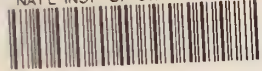


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DIELECTRIC MEASUREMENTS OF OIL SHALES AS FUNCTIONS OF TEMPERATURE AND FREQUENCY

Ramon L. Jesch
Robert H. McLaughlin

National Bureau of Standards
U.S. Department of Commerce
Boulder, Colorado 80303

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U.S. DEPARTMENT OF COMMERCE, Malcolm Baldrige, Secretary

NATIONAL BUREAU OF STANDARDS, Ernest Ambler, Director

PREFACE

This final report on Dielectric Measurements of Oil Shale as Functions of Temperature and Frequency terminates a two-year investigation on the Development of High Frequency Electromagnetic Mapping (HFEM) Technology funded by the U.S. Department of Energy, Laramie Energy Technology Center, Laramie, Wyoming. The contract was awarded to the Electromagnetic Fields Division of the National Bureau of Standards in 1980 for continuation of oil shale work started under Purchase Order No. DE-AP22-80PC10236 in 1979.

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A high-temperature sample holder designed by the National Bureau of Standards was used to determine the dielectric properties of approximately 40 oil shale samples as functions of temperature and frequency. A description of the sample holder characterization is given along with the measurement procedure and the sample preparation. Experimental results are given for different oil shale samples, varying in yield from 0.024 L/kg (6 gal/ton) to 0.338 L/kg (81 gal/ton), that were measured at room temperature (25°C) up to 500°C and within the frequency range of 5 - 1000 MHz.

Key words: dielectric properties, dielectric constant, electromagnetic, high-temperature sample holder, oil shale.

1. INTRODUCTION

In response to a request from the Department of Energy (DOE), the National Bureau of Standards (NBS), in 1979, undertook a program to develop the technology to utilize High Frequency Electromagnetic Mapping (HFEM) techniques for evaluating rubblized oil shale in the cold retort state in the modified in situ process. This technology development should also be applicable for using HFEM techniques for diagnosing, monitoring, controlling and evaluating modified in situ retorts after they are ignited. The modified in situ retorting process appears to offer significant environmental and economic advantages compared to surface retorting methods. Developmental work is needed to formulate optimum techniques for preparing modified in situ retorts. The efficiency of the prepared retort is governed, among other things, by porosity distribution, void ratio and its distribution and rubble size within the retort. There is presently no economical method of measuring the physical state of a cold retort, and, therefore, there is no way to predict the efficiency of the retort prior to its ignition. One possible technique for economically determining the physical state of the retort is by HFEM techniques.

The work performed during the period October 1979 to September 1980 was reported in the NBS Final Report SR-723-2-81, "Dielectric Properties of Oil Shale as a Function of Frequency" which contained the results of measurements the National Bureau of Standards made of ten half-core oil shale samples to determine their electromagnetic (EM) properties of permittivity (dielectric and attenuation constants) as a function of frequency. The samples were furnished by the Laramie Energy Technology Center (LETC), Laramie, Wyoming, and were taken from a core which was drilled between Occidental's retorts #7 and #8 near Rifle, Colorado. The measurements were taken within the frequency range of 1 - 2000 MHz at various frequency intervals. The use of EM properties of permittivity to characterize oil shale for electromagnetically mapping a retort is one possible technique being considered by the DOE for determining the physical state of a retort prior to its ignition.

The work performed during the period October 1980 to September 1981 was reported in the NBS Final Report SR-723-8-82, "Development of High Frequency Electromagnetic Mapping (HFEM) Technology" which was later published as DOE publication DOE/LC/10417-T1. This report described the baseline data work required to design a high-temperature sample holder and experiments for determining the EM properties of oil shale samples at elevated temperatures (200-500°C). A section was devoted to the theoretical approach for modeling EM sensing techniques for oil shale retorts. The retort was assumed to be a spheroid with an average dielectric constant and numerical results were presented. Finally, the measurement results were given for the spent and raw shale samples that were obtained from portions of the ten half-core samples plus the results of the EM transmission measurements taken on oil shale samples at the LETC in Laramie, Wyoming.

