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MICROWAVE RESONANT ABSORPTION OF POTENTIAL EXOTHERMIC COMPOUNDS

FINAL REPORT

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FINAL REPORT

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An investigation was conducted into the microwave resonant absorption of ten energetic compounds over the frequency range from 2.5 to 18 GHz. The samples ranged in sensitivity from relatively inert liquid fuels to the most sensitive explosive initiators. Low power testing showed no strong frequency dependence for any of the materials tested. High power tests were conducted at an S-band frequency at power densities exceeding 1 kW/cm^2. No detonations, deflagrations or decompositions were observed. As a result of these tests one can conclude that, for the materials tested and in the frequency range investigated, no susceptibility to direct high power microwave radiation exits for power density levels less than one kilowatt per centimeter squared.

Microwave Absorption by Potentially Exothermic Compounds

Final Report

1. INTRODUCTION

Microwave radiation is believed capable of initiating explosive reactions in exothermic materials through dielectric breakdown phenomenon such as surface flashover, grain to grain arcing, or resistance heating resulting from induced currents in an antenna. These mechanisms require relatively intense RF radiation to produce detonation.

This project was designed to determine if any alternate coupling schemes exist whereby internal molecular mechanisms could be exploited to initiate detonation at more modest microwave power flux levels.

Hence, the objective of these tests was to determine if there exist specific frequencies in the microwave region where significant resonant absorption takes place in certain exothermic materials, and then to see if HPM radiation could be used to detonate those compounds. Further, one wanted a determination of the power flux and energy fluence levels required for initiation of detonation or deflagration.

This report documents the methods and procedures which were to be used to characterize the microwave coupling into the various explosives and propellants.

The general approach used consisted of two parts - a low power test phase and a high power test phase.

In the low power test, a sample of the explosive material was placed in a section of waveguide and radiated with very low power microwave radiation across a broad range of frequencies. A sample of each material was exposed to CW microwave energy from 2.5 to 18 GHz and then to determine the amount of incident energy absorbed in the sample leading to the determination of the microwave absorption coefficient as a function of frequency..

The planned approach for the high power test was to subject the exothermic sample to high power density levels of microwave energy at the frequency nearest the maximum absorption frequency that could be obtained with the equipment available and to determine at what power flux level the sample explodes of burns. In these tests, a sample of exothermic material was to be placed on a test stand, to simulate "free space", and exposed to direct radiation from a high power microwave source and the results observed. Two samples of each material were to be subjected to the HPM radiation so that a meaningful data base could be compiled.

The compounds which were tested under this contract are listed in table 1 along with the material source. The list is made up of materials which range from relatively stable compounds (such as ammonium nitrate) to extremely sensitive compounds (such as lead azide).

2. LOW POWER TESTS

The low power tests were carried out in the following manner. Due to the chemically unstable nature of some of the compounds and for ease of handling, the exothermic materials numbered 1 through 8 in table 1 were contained within a specially constructed cell. This sample holder consisted of a short piece (0.25 cm) of double-ridge waveguide onto which a mylar window was glued. The exothermic material was then inserted into the pocket formed from the mylar and waveguide shim and a cover window then glued onto the flange to form a sealed cell as depicted in Figure 1. A special cement, Loctite 404, was used because of its compatibility with the exothermic materials.

Table 1. Compounds Tested

	Compound	Source
1	Lead Azide	Broco Inc, Rialto CA
2	Lead Styphnate	"
3	PETN	"
4	Comp B	"
5	Black Powder	"
6	Nitrocellulose (12.6% Nitration)	"
7	Boron Barium Chromate	ICI America, Valley Forge, PA
8	M30 (Gun Propellent)	Radford Army Ammunition Plant, Radford, VA
9	JP4	General Dynamics
10	Diesel #2	"

The fuels, JP4 and diesel, were tested at General Dynamics' HPM facility in Rancho Cucamonga using double-ridge waveguide cells which were 12 inches long. The cell was sealed at one end with a mylar window and a smidgen of high vacuum grease applied to the waveguide flange wall. The cell was positioned so that the waveguide axis was vertical so that each of the fuels was able to be poured into the cell to the correct level and the waveguide then closed. A second mylar window was not required in these two cases.

For each material two waveguide cells were needed for testing over the frequency range from 2.5 to 18 GHz. One cell was constructed using WRD250 double-ridge waveguide and was used to measure absorption as a function of frequency in the range from 2.5 to 7.5 GHz. The other cell was made from WRD750 double-ridge waveguide and was used in the range from 7.5 to 18 GHz.

3.0 TEST FACILITIES

The low power exothermic susceptibility tests were conducted at General Dynamics' test facility at building 800 in Rancho Cucamonga, CA and at the explosive storage area of Broco, Inc. in Rialto, CA. All the exothermic compounds were tested at Broco, Inc. except for the fuels, JP4 and diesel, which were tested at General Dynamics

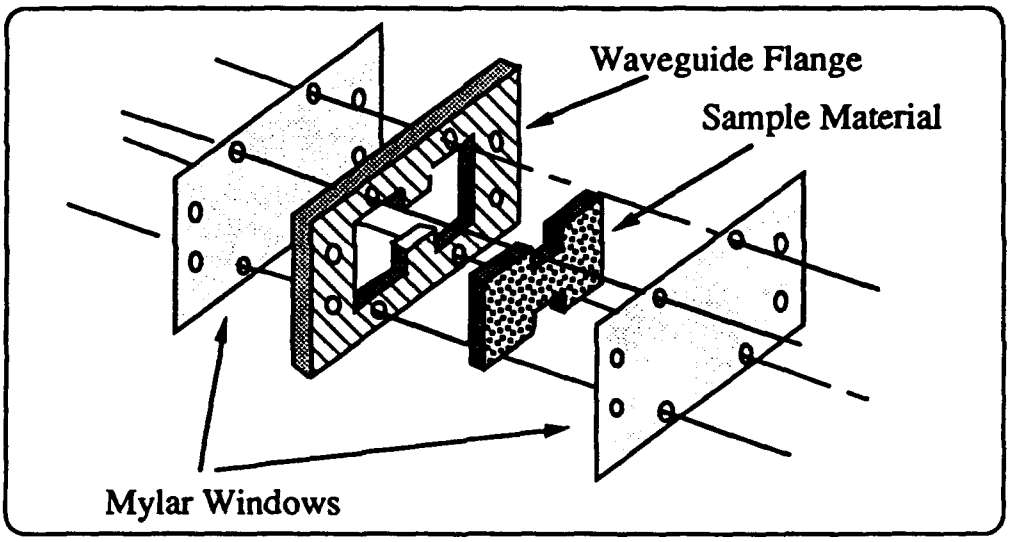

Figure 1 Low Power Sample Holder

3.1 GENERAL DYNAMICS HPM LAB

The low power calibration testing and the fuel testing was conducted in the general laboratory area of building 800. Although General Dynamics has a large shielded enclosure which provides a minimum of 100 dB of shield effectiveness over the full RF frequency spectrum at the Building 800 facility, it was not required for these tests since all the microwave radiation was contained within the double-ridge waveguide.

3.2 BROCO, INC. STORAGE AREA

The Broco explosive storage area is located about 10 miles northeast of building 800 in Rialto, CA. The facility is used as a storage area for commercial customers and also has a large open area that was used for the low power tests.

4.0 LOW POWER TEST PROCEDURE

As part of the pretest phase, each sample was weighed and identified for later analysis. The results are summarized in Table 2. All test equipment was assembled at this point and proper operation verified through the determination of the microwave absorption coefficient for liquid water. The results are found in the calibration section.

The low power test setup as shown in Figure 2, consists of a microwave synthesizer which acts as the microwave source generator and two dual port power meters use in the power level determinations. The incident and reflected power levels were measured along with the power level transmitted through the test sample. The power absorbed by the test sample can be calculated from this information.

