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MEMORANDUM REPORT BRL-MR-4002

BRL

CENTIMETER WAVE ABSORPTION
IN MOLECULAR CRYSTALS

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1. INTRODUCTION

There have been many studies of the dielectric properties of materials at very high (i.e., optical) frequencies and at relatively low (megahertz or less) frequencies. Relatively few have been performed on materials at centimeter or millimeter wave frequencies (Megaw 1973; Von Hippel 1954). In this paper we report the results of centimeter wave absorption measurements performed from 2 to 20 GHz on several ionic molecular solids and two ionic salts from room temperature up to about 250° C. The ionic salts potassium bromide and sodium chloride were chosen for study because they are expected to show mainly high frequency ionic/electronic dielectric losses and to have no dipolar contribution to their dielectric polarizability (Kittel 1971; Blakemore 1969). The ionic molecular solids studied were potassium nitrate, potassium nitrite, sodium nitrate, and sodium nitrite. In the frequency range studied, most of the dielectric losses in these media are expected to be dipolar. In the nitrites, the losses are expected to be due to the permanent dipole moments of the nitrite ions (Megaw 1973; Jona and Shirane 1962) which are excited to "hopping modes" when the temperature is increased. In sodium nitrate, where there is no permanent dipole moment, the centimeter wave absorption is found to be similar to that seen in ionic salts. In potassium nitrate, the development of a dynamically induced dipole moment at elevated temperatures (Lu and Hardy 1990, 1991; Jona and Shirane 1962) leads to interesting absorption characteristics in this frequency regime. The theoretical section briefly discusses the mechanisms underlying the dielectric behavior of these classes of ionic molecular solids.

2. EXPERIMENT

Figure 1 shows a schematic diagram of the powder sample heater used in the reflectometer for the "loose" powder sample measurements. The salts to be measured were first ground to a fine powder with an analytical mill. Since some of these materials may absorb moisture from the air, the powders were then heated in a drying oven for 4–6 hours at 135° C. The sample material was then packed loosely in the powder sample heater tray to a thickness of about 1.1 cm (7/16 in) and the surface smoothed off. A thermocouple set into the base plate measured the temperature of the bottom of the powder sample while other thermocouples monitored the temperatures at different heights within the sample. Since the samples are relatively thick, variations in the temperatures from the bottom to the top of the

samples of 10° C or so are common. During the experiment, while the sample is being heated to higher temperatures, 20 to 30 minutes are required between temperature measurements in order to minimize the temperature differences throughout the sample. The reported temperature is an average of temperatures from TC1, TC2, and TC3 and was usually within a couple of degrees of TC3, the thermocouple located at 0.64 cm (1/4 in) above the base plate.

In the compressed potassium nitrate sample, the experiment is somewhat different. This sample was produced by pressing potassium nitrate powder in a 15.2-cm by 15.2-cm (6-in by 6-in) mold to a thickness of 8.1 mm with a heavy hydraulic (180 metric ton) press. This resulted in a hard, nearly fully dense tile of KNO₃ without the use of a binder. During reflection measurements, the sample was attached to a 0.64-cm (1/4-in) aluminum plate by thermally conducting paste and then placed directly on top of the 15.2-cm² (6-in by 6-in) top plate of the heater. Another 0.64-cm-thick (1/4-in) aluminum plate was then placed on top of the sample during the heating process. Thermocouples were located in the plates below and above the sample so the temperature could be monitored. When the temperatures of the plates above and below the KNO₃ tile sample were within about 5° C, the top plate was quickly removed and a reflection measurement was made. In this way, the sample temperature was more accurately known than in the loose powder samples.

The radar reflection measurements were made from 2 to 20 GHz on the Ballistic Research Laboratory (BRL) HP8510B Centimeter Wave Reflectometer as diagrammed in Figure 2. This reflectometer is in the form of an "arch" with the sample resting on a 30.5-cm (12-in) high pedestal surrounded by radar-absorbing material. The bistatic angle for these tests is 5°. For the loose powder samples, the powder sample heater assembly is first placed into the reflectometer without the powder and a baseline reflection measurement is made. The powder is then added to the powder sample tray and reflection measurements are made at various temperatures. Once the desired temperature is attained, the swept frequency measurement from 2 to 20 GHz takes about 45 seconds to complete. The powder samples are measured through several temperature cycles to ensure that the data are reproducible.

3. THEORY

The microwave dielectric behavior of the sodium and potassium nitrites and nitrates reported in this section provides substantial evidence of two generic loss mechanisms for ionic molecular solids.

In the nitrites, the charges making up the nitrite ion lie on the vertices of an isosceles triangle. This geometrical arrangement of charges produces a permanent dipole moment (Megaw 1973). Since it is known that in potassium nitrite the nitrite ions are dynamically disordered and undergo quasi-free rotation at 300 K and above, an external microwave field will couple directly to the dipole moment, drive the rotations, and thus experience loss (Adams, Pogson, and Sharma 1988). The temperature dependence of this loss will be weak. In the case of sodium nitrite, the nitrite ions are ordered well below 440 K and will show little microwave activity (Megaw 1973). However, as the temperature is raised towards 440 K, the nitrite ions begin thermally activated 180° hopping, and this motion again begins to couple strongly to the microwave field. At and above 440 K, the nitrite ions go collectively (Lu and Hardy 1992) into quasi-free rotations; and the behavior is qualitatively similar to that of potassium nitrite.

In the nitrates, the situation is more subtle. The nitrate ion, being an equilateral triangle, has no permanent dipole moment (Megaw 1973). As a consequence, when nitrate ions in sodium nitrate begin to rotate, they do not, to lowest order, couple to a microwave field. This is not only because they are nonpolar, but also because the sodium ions lie exactly between the centroids of the nitrate ion triangles, and thus (again to lowest order) are unaffected by the nitrate rotation.

Figure 3 shows a representation of the KNO_3 lattice. When potassium nitrate is in the room temperature phase (below about 117°C), the potassium ions lie off the nitrate ion centroid line as indicated by position 1 in the diagram (Megaw 1973; Lu and Hardy 1990, 1991). This loss of symmetry means that, should the nitrate ions become free to rotate, their corners (and centroids) will successively be displaced normal to the nitrate ion planes as each oxygen ion in turn is closest to the potassium ion. Since the potassium ions must, by Newton's third law, move in the opposite direction, a dipole moment which fluctuates

synchronously with the rotational motion results. Since the order/disorder transition in this system results from just this nitrate ion transition to quasi-free rotation, the observed microwave loss will rise steeply to a maximum at the transition, exactly as is observed. However, above the transition the potassium ions begin to move along the arrow towards position 2 and approach the nitrate ion centroid line. This movement causes an increase in the symmetry of charges about the potassium ion and thereby a reduction of the effective dipole moment and the mechanism which produces microwave losses in this material. Thus, the losses will begin to drop off above the temperature of the order/disorder transition.

