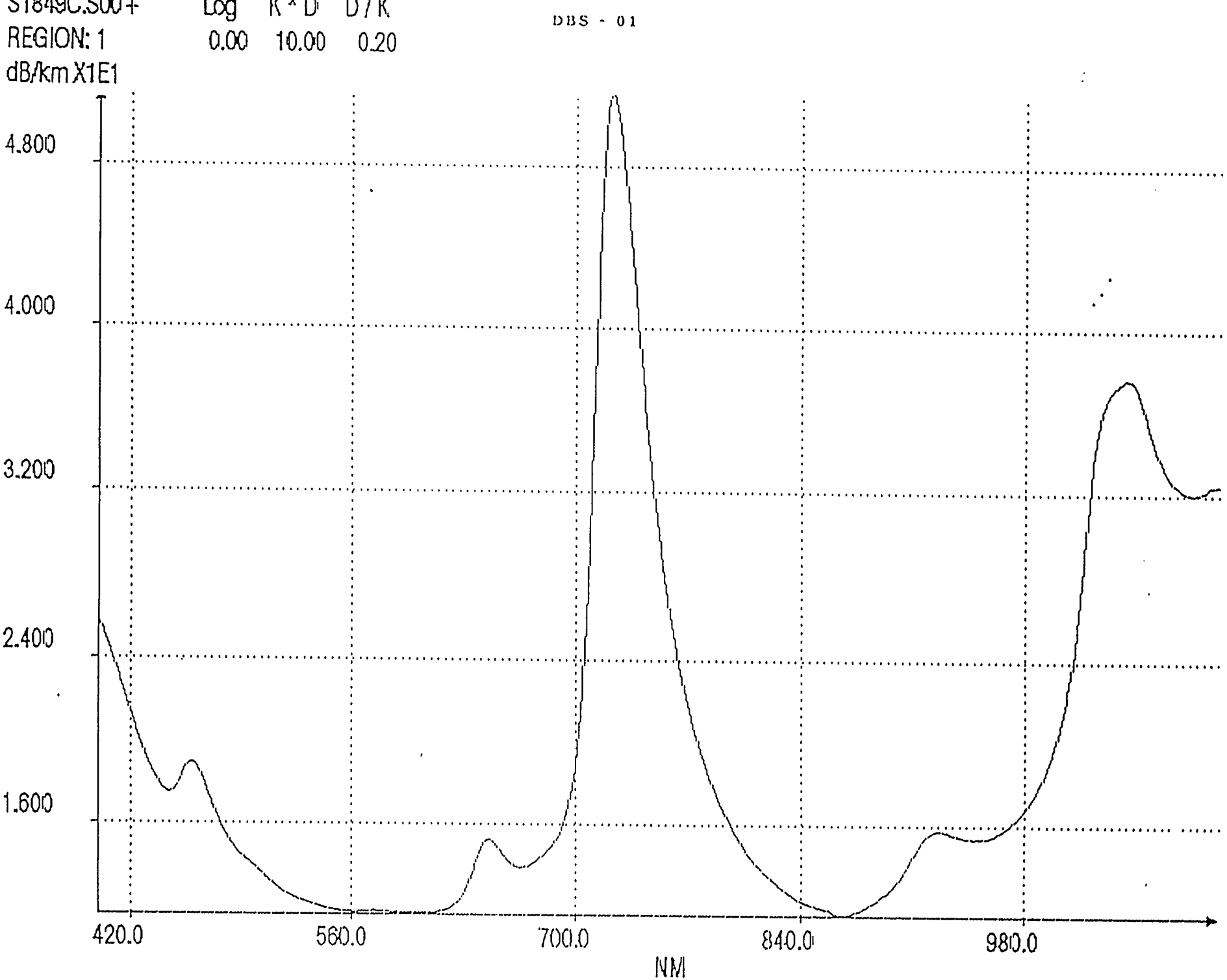