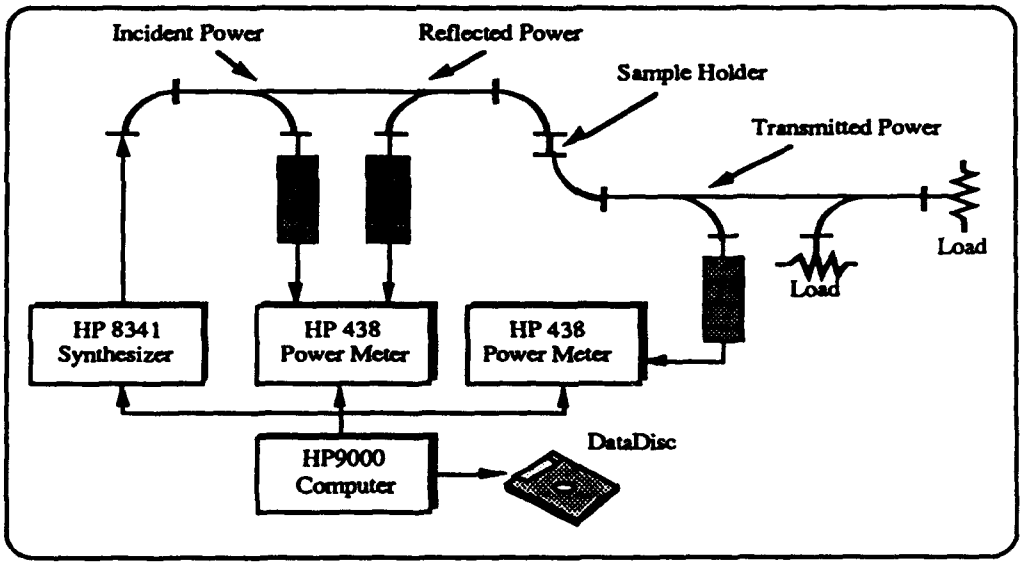

Figure 2. Low Power CW Test Layout

For material characterization the low power CW test procedure consisted of running a test with an empty sample holder in the waveguide. This provided absorption values for the empty holder, which were used later in the data analysis. Then the sample holder containing the exothermic material was placed in the waveguide instead of the empty holder. The automatic computer program was initiated and ran the test on the exothermic material sample.

4.1 INSTRUMENTATION AND DATA ACQUISITION

The test instrumentation employed in the low power testing consists of a microwave synthesizer, power meters, various power sensors, and a HP Integral Personal Computer for equipment control. This equipment enabled power measurements to be taken across the microwave bands from 2.5 to 18 GHz in two stages, from 2.5 to 7.5 GHz and, after switching to the high frequency set, from 7.5 to 18 GHz.

4.2 DATA RECORDED AND PRELIMINARY ANALYSIS

The equipment, under the control of the computer, sent an rf signal through the waveguide bidirectional coupler where the incident and reflected powers were measured, through the sample holder cell and then into a second bidirectional coupler where the transmitted power was measured. The computer recorded the frequency of RF radiation, the incident power, P_i, the power transmitted through the sample, P_t, and the power reflected from the sample, P_r. The frequency of the synthesizer is stable to within a few Hertz.[1] The power determinations were made using correction tables for the frequency dependence of the power heads and for the coupling of the directional coupler. The computer then calculated the following coefficients:

$$T = \text{Transmission Coefficient} = P_t/P_i \quad (1)$$

$$R = \text{Reflection Coefficient} = P_r/P_i, \quad (2)$$

$$A = \text{Absorption Coefficient} = 1 - T - R \quad (3)$$

The final computation is that of the insertion loss, I_L, defined as follows:

$$I_L = 10 * \log_{10} [P_t/(P_i - P_r)]$$

$$= 10 * \log_{10}[T/(1-R)] \quad (4)$$

This data is then stored internally for each frequency in the range.

At this point it is convenient to define the net incident power as $P_n = P_i - P_r$.

4.3 DATA OUTPUT

The data is output onto paper for a hard copy and also onto a Hewlett Packard Integral Personal Computer (IPC) diskette for more permanent storage and easy retrieval. The data stored on the magnetic diskette was used for further analysis.

Note that to go from 2.5 to 18 GHz two sweeps are required using two different double-ridged waveguide apparatus; one from 2.5 to 7.5 GHz and the other from 7.5 to 18 GHz. The frequency step in the low frequency band was 100 MHz while the frequency step in the high frequency was taken as 250 MHz. These frequency steps, although essentially arbitrary, were chosen so as to give a reasonable number of intermediate points.

In the magnetic diskette file, the first four lines were used for parameter information such as run number, date of the test, type of material tested etc. As the tests evolved the the material sample thickness, z, was also added into the header information. Beginning the fifth line the following quantities were output to the magnetic file: the frequency in GHz, P_i in dBm, P_t in dBm, P_r in dBm, I_L in dB, A, T and R.

4.4 FINAL ANALYSIS

The data stored on the Hewlett Packard IPC diskette was then transferred to a Hewlett Packard 9000 where the low and high frequency files were combined into a single file. The header lines on the second, high frequency file were truncated.

The determination of the absorption coefficient was made in the following manner. The absorption coefficient, α, is related to the transmitted and net incident power, P_n, by the relation

$$P_t = P_n e^{-\alpha z} = (P_i - P_r) e^{-\alpha z}. \qquad (5)$$

Hence

$$\alpha = -1/z * \ln[P_t/(P_i - P_r)]$$

$$= -1/z * \ln[T/(1-R)]. \qquad (6)$$

The absorption coefficient, α, includes the losses in the waveguide and in the windows which bound the material sample as well as those due to absorption by the materials itself. In order to correct for losses in the empty cell and waveguide one can calculate an empty cell absorption coefficient,

$$\alpha_{ec} = -1/z * \ln[T_{ec}/(1-R_{ec})] \qquad (7)$$

where the subscript ec denotes the values for the empty cell without any exothermic material present. Letting α_m be the absorption coefficient of the material excluding the losses associated with the sample holder, then the total transmitted power is simply

$$P_t = P_n \ e^{-\alpha_{ec} z} e^{-\alpha_m z} = P_n \ e^{-\alpha z} \qquad (8)$$

The absorption coefficient of the material, α_m, is found from the difference between the two measured coefficients. That is,

$$\alpha_m = \alpha - \alpha_{ec} =$$

$$= -1/z * \ln[T(1-R_{ec})/T_{ec}(1-R)] \qquad (8)$$

This is how the low power data was analyzed. The results of this analysis are presented in section 7.

5.0 HIGH POWER TESTS

The high power test were conducted during the the week of 1989 December 18-22 at Broco's explosive storage field in Rialto, California. Tests at S-, X and Ku-bands were planned. The Vitro System was to be used for the x- and Ku-band tests. The Vitro System consists of a control unit, a modulator unit, an X-band transmitter unit and a Ku-band transmitter. An operating system consists of the control unit, the modulator unit and either transmitter unit. Although this system was checked and operated in the laboratory just prior to being shipped to Broco, when it was set up in the field it failed to operate. This equipment failure prevented the X-and Ku-band high power tests.

Tests were conducted at S-band using the Cartpack source.

5.1 HIGH POWER TEST OBJECTIVES

The objectives of these tests were to see if high power microwave (HPM) radiation could be used to detonate exothermic compound samples and to determinate the power flux and energy fluence levels required for initiation. This test was to determine and characterize the vulnerability of various explosives and propellants to direct initiation from high power microwave radiation.

5.2 APPROACH

In these high power tests, a sample of explosive material was to be placed on a test stand, to simulate "free space", and

exposed to direct radiation from high power microwave sources until initiation occurs or a specified HPM radiation dose level had been reached. Two samples of each material were to be subjected to the HPM radiation so that a meaningful data base could be compiled.

5.3 TEST MATERIAL SAMPLE HOLDER

Due to the chemically unstable nature of some of the test samples and for ease of handling, the samples were contained within a sample holder. The sample holder designed for the high power tests is depicted in Figure 3. No metal parts of any kind were used in the high power test sample holder. This assured that no arcing occurred near the sample material. Also, using this holder prevented any of the explosive material from coming into contact with the handler or the environment. The holder is made from a teflon block which is 2" by 1" by 1/4". The holder was constructed of a teflon since it was thought to be transparent to microwaves, however, testing showed that, indeed, some microwave absorption takes place. See the calibration section of this report.

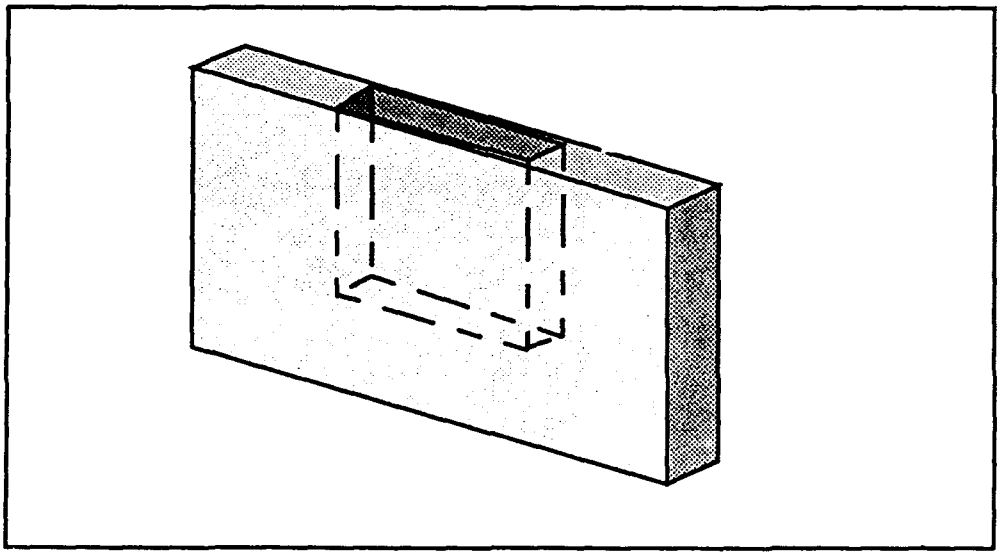

Figure 3 High Power Sample Holder

5.4 PORTABLE SHIELDED ENCLOSURE

A portable test chamber was constructed for the high power tests. The chamber is 16 feet long, 4 feet wide, and 4 feet high. The walls of the test chamber are covered by aluminum foil on the inside and fine mesh metal screening on the outside so as to provide sufficient RF shielding to prevent the escape microwave radiation. Absorbing material was placed inside the chamber in order to reduce internal reflections.