This report covers the work performed from October 1981 to September 1982, validating the accuracy of the high-temperature sample holder at elevated temperatures up to 500°C. Measurement results are given for approximately 40 oil shale samples that were measured in the high-temperature sample holder at room temperature and up to 500°C and within the frequency range of 5 - 1000 MHz. The oil shale samples were furnished by LETC and had yields ranging in value from about 0.025 L/kg (6 gal/ton) to 0.338 L/kg (81 gal/ton).

2. SAMPLE HOLDER CHARACTERIZATION

As previously reported in the NBS Final Report SR-723-8-82, the high-temperature sample holder was characterized at room temperature (25°C) by measuring three known dielectric standards. Inserting these standards in turn as Y_L in the sample holder allowed the elements Y_1 , Y_2 , and Y_3 of the π -network representation of the intervening circuit (figure 1) to be solved at each frequency, thereby allowing the dielectric properties of an unknown sample to be determined. However, once the sample holder is heated, changes occur in its physical properties and dimensions which, in turn, change the sample holder calibration as a function of temperature making it necessary to measure the three known dielectric samples at every temperature and frequency before the dielectric properties of the unknown oil shale samples can be correctly determined at elevated temperatures up to 500°C.

2.1 Dielectric Samples

Four known dielectric samples consisting of quartz, glass, ceramic and molybdenum were used to characterize the sample holder at room and elevated temperatures. While only three known dielectric samples are actually required to characterize the sample holder, the fourth dielectric standard sample is used as the unknown sample which allows a direct comparison to be made between known standard values and measured values as a function of frequency and temperature. These four samples were chosen because of their well-known dielectric properties that remain fairly constant over the whole temperature and frequency range. Moreover, their constants of thermal expansion closely resemble that of the large ceramic bead which holds the sample throughout the measurement run. This means, that once the sample holder is heated to 500°C, the standard samples will not expand at a faster rate than that of the ceramic, preventing the bead from fracturing.

2.1.1 Preparation of Samples and Sample Holder

The four dielectric samples were machined into disks 2.540 cm (1.000 inch) in diameter and 6.477 mm (0.255 inch) thick. The diameter of the sample cavity in the ceramic bead was made approximately 0.051 mm (0.002 inch) larger than the sample disk to allow for thermal expansion. After loading the sample