4. RESULTS AND DISCUSSION

Figure 4 shows results of measurements on potassium nitrite from 46° C to 114° C. As expected from the above discussion, this material shows significant losses above room temperature and up to at least 114° C. The temperature variation of the loss is relatively weak with the losses slightly decreasing as the temperature is raised. This Debye-like temperature dependence is in agreement with what is expected for a polar solid of this type (Kittel 1971; Blakemore 1969). Figure 5 shows the reflectometer results for sodium nitrite from room temperature to above 150° C. Measurements taken from room temperature up to 70° C show no increase in absorption over the baseline measurement. At about 100° C, losses begin to take place and rapidly increase as the temperature is raised to above 150° C. This result is interesting since the temperature at which thermally activated hopping is expected to occur is around 440 K (167° C). This implies that some dipolar reorientation does occur below 440 K and that nitrite ion hindered rotations or "hopping" may be occurring over a range of temperatures up to 440 K.

The results of measurements on sodium nitrate are shown on Figure 6. In this case, the losses are small even as the sample is heated to about 240° C which is above the order/disorder transition temperature. This is expected because of the high symmetry of this system and the lack of the development of a dynamically induced dipolar loss mechanism. These results are similar to results for the ionic salts potassium bromide and sodium chloride shown in Figures 7 and 8, respectively. In each of these salts, the losses are very small at microwave frequencies as would be expected for salts containing no dipolar contributions to their dielectric polarizability (Kittel 1971; Blakemore 1969).

Figure 9 shows the results of measurements on a loose powder sample of potassium nitrate. Here the sample was heated from room temperature to 156° C. From room temperature to about 100° C, reflection measurements show no appreciable losses. At 117° C, however, the losses begin to occur with the absorption peak moving to lower frequencies as the temperature is increased. The loss mechanism seems to be weakening as the temperature is raised above 148° C, as is indicated in the figure by the 153° C and 156° C data. Such a result would be in alignment with the previous theoretical discussion which predicts the losses to drop off above the transition temperature of about 128° C.

Results of measurements on a nearly fully dense, 8.1-mm-thick, pressed powder sample of potassium nitrate are shown in Figures 10 and 11. As in the loose powder sample, the losses do not begin to occur until the temperature is raised above 100° C. Above this temperature, a rapid increase in the absorption peaks occur until the temperature is raised to 118° C. Above this temperature, the two large absorption peaks move to lower frequencies and become weaker. A weaker, low-frequency peak (below 5 GHz) continues to grow as the temperature is raised to above 136° C. The same behavior was seen in the loose powder potassium nitrate sample for a weak, low-frequency absorption peak. All the results for potassium nitrate are strong evidence for the development of a dipolar loss mechanism above about 100° C as discussed above. The weakening of the losses above the transition temperature can be explained by the movement of the potassium ions towards the centroid line joining the nitrate ions. This results in a decrease in the dynamically induced dipole moment and thus the mechanism for loss in this material.

5. SUMMARY

We have studied the temperature-dependent centimeter wave absorption characteristics of several inorganic salts. Some were ionic salts and some were ionic molecular crystals. The ionic salts behaved as expected, with very low losses at the temperatures measured. This is clearly due to the fact that a strong dipolar loss mechanism is not present in these materials. Several ionic molecular crystals were studied. The nitrites, which have a permanent dipole moment, showed absorption only at temperatures high enough to excite "hopping" modes. For potassium nitrite, losses were seen at all the temperatures measured and for sodium nitrite they were seen above about 100° C. The nitrates exhibited interesting behavior with

sodium nitrate showing very little loss at the temperatures measured and potassium nitrate showing strong temperature-dependent absorptions above about 105° C. The centimeter wave absorption observed in potassium nitrate near the order/disorder transition temperature is likely caused by a dynamically induced dipolar loss mechanism.