5.5 HIGH POWER TEST SETUP

The general test layout for the high power pulsed testing is depicted in Figure 4. The antenna used for the S-band tests was a 2x2 pyramidal horn array. Focusing horn lenses were available in both X and Ku-bands.

5.6 INSTRUMENTATION

Pulsed power measurements require the use of detectors and oscilloscopes (rather then CW power meters) for measuring peak power levels. Forward power inside the waveguide was to be measured using a microwave crystal detector and an oscilloscope, a standard technique for power and pulse width determination.

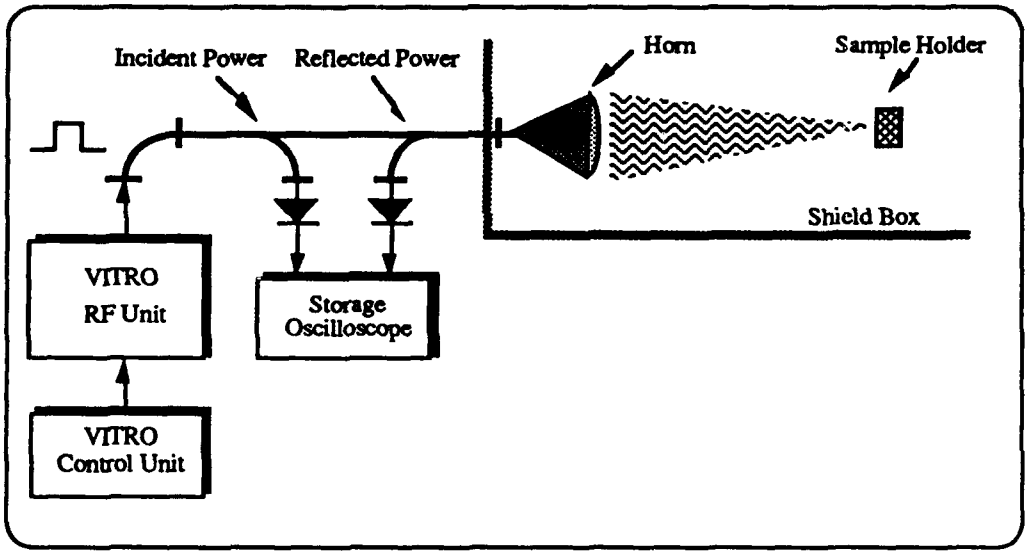

Figure 4 High Power Test Layout

5.7 HIGH POWER TEST PROCEDURE

The test objective is to determine the amount of microwave energy required by a given amount of exothermic material to cause the exothermic material to explode.

Initially an empty teflon sample holder was placed at the focus of the microwave horn lens. The temperature of this holder was recorded. Then this holder was irradiated with HPM radiation at the maximum power level and at the maximum pulse repetition frequency the source was able to generate for a period of one

minute. Then its final temperature was recorded. The teflon showed no significant temperature rise.

The next step in the high power pulse test sequence placed a sample holder containing one of the exothermic materials at the focal point of the microwave antenna. The sample was radiated with microwaves at maximum source power until detonation of the sample or until one minute has elapsed. The maximum flux density, the maximum pulse repetition frequency and total incident energy were recorded. this procedure was then repeated for a second sample of each energetic material.

5.8 FAILURE CLASSIFICATION

This was to be a GO / NO GO type test since the outcome was to be either a detonation/deflagration of the explosive material or no detonation/deflagration. Material detonation/deflagration was to be referred to as a susceptibility.

6. CALIBRATION

Liquid water was used as the material with which the capabilities of the device were to checked. Figure 5 is a plot of the liquid water absorption coefficient versus frequency for a liquid water sample 1.0 cm thick. As a further check on the accuracy and utility of this device a teflon sample 1.91 cm (0.75 inches) thick was measured. The results of that determination are also plotted in Figure 5. In addition, included on that figure are absorption coefficients for JP4 and Diesel fuel #2. Figure 6 is a plot of the liquid water absorption coefficient versus frequency for a liquid water sample 0.25 cm thick.

Note that most of the measured water data on the first graph, Figure 5, falls between the absorption coefficient values found in the open literature. The only points which do not fall within the published data range are the ones where the frequency is above 15 GHz. This can be understood as a result of the interaction of the microwaves with the liquid water - causing heating of the small sample. Note also that as the temperature goes up the liquid water absorption coefficient goes down.

In the second graph, Figure 6, the measured data does not fall within the published range, but in general lies just above the accepted values. The only exception occurs when the frequency is near the maximum of the apparatus where the data appears to agree with the published results. This agreement must be considered fortuitous in view of the results obtained in the previous plot. Nevertheless, the graph does clearly show the frequency dependence of the absorption coefficient. Hence one can conclude that the apparatus will measure significant absorption should it be present.