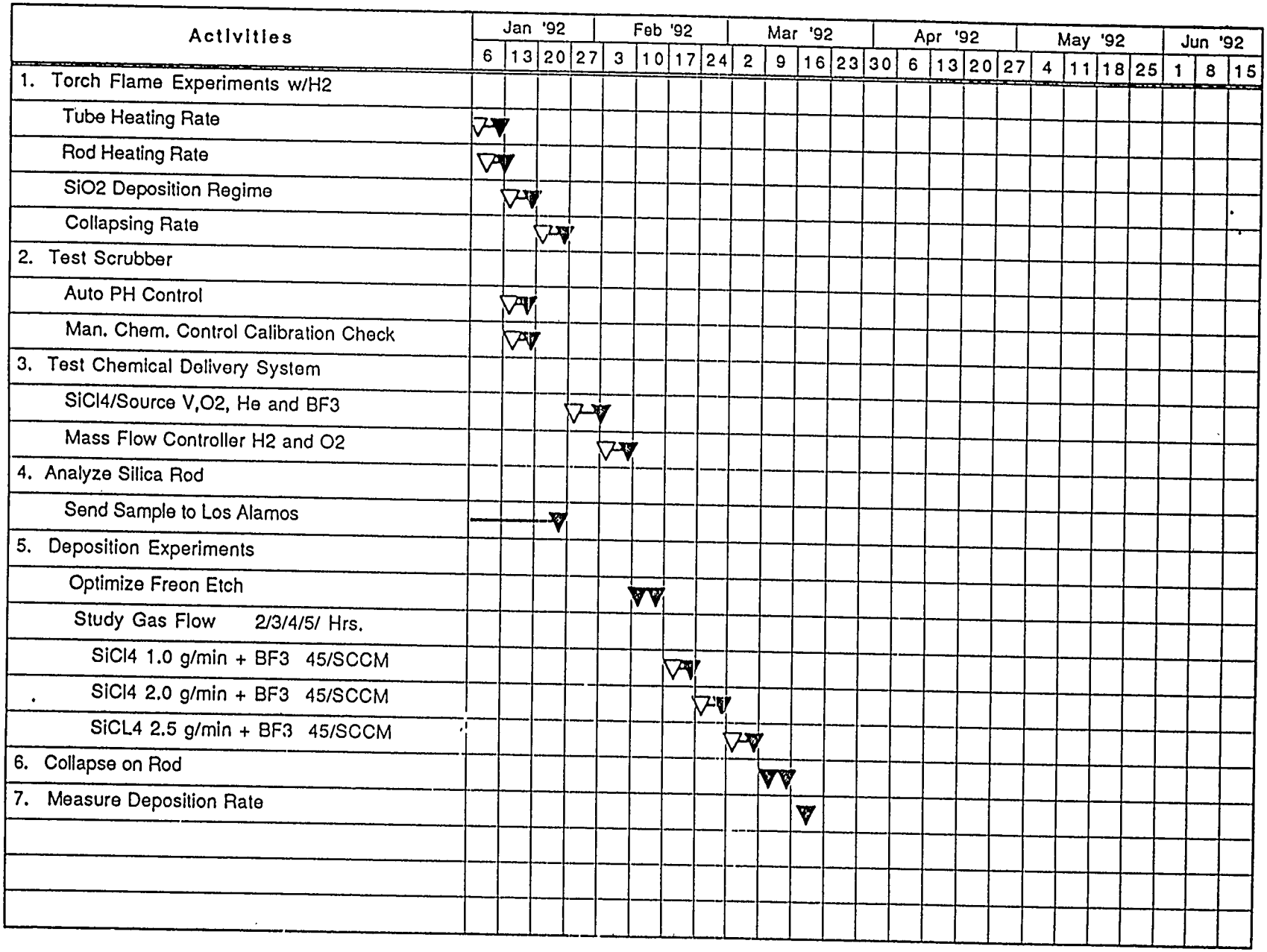