POWDER SAMPLE HEATER

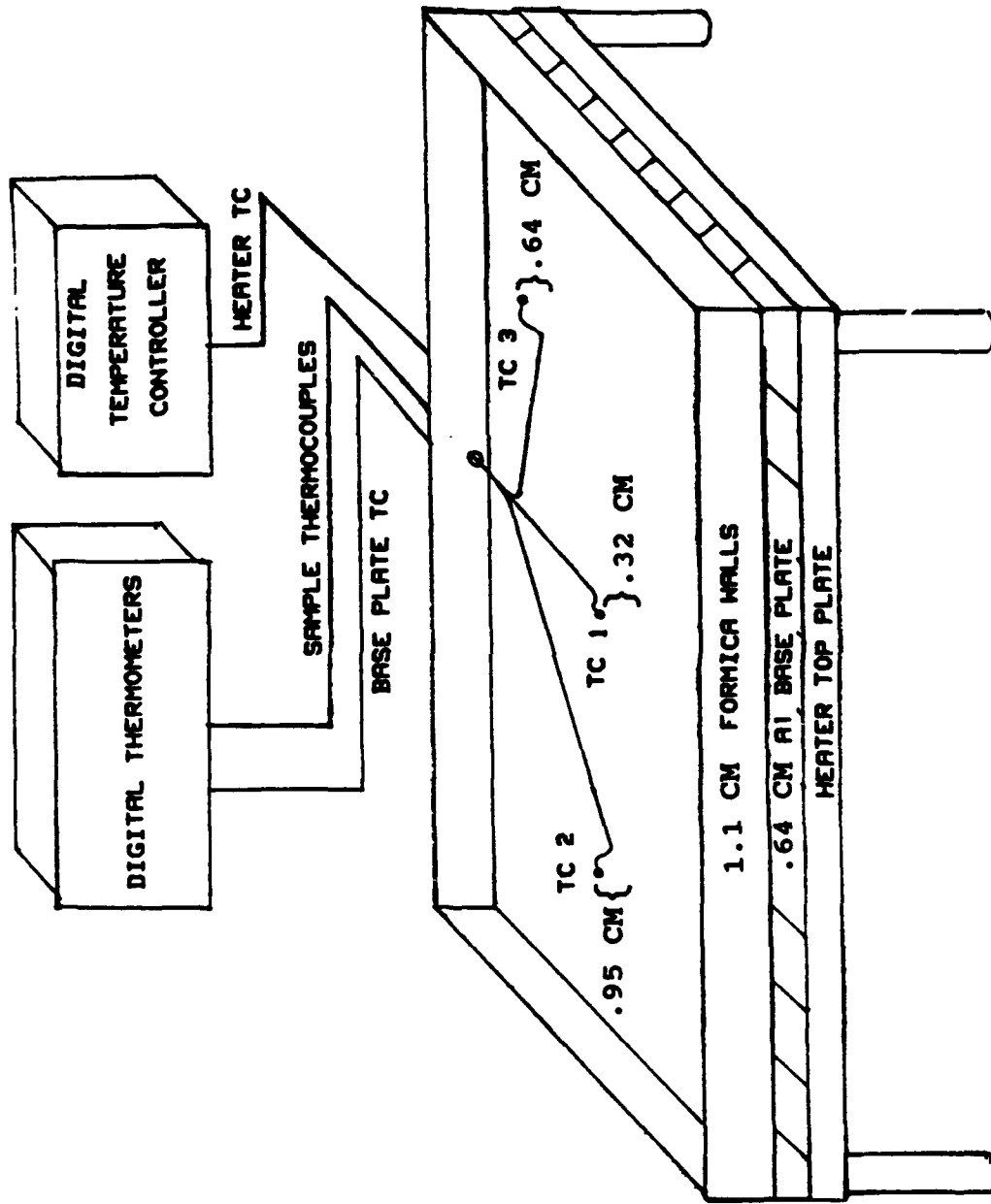


Figure 1. Digitally Controlled Powder Sample Heater.

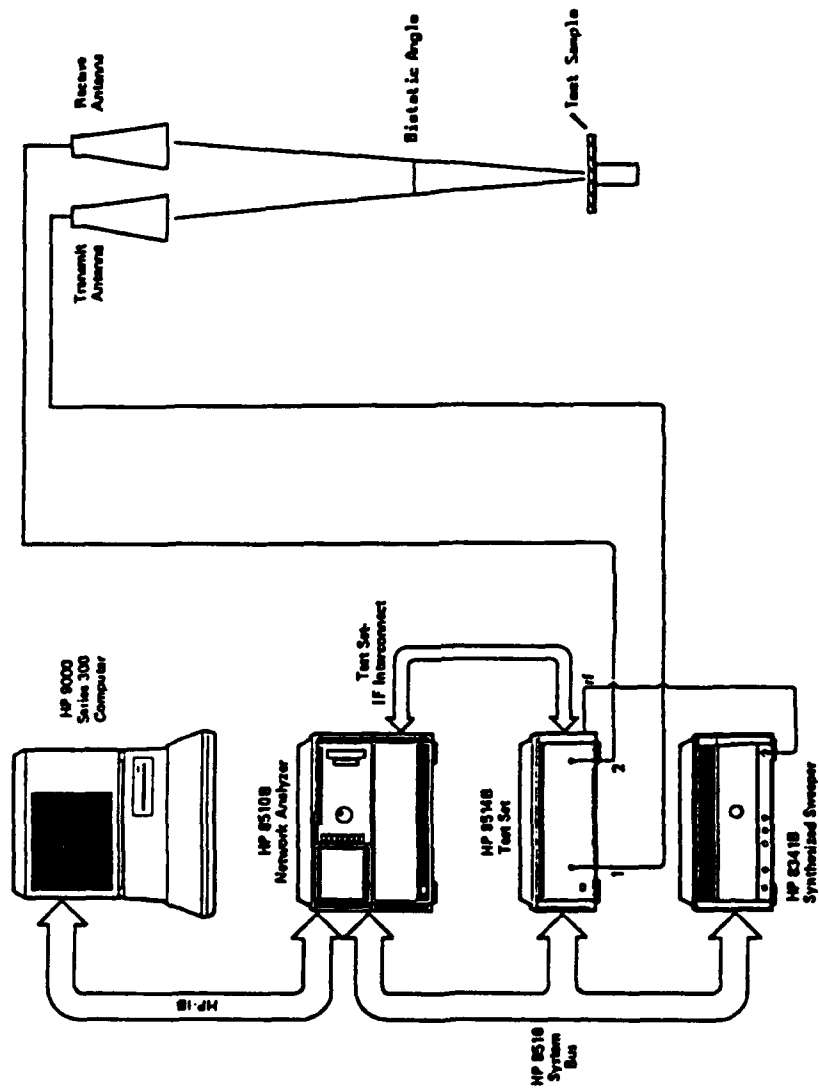


Figure 2. BRL HP8510B Centimeter Wave Reflectometer.

KNO₃ STRUCTURES

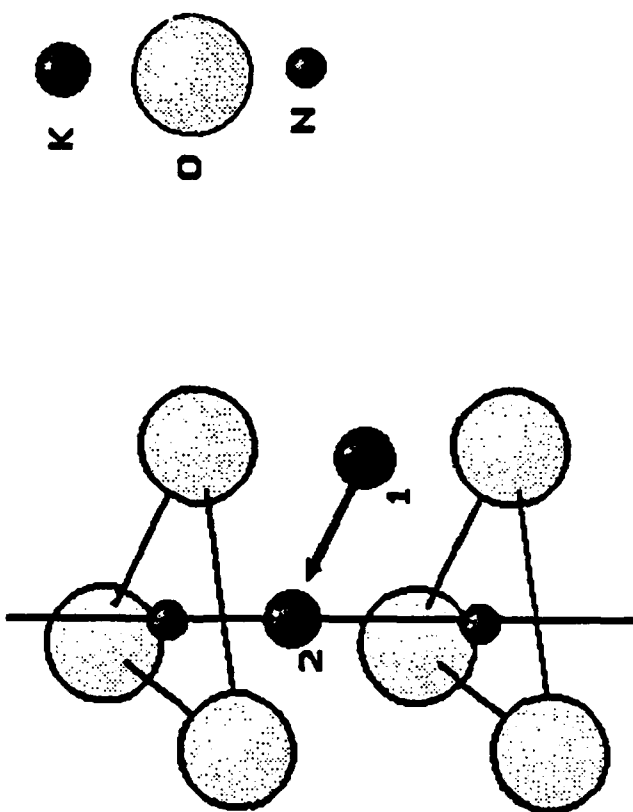


Figure 3. Diagram of KNO₃ Lattice.