π - Network

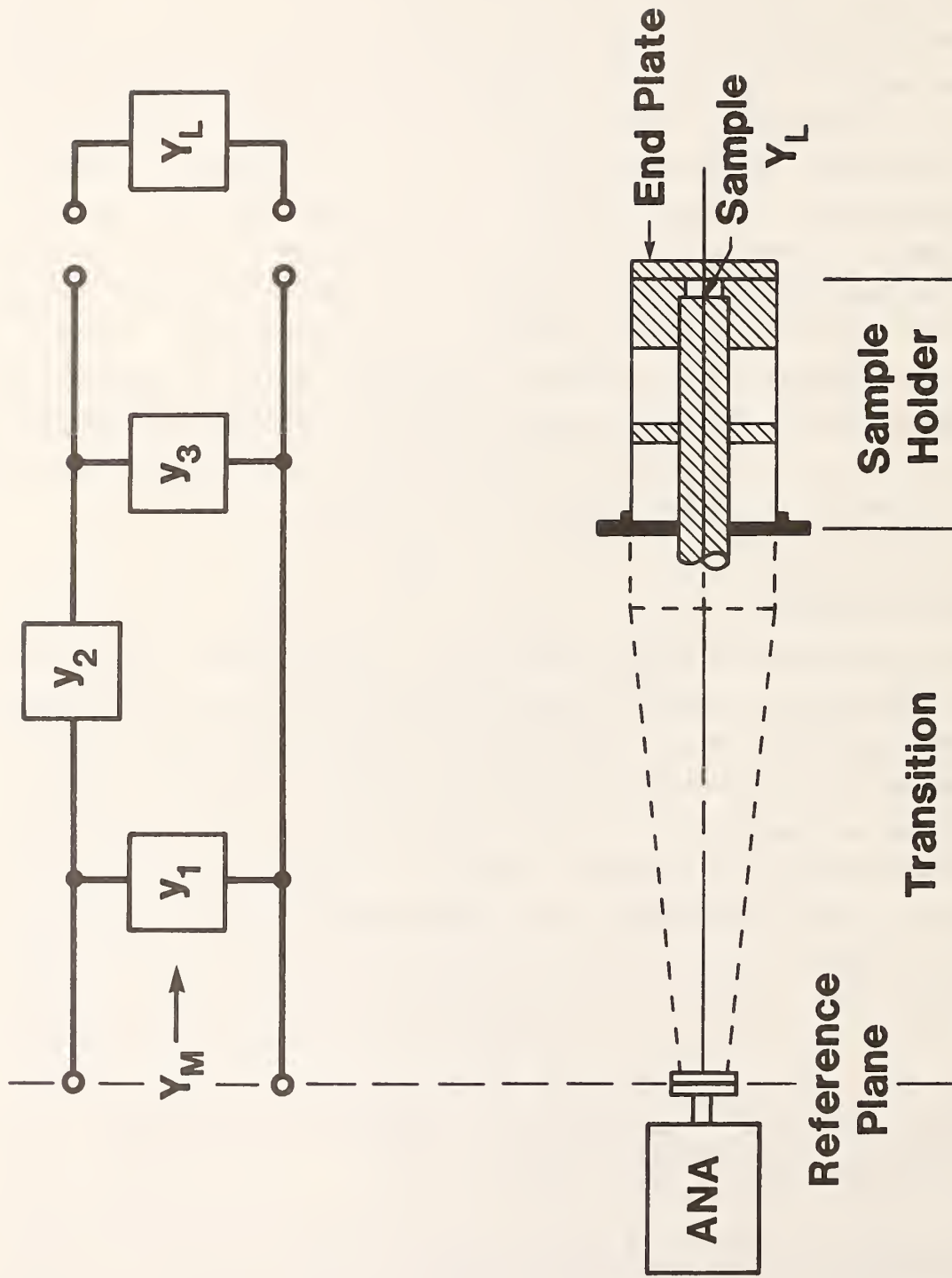


Figure 1. Pi-network model of transition and sample holder.

disk in the cavity, the endplate was placed in position on the sample holder and preloaded by tightening the compression springs over the bolts holding the spider clamp as shown in figure 2. Typically, 454 kg (1,000 lbs) of pressure is applied to the endplate and to the sample. This helps to insure that proper electrical contact is maintained between the sample and the conductors throughout the heated measurement run. Also, this type loading simulates an overburden pressure of approximately 8790×10^3 Pa (1275 psi). Finally, insulation is placed around the outside of the sample holder and endplate to achieve better temperature regulation throughout the heating cycle.

2.2 Measurement Procedure and Results of Standard Samples

The EM properties of permittivity (dielectric and attenuation constants) of the dielectric samples measured in the sample holder were obtained from the measured admittance. The admittance, in turn, was obtained by measuring the input reflection coefficient of the holder with its sample at the plane of measurement with an Automatic Network Analyzer (ANA). This technique was previously described in detail in NBS Final Report SR-723-8-82. The measurement uncertainty of reflection coefficient used in these measurements was 0.004 in magnitude and 0.4 degrees in angle. Just prior to the measurement run, the sample was evacuated by means of a vacuum pump connected to the sample holder. Approximately 10 minutes was required to pump the system down to less than 4.66 Pa (35 microns of mercury), assuming that no leaks were present around the gold wire seals. The sample was then purged with nitrogen to remove any residual oxygen. The evacuation and purging operation was used on all the oil shale samples and also on the molybdenum standard sample, which had the characteristic of oxidizing quite rapidly when heated.

Measurements on the dielectric samples were taken between the frequencies of 5 and 1,000 MHz in 5 MHz steps and at temperatures of 25°C (room temperature), 50°C, 100°C, 150°C, 200°C, and then in 25° steps up to 500°C. High quality thermocouples (chromel-constantan) were used to monitor temperatures throughout the complete test cycle. The time/temperature heating profile that was finally employed was 1.6°C/min, which gave uniform heating of the sample holder and sample over the whole temperature range up to 500°C. Figure 3 shows the heating profile that was used for the permittivity measurements. These measurements take a total time of 5 hours per sample to complete, making