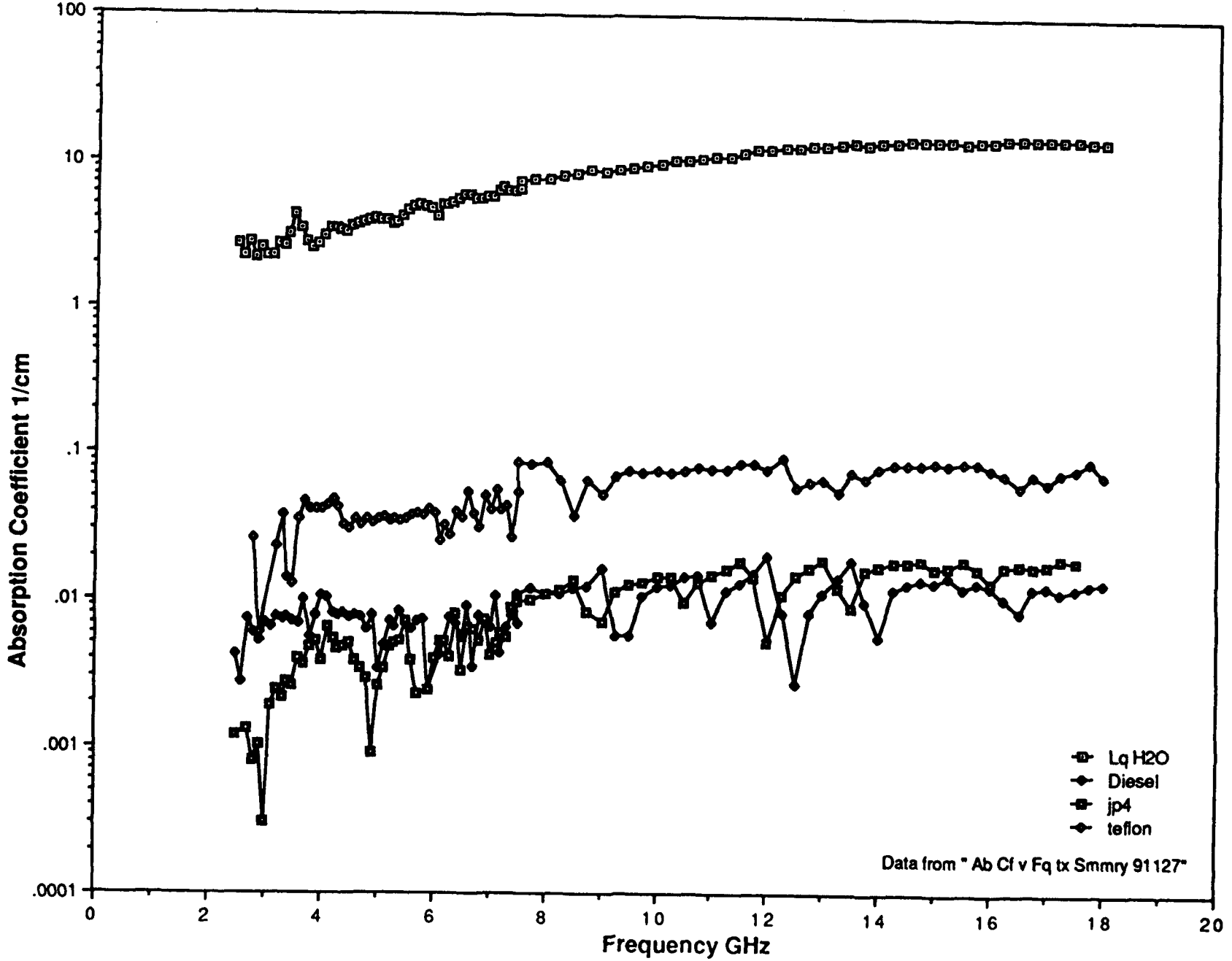

Figure 5. Absorption Coefficient versus Frequency

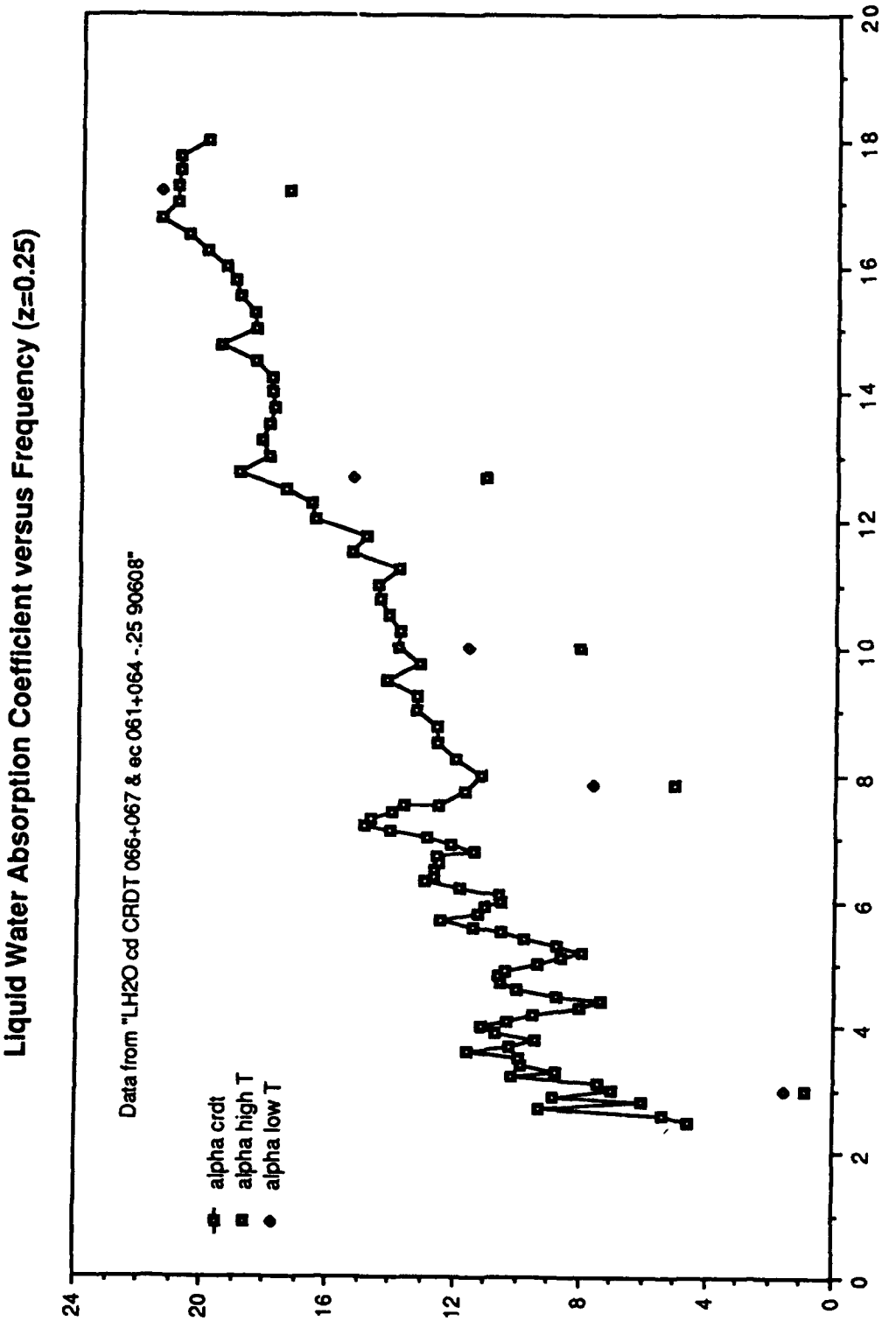
Liquid Water Absorption Coefficient versus Frequency (z=0.25)
Data from "LH2O cd CRDT 066+067 & ec 061+064 -.25 90608"
alpha crdt
alpha high T
alpha low T
Absorption Coefficient 1/cm
Frequency GHz
0
4
8
12
16
20
24
0
2
4
6
8
10
12
14
16
18
20

Figure 6.

7. EXPERIMENTAL RESULTS

A sample of each material was subjected to a low power frequency scan as well as high power S-band radiation. The results of those tests are as follows.

7.1 LOW POWER TEST RESULTS

Lead Azide

The results of the low power scan are presented in Figure 7. From the plot it is possible to say that the maximum value for the absorption coefficient is less than 3 cm^{-1}.

Lead Styphnate

The results of the low power scan are presented in Figure 8. From the plot it is possible to say that the maximum value for the absorption coefficient is less than 8 cm^{-1}.

PETN

The results of the low power scan are presented in Figure 9. From the plot it is possible to say that the maximum value for the absorption coefficient is less than 6 cm^{-1}.

Comp B

The results of the low power scan are presented in Figure 10. From the plot it is possible to say that the maximum value for the absorption coefficient is less than 6 cm^{-1}.

Black Powder

The results of the low power scan are presented in Figure 11. From the plot it is possible to say that the maximum value for the absorption coefficient is less than 4 cm^{-1}.

Nitrocellulose (12.6% Nitration)

The results of the low power scan are presented in Figure 12. From the plot it is possible to say that the maximum value for the absorption coefficient is less than 6 cm^{-1}.

Boron Barium Chromate

The Boron Barium Chromate sample was purchased from ICI America, in Valley Forge, Pennsylvania. The results of the low power scan are presented in Figure 13. From the plot it is

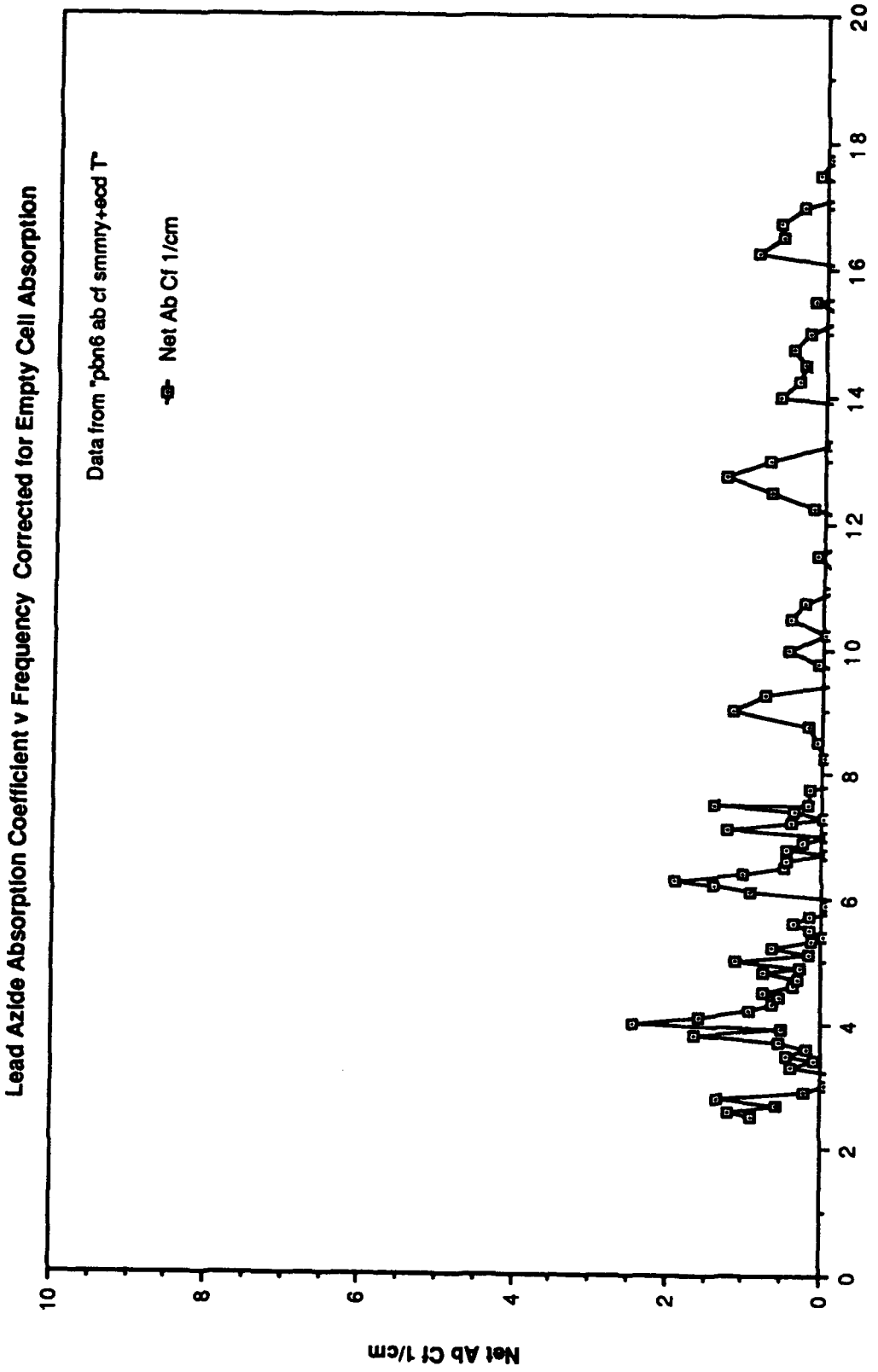
Lead Azide Absorption Coefficient v Frequency Corrected for Empty Cell Absorption
Data from "pbn6 ab cf smmry+eod T"
Net Ab Cf 1/cm
Net Ab Cf 1/cm
Frequency GHz
0
2
4
6
8
10
12
14
16
18
20