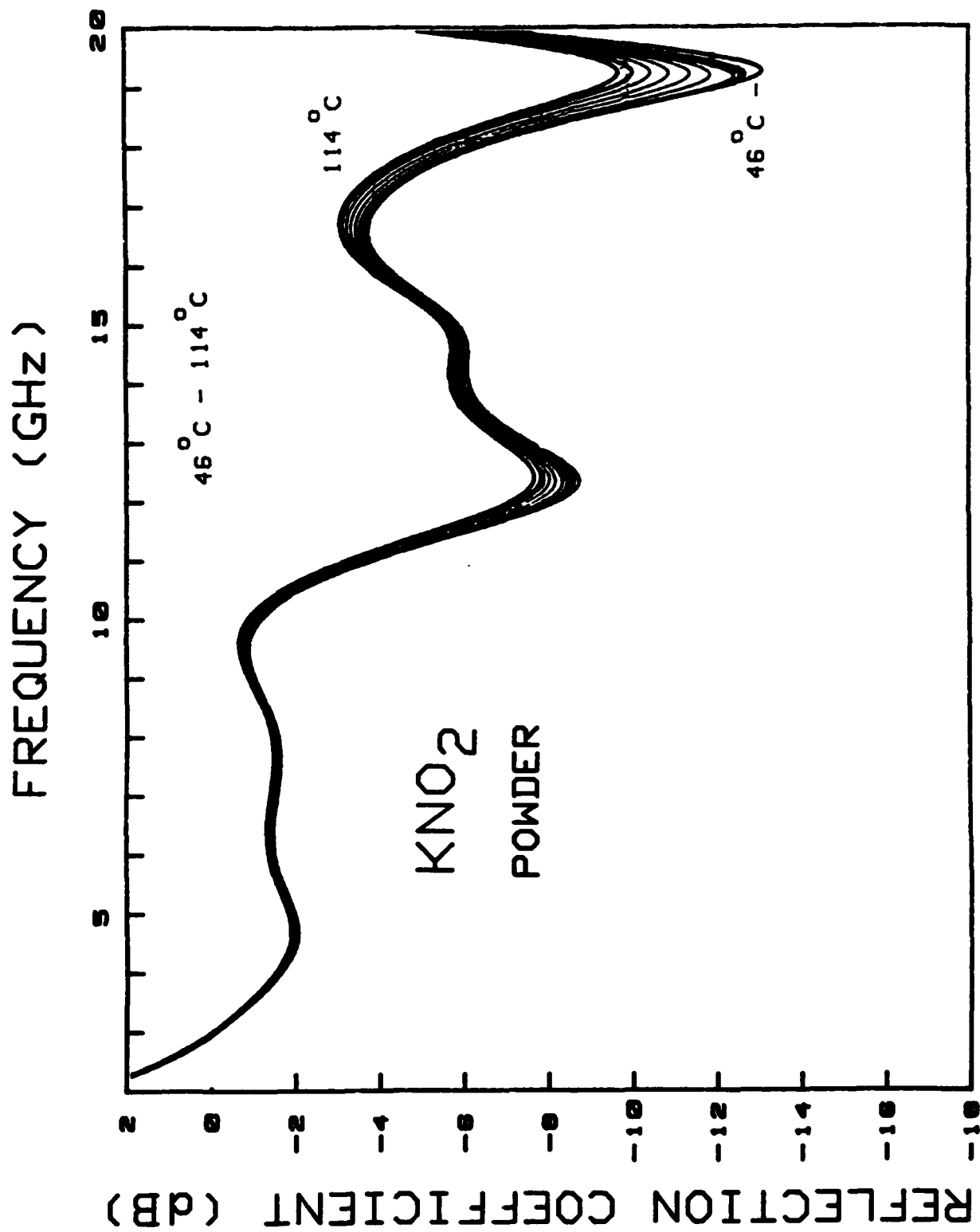


Figure 4. Microwave Absorption From 2 to 20 GHz From 46° C to 114° C for Potassium Nitrite Loose Powder.