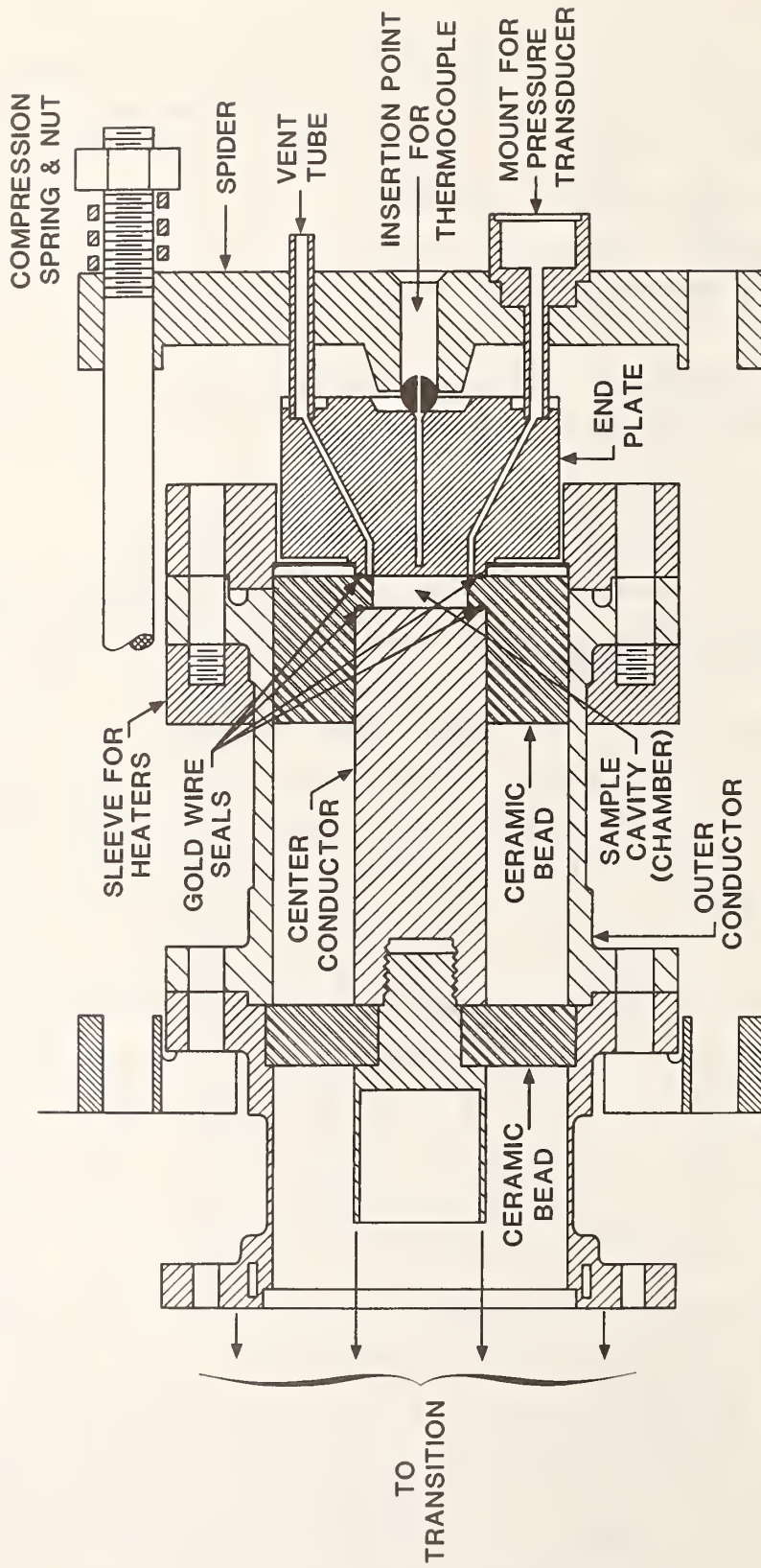


Figure 2. High-temperature sample holder.



Figure 3. Heating profile used for making the permittivity measurements.

it important that each measurement run is carefully prepared. A measurement run consists of taking measurements between 5 and 1,000 MHz in 5 MHz steps at each of the temperatures specified above up to 500°C. By the time the sample holder is loaded with a sample, measured over the full temperature range, allowed to cool down, unloaded, and a new sample reloaded, there is a total elapsed time of approximately one day.