Figure 7.

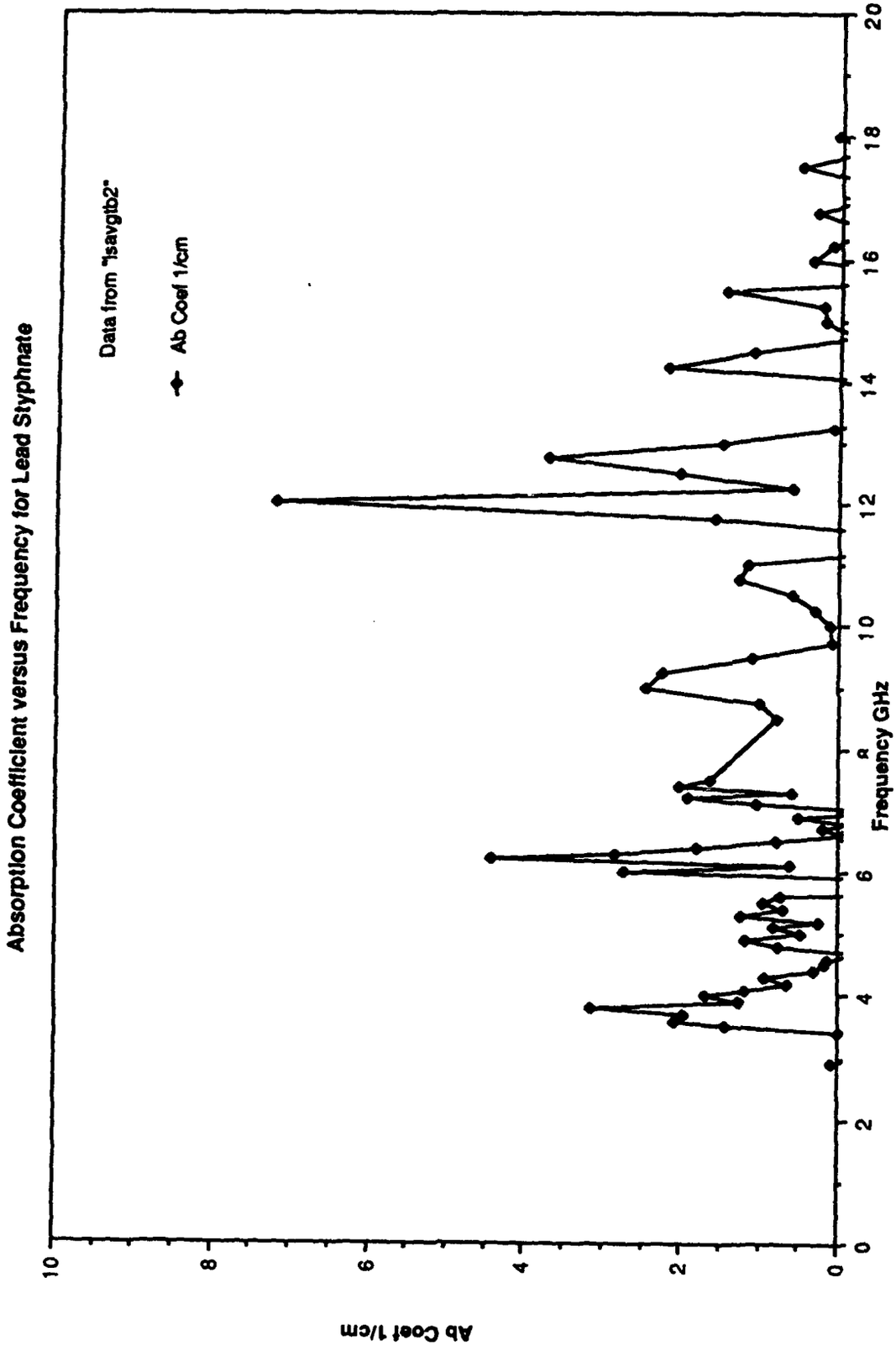
Absorption Coefficient versus Frequency for Lead Styphnate
Data from "lsavgtb2"
Ab Coef 1/cm
Ab Coef 1/cm
Frequency GHz
0
2
4
6
8
10
12
14
16
18
20

Figure 8.

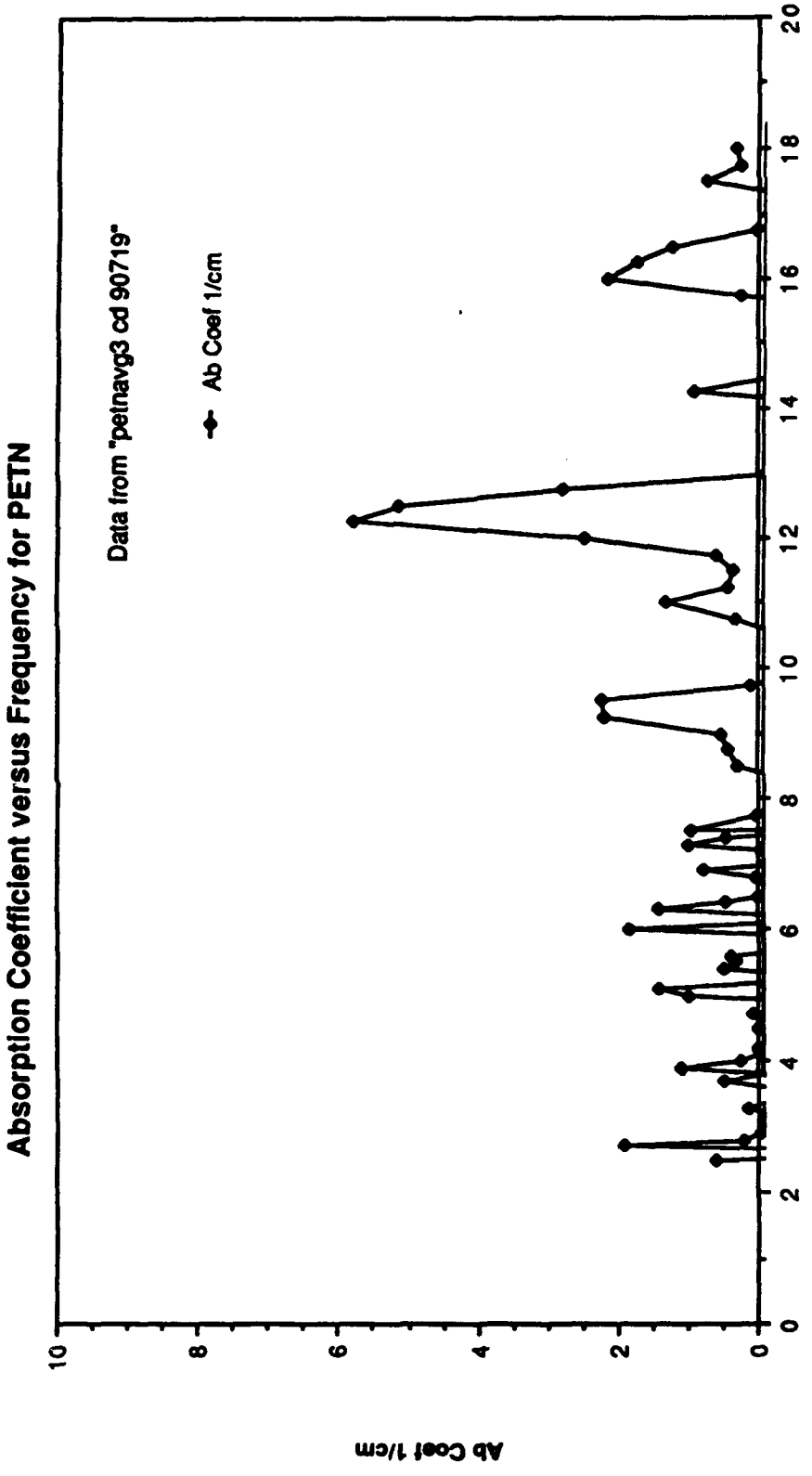
Absorption Coefficient versus Frequency for PETN
Data from "petnavg3 cd 90719"
Ab Coef 1/cm
Ab Coef 1/cm
Frequency
0
2
4
6
8
10
0
2
4
6
8
10
12
14
16
18
20

Figure 9.