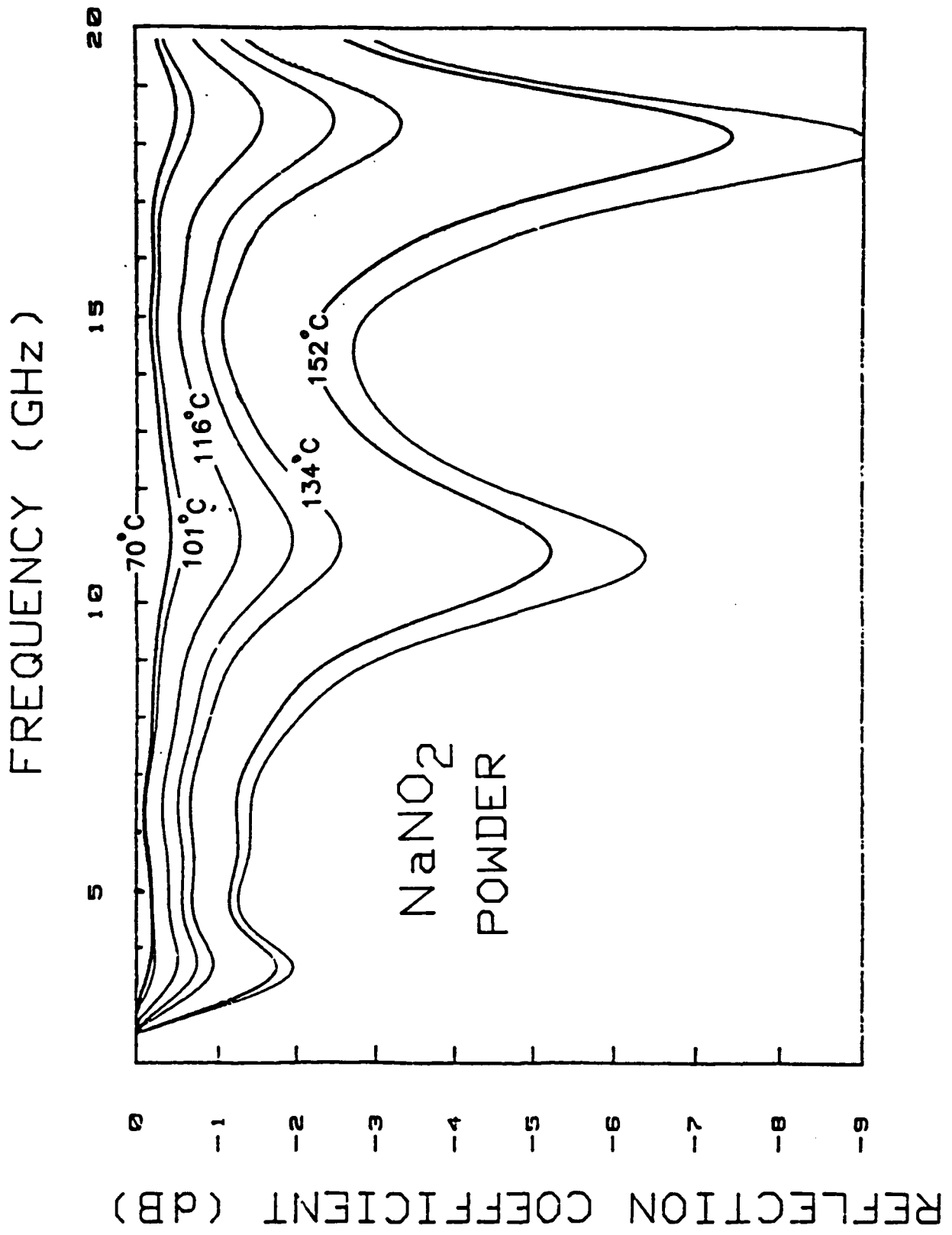


Figure 5. Temperature-Dependent Microwave Absorption for Sodium Nitrite Loose Powder.

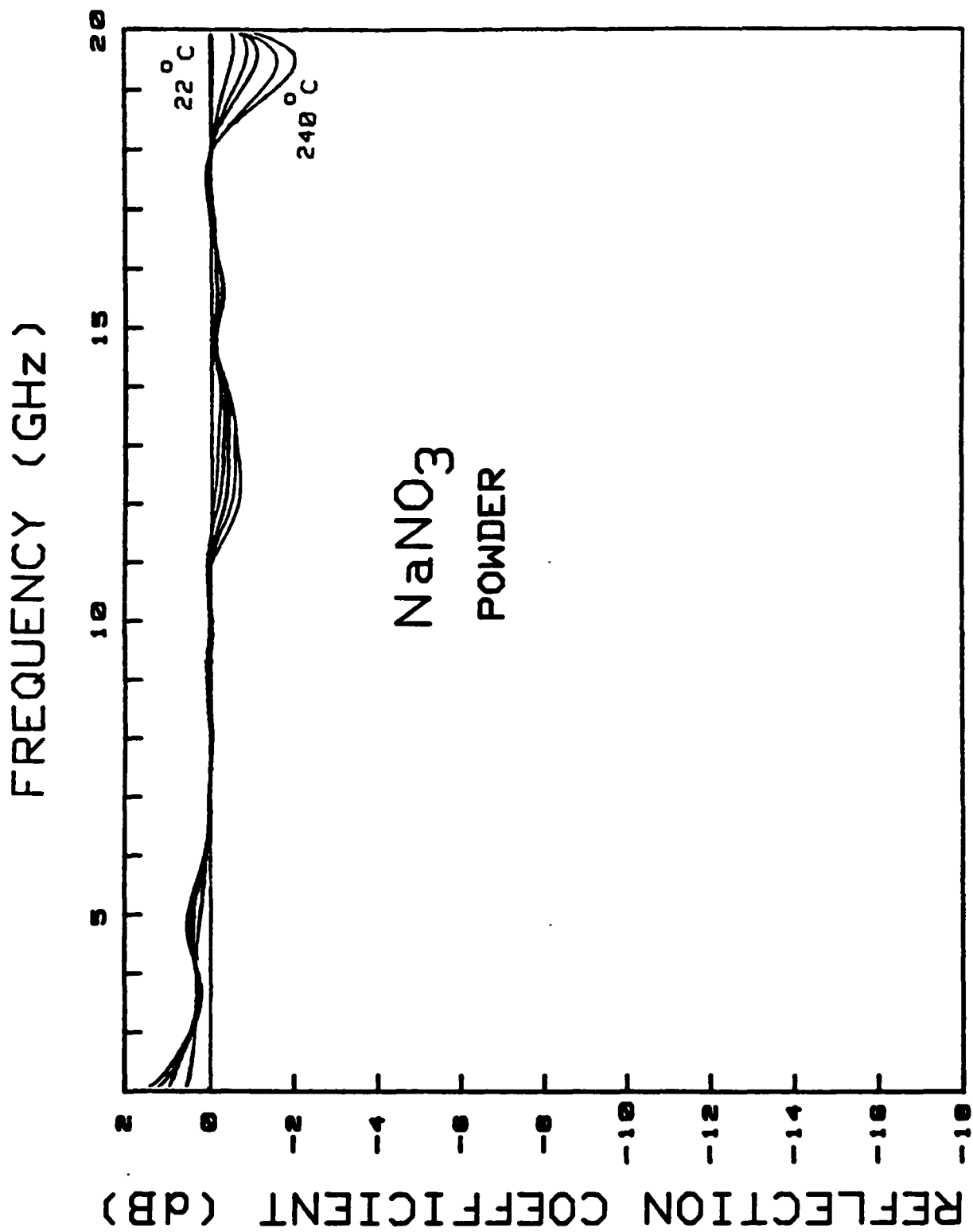


Figure 6. Temperature-Dependent Microwave Absorption for Sodium Nitrate Loose Powder.