Of the four standard dielectric samples that were measured, molybdenum, quartz, and ceramic samples were used to characterize the sample holder, while the standard sample of glass was used as the unknown material, to evaluate the characterization, because of its dielectric constant which approximates that of oil shale and its value which is well known and documented [1,2]. However, other combinations of these four standard samples can be used. Figure 4 shows the relative dielectric constant plotted from the measurement data obtained using a standard dielectric sample of glass as the unknown material as a function of temperature and compared with known standard dielectric values of glass obtained from other sources [3] and by extrapolation at these same temperatures. Moreover, these values are independent of frequency. Looking at the plot, one can see the close agreement between the measured values and the known standard values, which is an indication of the reasonably accurate results that can be achieved when using the high-temperature sample holder.

3. OIL SHALE MEASUREMENTS

3.1 Oil Shale Samples

The first series of oil shale measurements were made on a few samples of oil shale taken from a block of shale identified by the LETC as A-1. The measurements were investigative in nature to determine the integrity of the sample holder at room and elevated temperatures and determine the extent of additional measurements. With the successful conclusion of the first series of elevated temperature runs, the LETC requested the NBS to make additional measurements on a number of different oil shale samples varying in yield from 0.025 L/kg (6 gal/ton) to 0.338 L/kg (81 gal/ton). This new work was broad enough in scope to include the coordinated efforts of the LETC to investigate the organic and mineral matter of like oil shale samples and the University of Wyoming Physics Department who, funded by the DOE, proposed to develop a