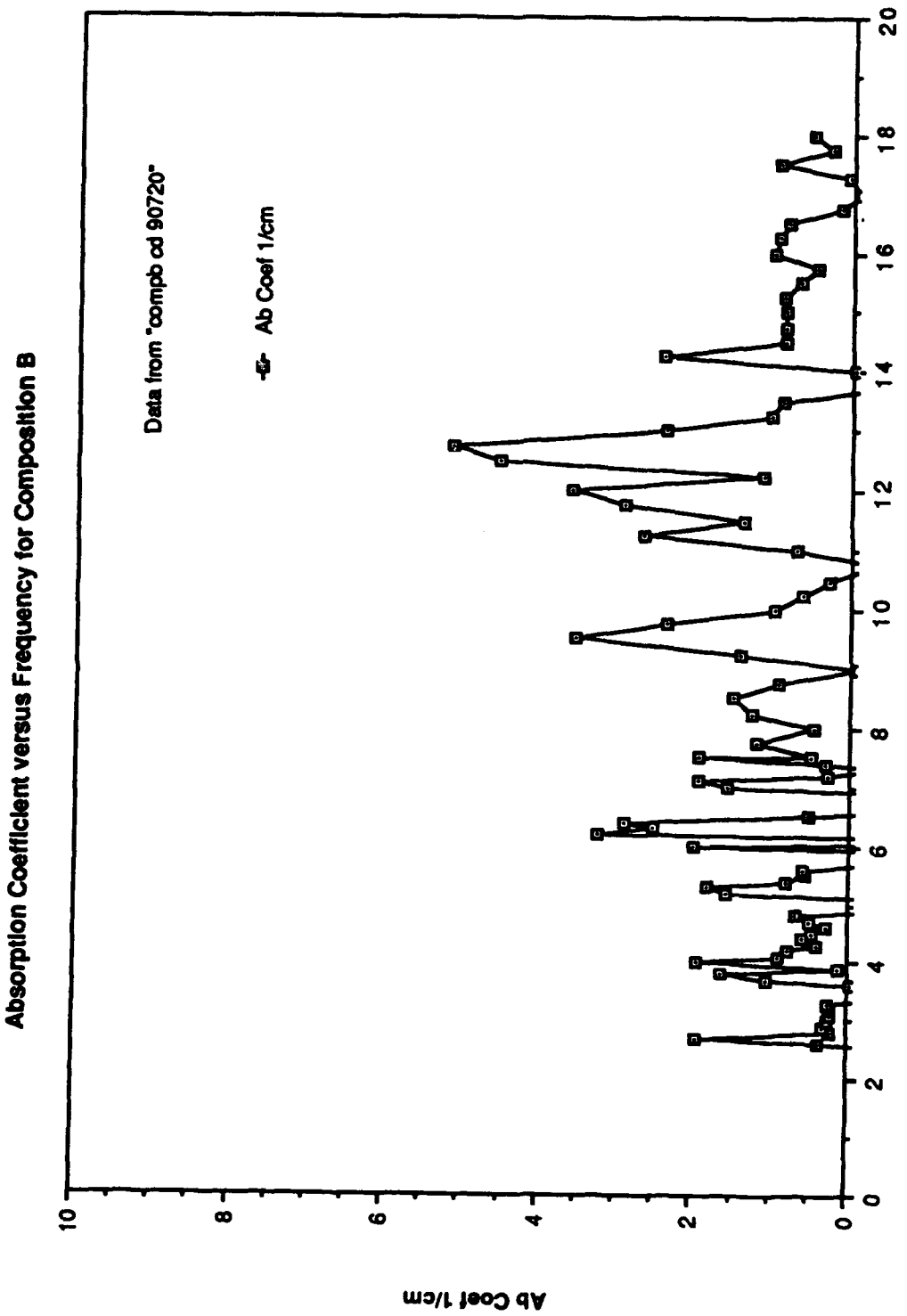
Absorption Coefficient versus Frequency for Composition B
Data from "compb od 90720"
Ab Coef 1/cm
Ab Coef 1/cm
Frequency GHz
0
2
4
6
8
10
0
2
4
6
8
10
12
14
16
18
20

Figure 10.

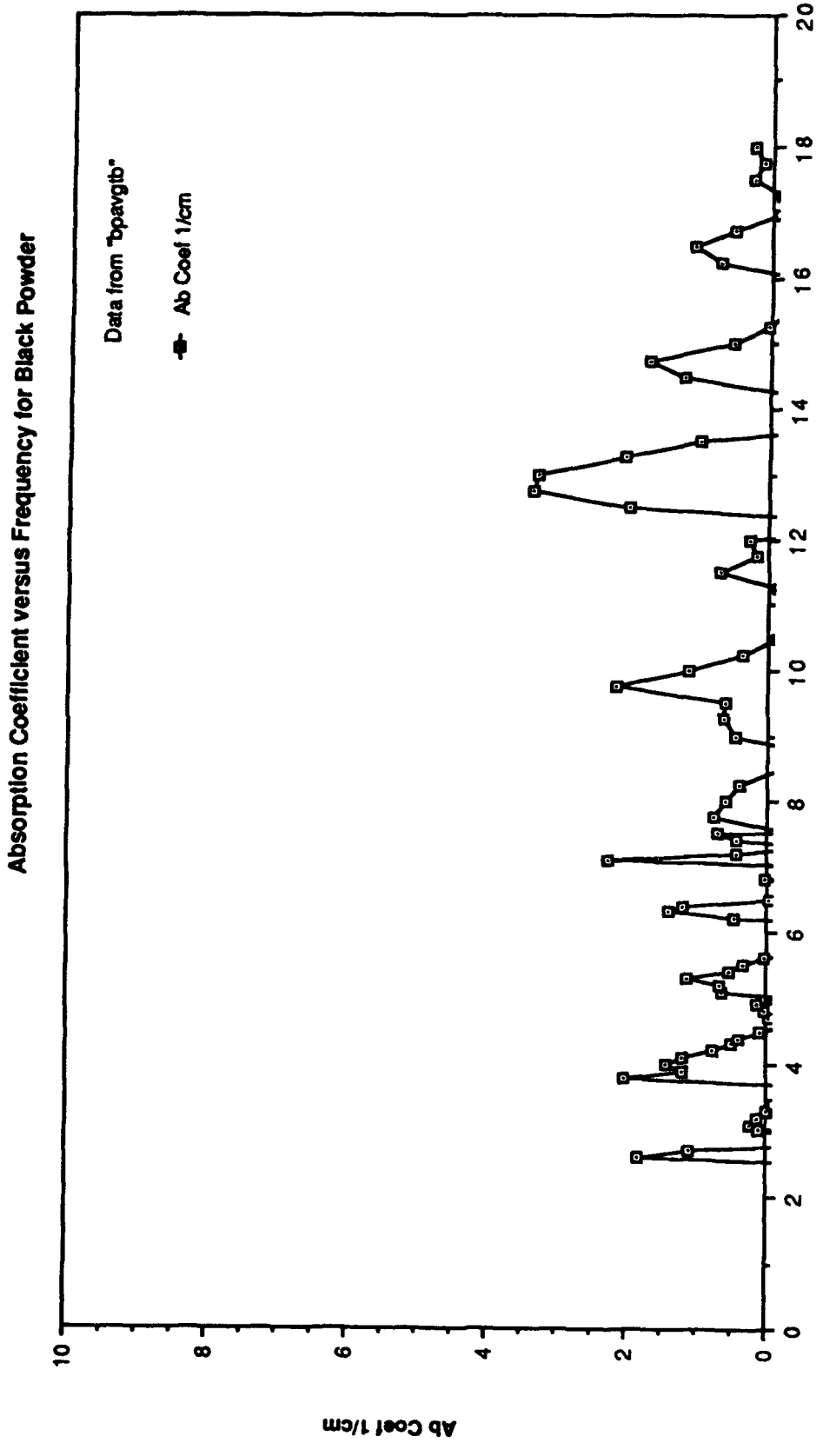
Absorption Coefficient versus Frequency for Black Powder
Data from "bpavgtb"
Ab Coef 1/cm
Ab Coef 1/cm
Frequency GHz
0
2
4
6
8
10
12
14
16
18
20

Figure 11.