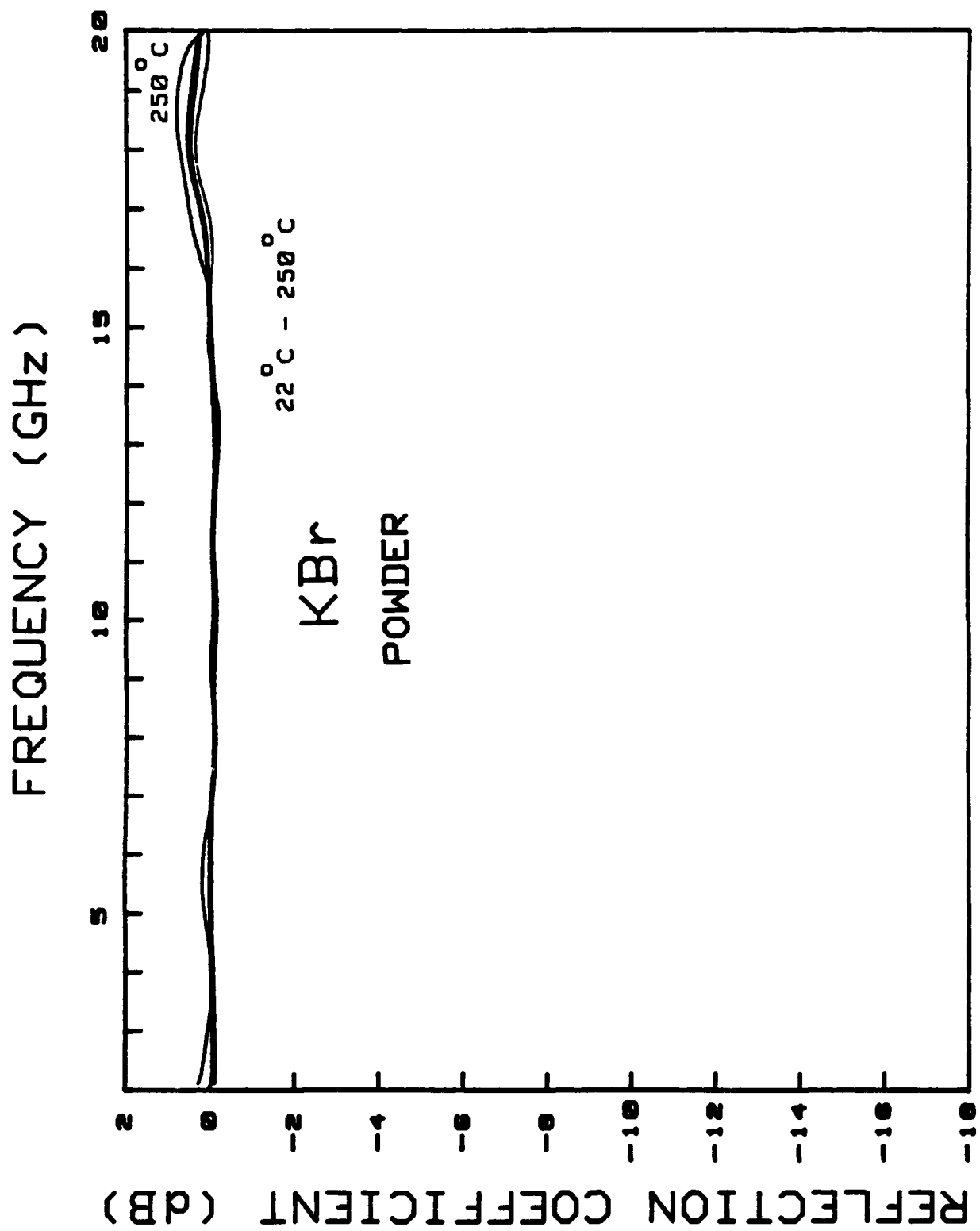


Figure 7. Temperature-Dependent Microwave Absorption for Potassium Bromide Loose Powder.

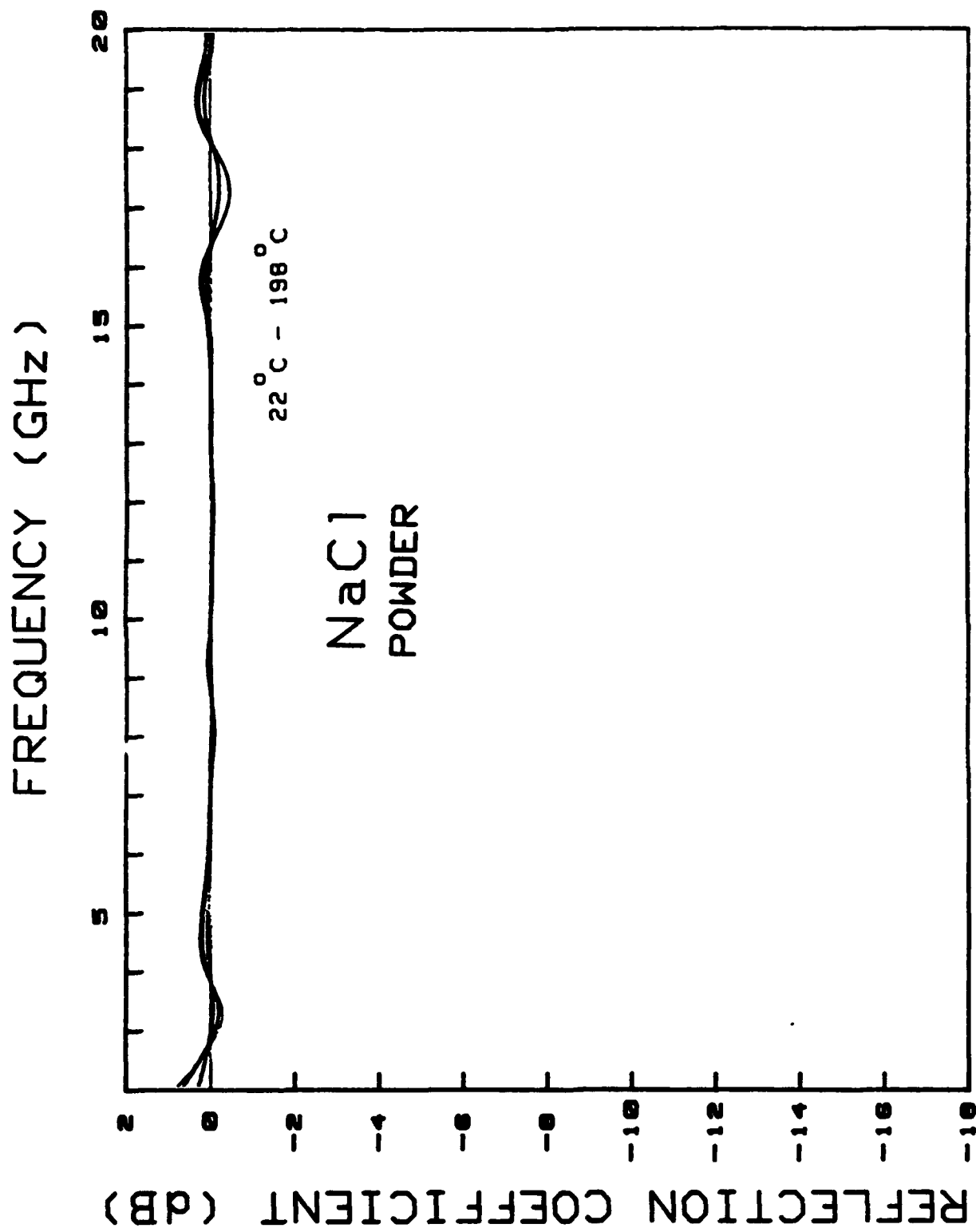


Figure 8. Temperature-Dependent Microwave Absorption for Sodium Chloride Loose Powder.