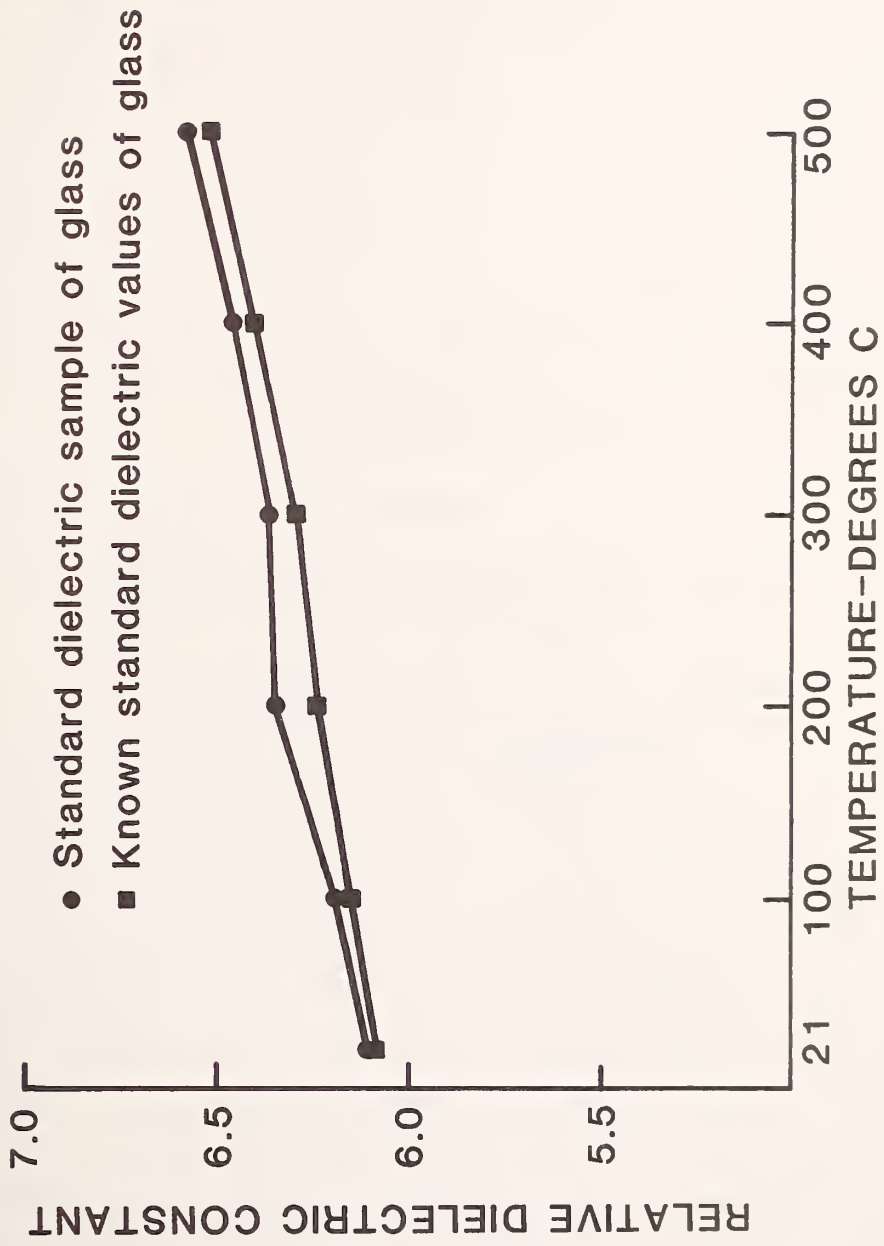


Figure 4. Plot of the relative dielectric constant of a standard sample of glass compared with known standard values of relative dielectric constant of glass as a function of temperature.

theoretical model of oil shale based upon its electrical and physical properties. Toward this end, provisions were made by the NBS to provide the University of Wyoming with dielectric measurement data on the oil shale samples to develop their model.

In the oil shale sample selection process, like oil shale samples were cut from the same bedding plane from blocks of shale. Different oil yield samples were selected from different block types. Several samples from each block type were sent to the NBS for dielectric property measurements at elevated temperature, while other like samples were tested at the LETC for material balance Fischer assay and minerals. Table 1 shows the density and the estimated oil yield information on the oil shale samples received by the NBS from the LETC for dielectric property measurements. With the exception of samples designated D and E, the LETC performed their measurements on like oil shale samples taken from the same block types.

Table 1

Density and oil yield information on oil shale samples received by the NBS from the LETC.

<u>Sample Designation</u>	Block Type	<u>Number of Like Samples</u>	Density	Oil Yield	
			<u>g/cu cm</u>	<u>L/kg</u>	<u>(gal/ton)</u>
C		3	2.28	0.088	21
D		3	2.04	0.158	38
E		4	2.09	0.142	34
F		3	1.82	0.234	56
G		3	1.90	0.204	49
I		4	1.70	0.280	67
J		4	1.57	0.338	81
L		2	2.57	0.025	6
P		2	1.63	0.309	74
S		2	2.28	0.088	21
T		2	2.19	0.113	27
U		2	2.05	0.154	37

3.1.1 Measurement Results of A-1 Oil Shale Samples

A history, containing information on dielectric properties, was established on oil shale samples taken from a block of shale identified by the LETC as A-1, which had a yield of about 0.094 L/kg (22.6 gal/ton). The samples were machined into disks 2.520 cm (0.992 inch) in diameter and 6.477 mm (0.255 inch) thick with the bedding planes of the samples kept parallel with the disk diameter. These samples were then measured at low frequencies in a dielectric sample holder of the Hartshorn Ward type [4] at frequencies of 0.01, 0.02, 0.04, 0.1, 0.2, 0.4, 1.0, 2.0, 5.0, and 10.0 MHz at room temperature (25°C). Figure 5 shows a plot of the dielectric constant and the imaginary part of oil shale sample A2 as a function of frequency. While a detailed error analysis was not performed, it is estimated that the measurement uncertainty is of the order of ± 1 to ± 2 percent on the dielectric constant and ± 5 percent on the imaginary part. This sample is representative of the other samples measured from this same group. These measurements give accurate results on the dielectric constant and imaginary part of the oil shale samples which, in turn, help to indicate small differences in loss and oil yield (as indicated by the dielectric constant) between samples. They also provide a larger data base on the dielectric properties of the samples over a wider frequency range.

Each oil shale sample was then measured in the high-temperature sample holder at the same frequencies and temperatures that were used for the standard dielectric samples. The sample holder was operated in the vented condition allowing gasses and shale oil to escape through a vent tube that is attached to the endplate as shown in figure 6. A balloon was placed on the other end of the vent tube allowing the shale oil given off by the sample to be collected into the balloon. When the balloon starts to expand, this is a good indication that the sample has reached the temperature where retorting has commenced. This occurs around 375°C, accompanied by a faint odor of hydrogen sulfide. At approximately 400°C, the first trace of shale oil can barely be seen at the neck of the balloon. By the time the temperature has reached 475°C, it appears that all effluents have been driven off the oil shale sample. The heating of the sample holder and samples continues until 500°C has been reached and the final measurement run taken. At this point, the heaters are turned off and the sample holder allowed to cool down to room temperature. However, for one measurement run, additional measurements were taken at 15-minute intervals for a total elapsed time of 45 minutes while the