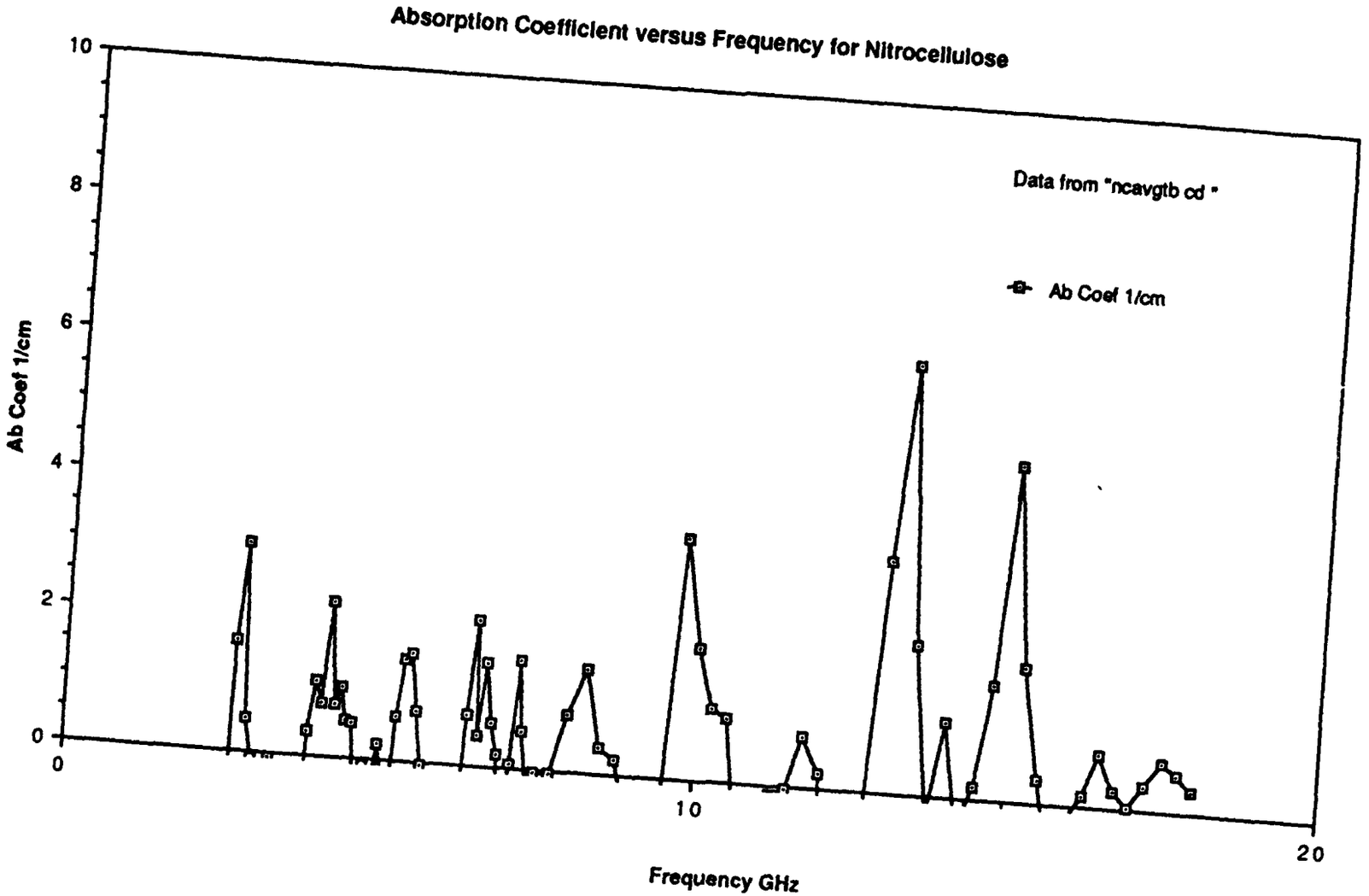
Absorption Coefficient versus Frequency for Nitrocellulose
Data from "ncavgtb cd "
Ab Coef 1/cm
Ab Coef 1/cm
10
8
6
4
2
0
0
10
20
Frequency GHz

Figure 12.

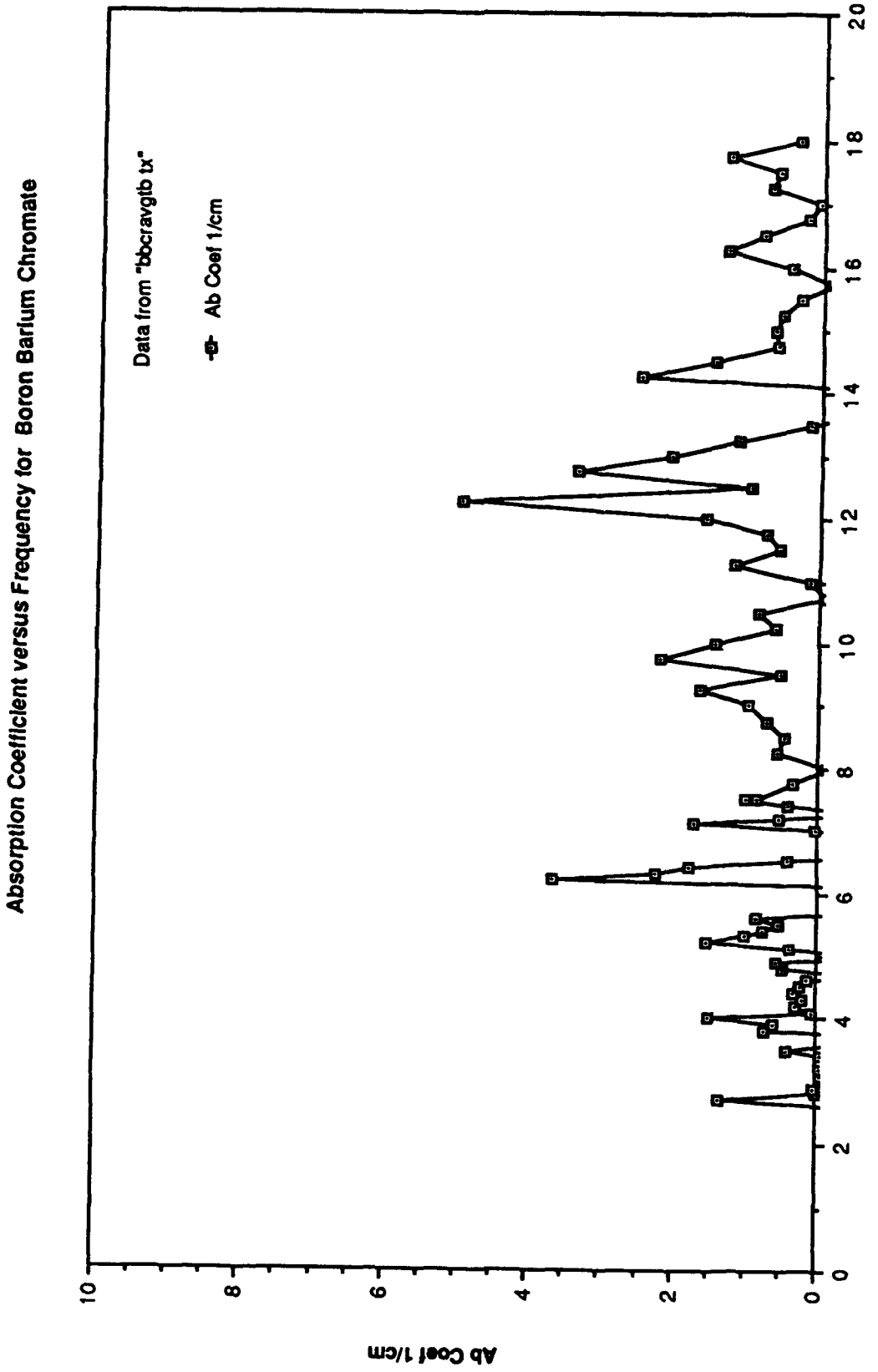
Absorption Coefficient versus Frequency for Boron Barium Chromate
Data from "bbcravgtb tx"
Ab Coef 1/cm
Ab Coef 1/cm
Frequency GHz
0
2
4
6
8
10
0
2
4
6
8
10
12
14
16
18
20

Figure 13.

possible to say that the maximum value for the absorption coefficient is less than 6 cm^{-1}.