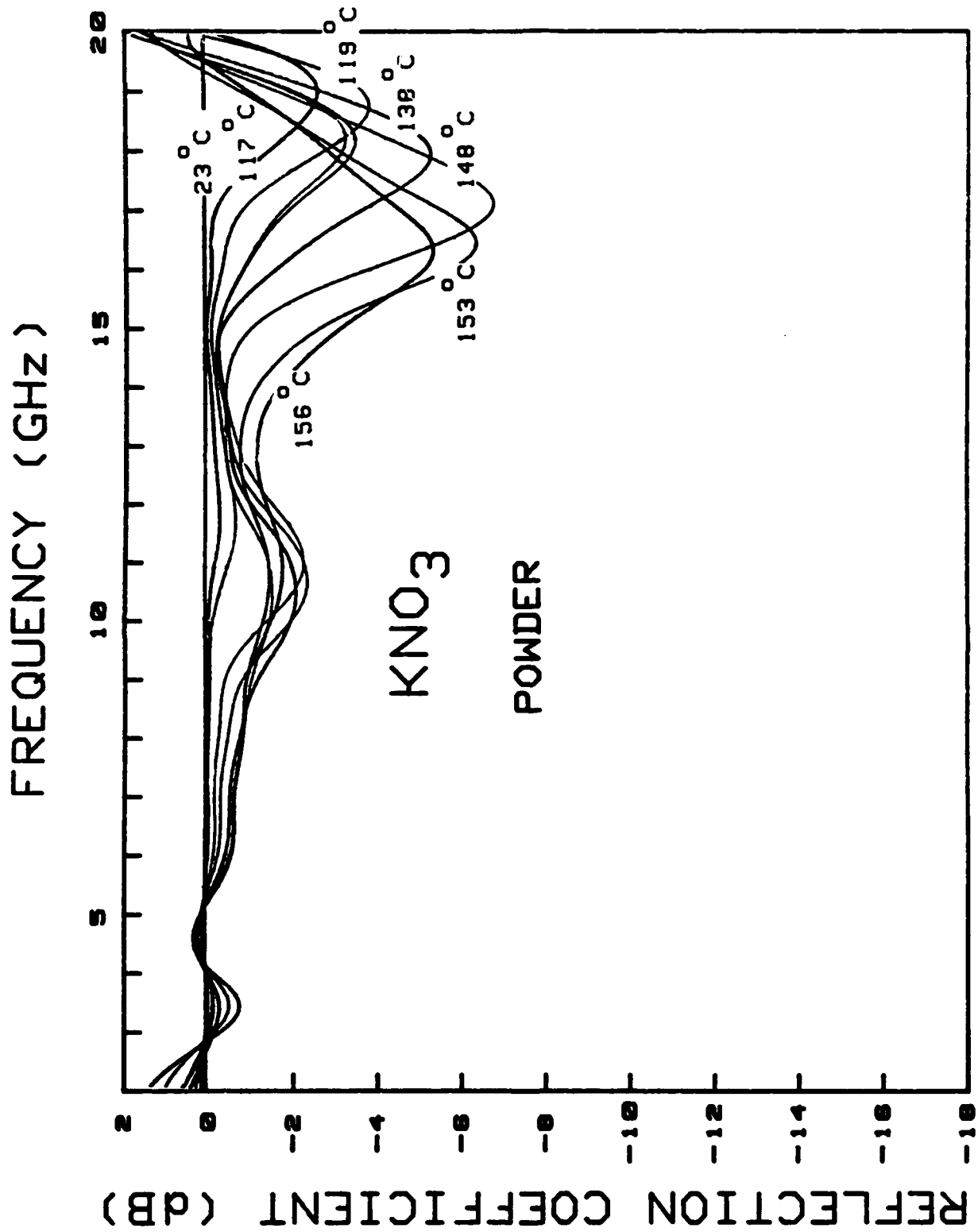


Figure 9. Temperature-Dependent Microwave Absorption for Potassium Nitrate Loose Powder.