S1849C.S00+ Log K * D D / K
REGION: 1 0.00 10.00 0.20
DBS - 01
dB/km X1E1
4.800
4.000
3.200
2.400
1.600
420.0
560.0
700.0
840.0
980.0
NM

Figure 4

APPENDIX 1

Glass Project Schedule



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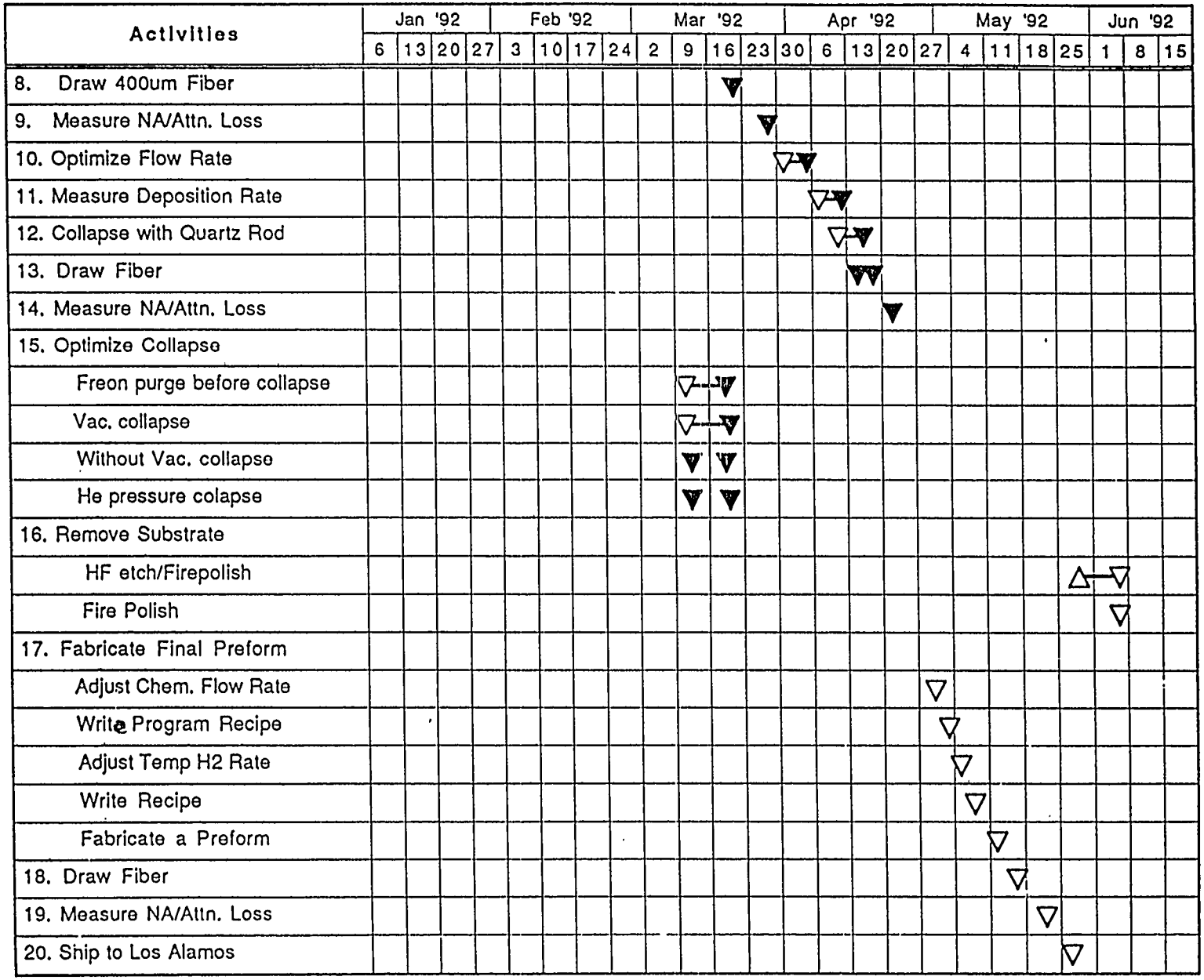
APPENDIX 1

Glass Project Schedule



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