M30 (Gun Propellent)

M30 (Gun Propellent) was obtained from the Radford Army Ammunition Plant in Radford, VA. The results of the low power scan are presented in Figure 14. From the plot it is possible to say that the maximum value for the absorption coefficient is less than 8 cm^{-1}.

JET-B (JP4)

The results of the low power scan are presented in Figure 5. From the plot it is possible to say that the maximum value for the absorption coefficient is less than 0.02 cm^{-1}.

Diesel #2

The results of the low power scan are presented in Figure 5. From the plot it is possible to say that the maximum value for the absorption coefficient is less than 0.02 cm^{-1}.

7.2 HIGH POWER TEST DATA

Table 3 summarizes the exposure levels achieved in the high power tests. No detonation, deflagration or decomposition of any of the samples was observed.

8. CONCLUSIONS

The plots of the measured data as a function of frequency for the exothermic materials: lead azide, lead styphnate, PETN, composition B, black powder, nitrocellulose, boron barium Chromate, and M-30 exhibit sharp peaks. Those peaks are most probably not due to internal resonant absorption, but rather are attributable to internal resonances in the measuring apparatus. These result from the discontinuities in the wave guide structure due to the dual windows on each cell required to protect the explosives from frictional forces. (When the windows are not present, the resonances disappear.) The data is useful, however, in establishing upper bounds on the magnitude of the absorption coefficients.

Although, in the case of JP4, some "noise" appears on the plot of absorption coefficient versus frequency the frequency dependence is clearly a rising function of frequency. For diesel fuel the absorption coefficient plot does exhibit some noise, however, the data appears to be valid, but does not exhibit any frequency dependence. The difference between the way the data

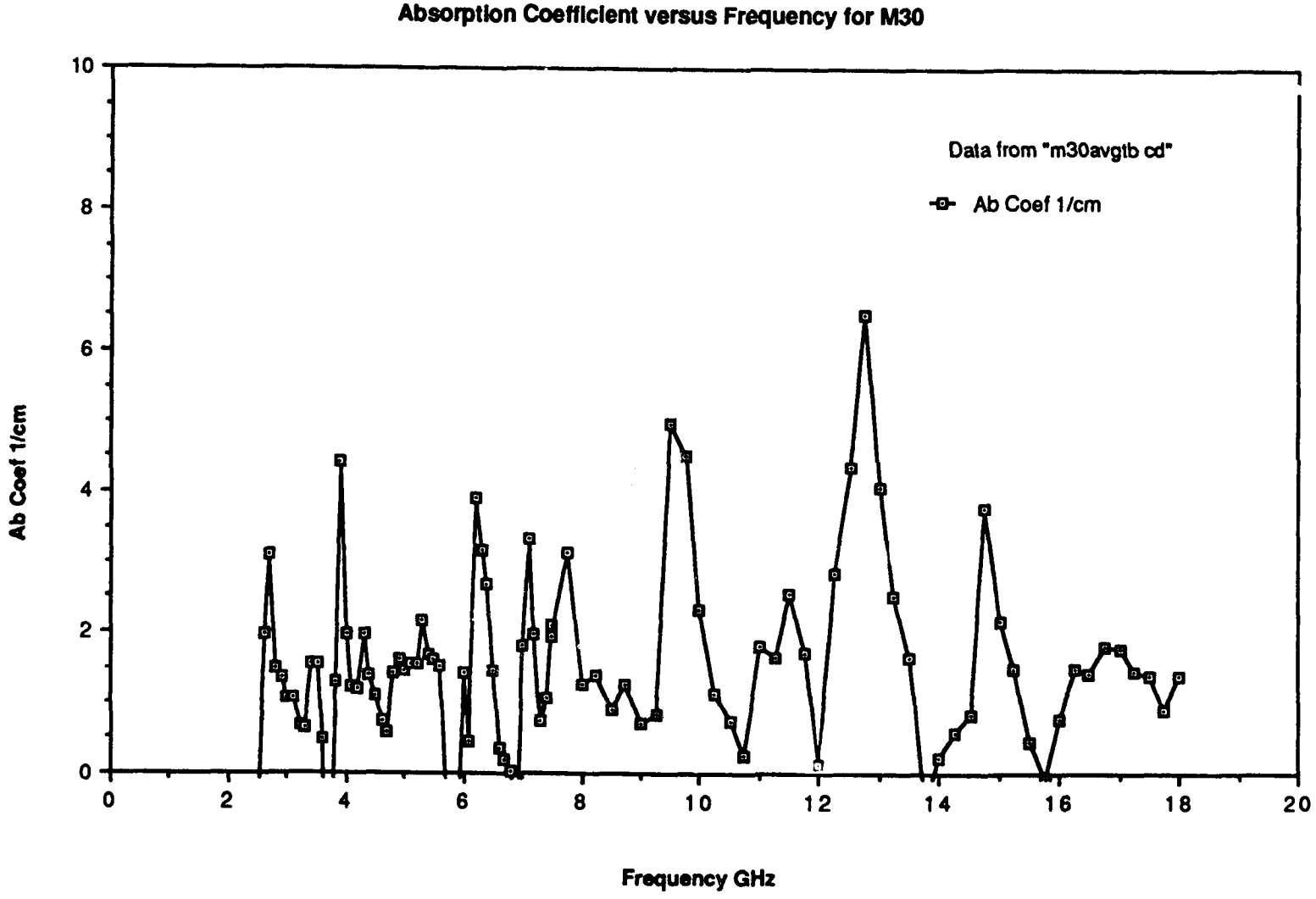
Absorption Coefficient versus Frequency for M30
Data from "m30avgtb cd"
Ab Coef 1/cm
Ab Coef 1/cm
Frequency GHz
0
2
4
6
8
10
12
14
16
18
20

Figure 14.

Table 3. High Power Test Data

Material	Sample #	Frequency GHz	Peak Power Transmitted MW	Power Density kW/sq cm	Pulse Duration µs	Energy Fluence mJ/sq cm	Pulse Repetition pps	Energy Total (Dose) Joules/sq cm
Lead Azide	7	2.88	2.09	1.13	6.4	7.22	50	21.7
	8	2.88	2.09	1.13	6.4	7.22	50	21.7
Lead stephnate	10	2.88	2.09	1.13	6.4	7.22	50	21.7
	9	2.88	2.09	1.13	6.4	7.22	50	21.7
PETN	11	2.88	2.09	1.13	6.4	7.22	50	21.7
	12	2.88	2.09	1.13	6.4	7.22	50	21.7
Composition B	41	2.88	2.09	1.13	6.4	7.22	50	21.7
	42	2.88	2.09	1.13	6.4	7.22	50	21.7
Black Powder	43	2.88	2.09	1.13	6.4	7.22	50	21.7
	44	2.88	2.09	1.13	6.4	7.22	50	21.7
Nitrocellulose	45	2.88	2.09	1.13	6.3	7.11	50	21.3
	46	2.88	2.09	1.13	6.3	7.11	50	21.3
M30	50	2.88	2.09	1.13	6.3	7.11	50	21.3
	49	2.88	2.09	1.13	6.3	7.11	50	21.3
Boron Barium Chromate	47	2.88	2.09	1.13	6.5	7.34	50	22.0
	48	2.88	2.09	1.13	6.5	7.34	50	22.0
JP4	1	2.88	2.09	1.13	6.5	7.34	50	22.0
	2	2.88	2.09	1.13	6.5	7.34	50	22.0
Diesel	3	2.88	2.09	1.13	6.5	7.34	50	22.0
	4	2.88	2.09	1.13	6.5	7.34	50	22.0
Empty Hoilder	0	2.88	2.09	1.13	6.5	7.34	50	22.0

was taken for diesel and JP4 is in the magnitude of the sample cell - 12 inch cells were used in these tests - and the fact that only one window was required for these tests rather than two as was required for the previously described measurements on the exothermic materials.

The high power tests indicated that at one specific frequency, 2.88 GHz, there was no susceptibility of any of the energetic materials. Since the low power tests indicated no specific, sharp frequency dependence, one can conclude that explosives, by themselves, are not susceptible to high power microwave radiation in the frequency range considered and provided the power density does not exceed 1 kW/cm^2.

In summary the objectives of this contract effort were achieved. From this investigation it is clear that there is no susceptibility of any of the energetic materials considered to high power microwave radiation in the range from 2.5 to 18 GHz at power densities less than 1 kW/cm^2.