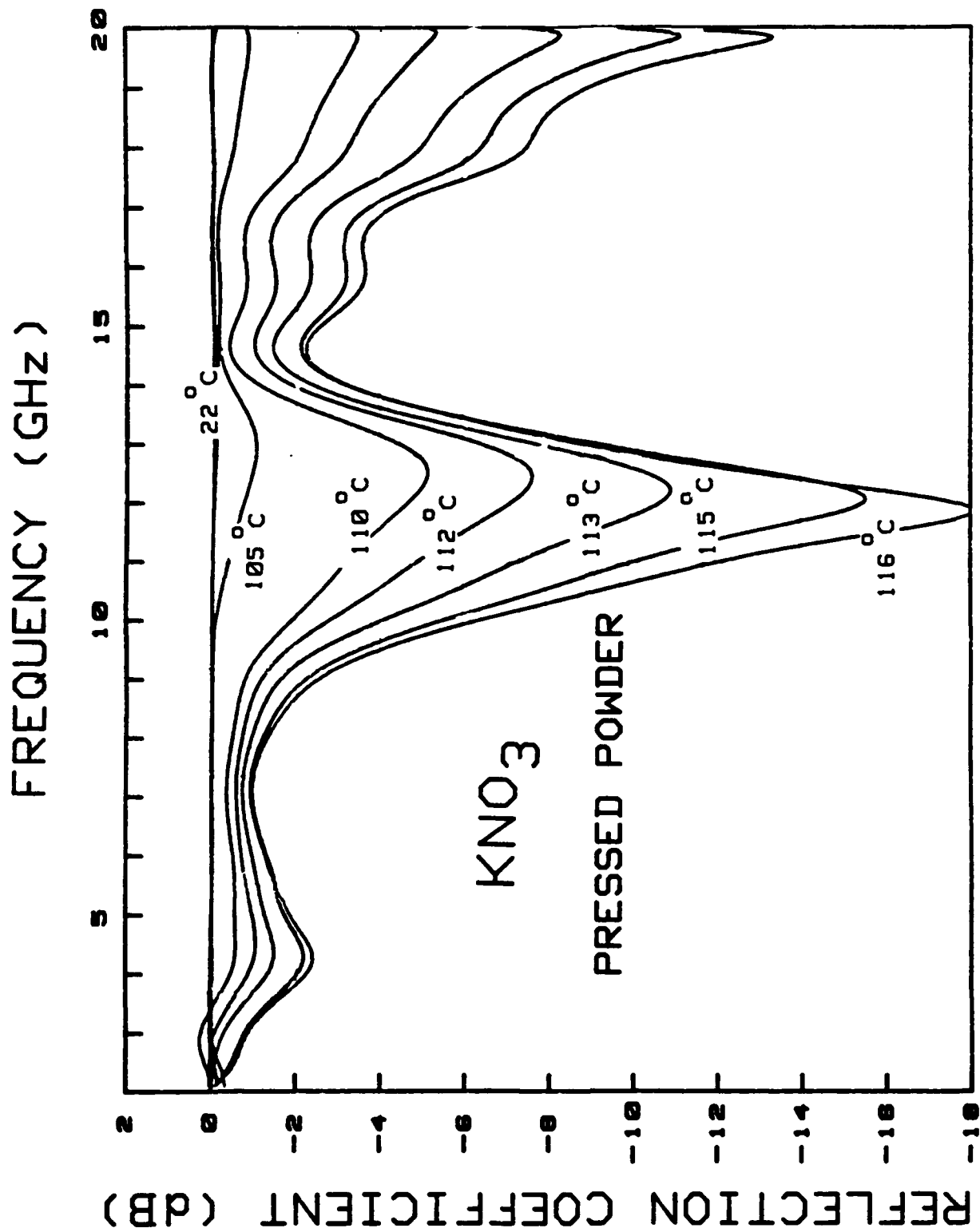


Figure 10. Temperature-Dependent Microwave Absorption From 22° C to 116° C for Nearly Fully Dense Potassium Nitrate Pressed Powder.

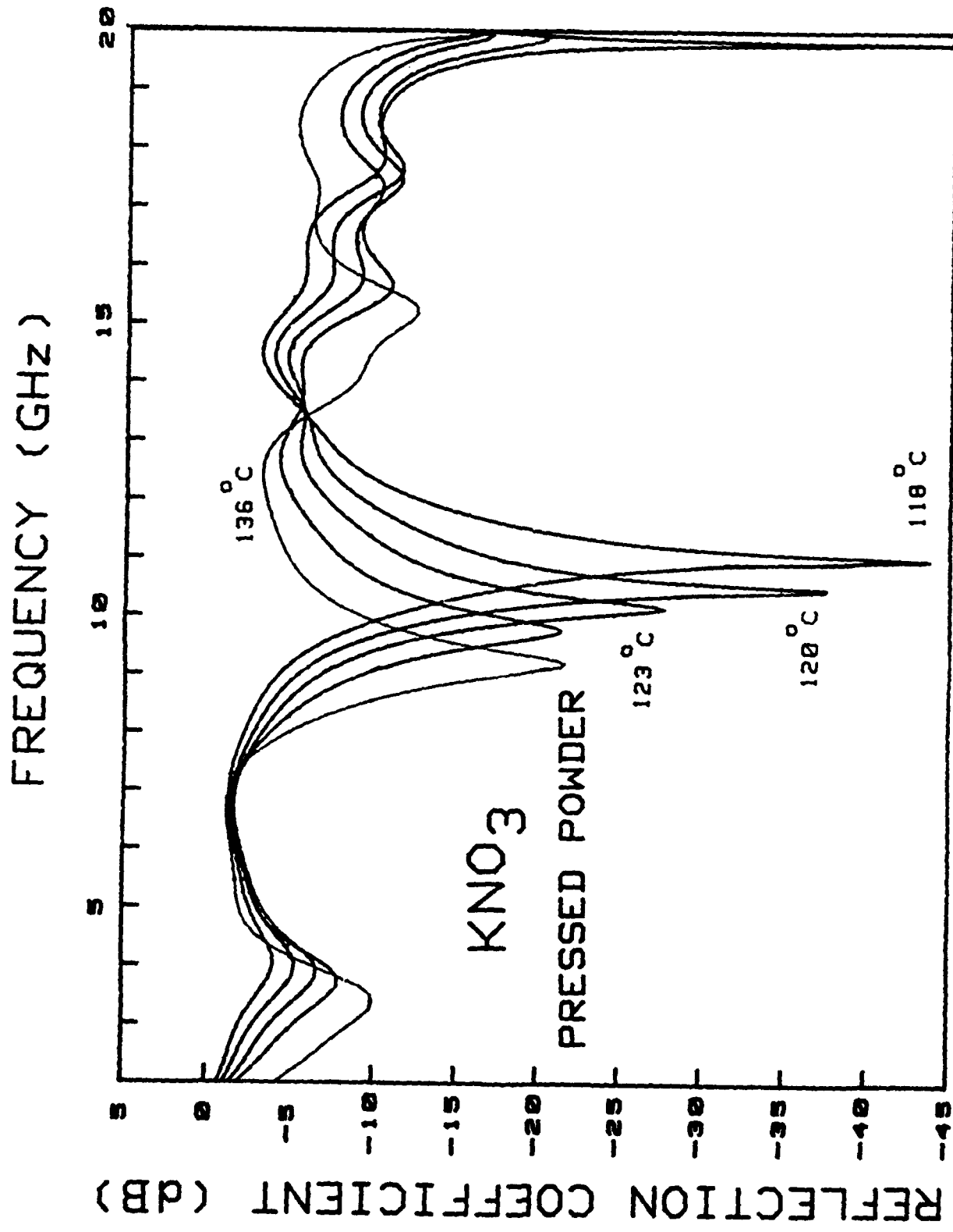


Figure 11. Temperature-Dependent Microwave Absorption From 118° C to 136° C for Nearly Fully Dense Potassium Nitrate Pressed Powder.

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