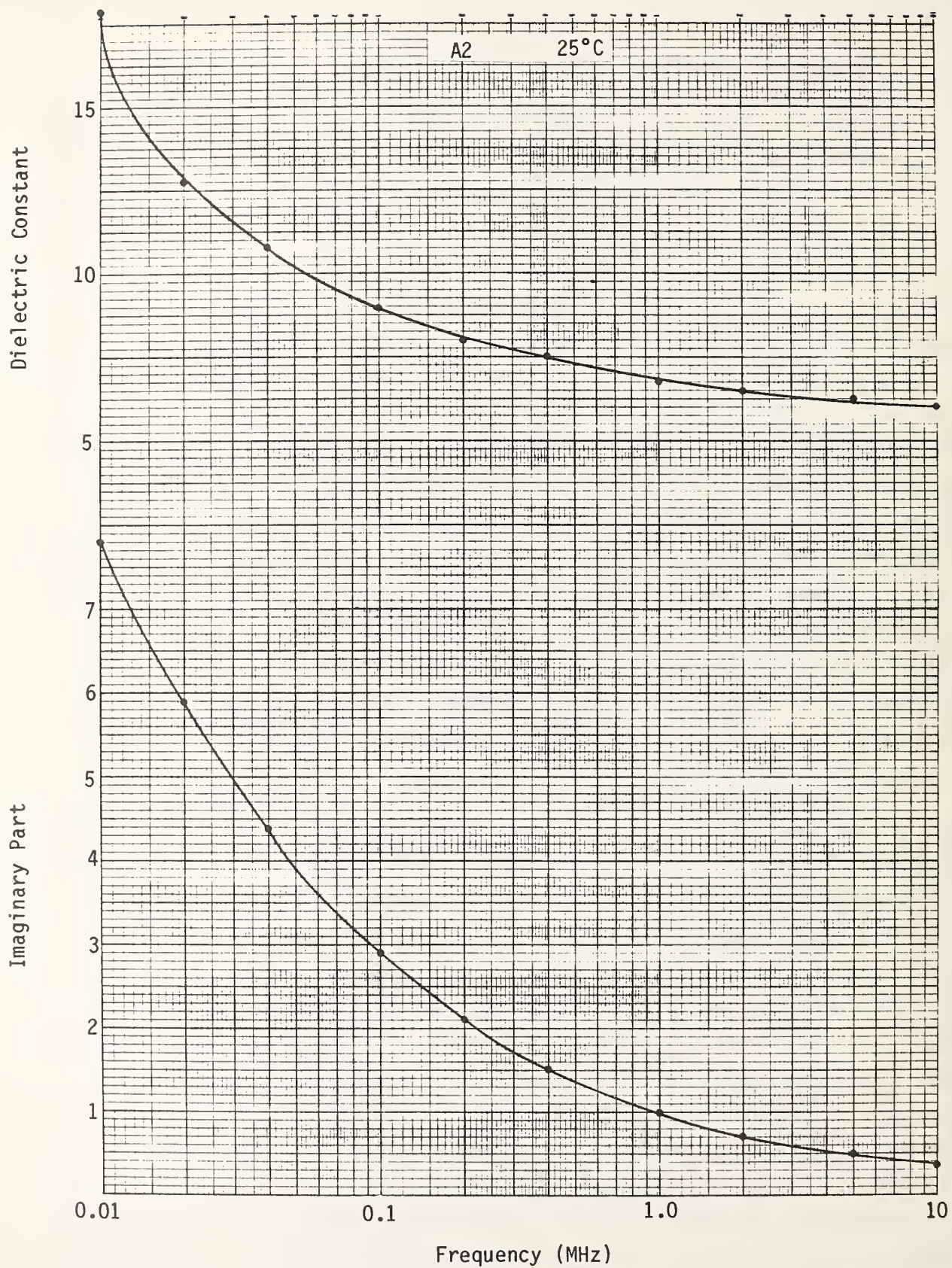


Figure 5. Plots of dielectric constant and imaginary part of oil shale sample A2 as a function of frequency at room temperature (25°C).

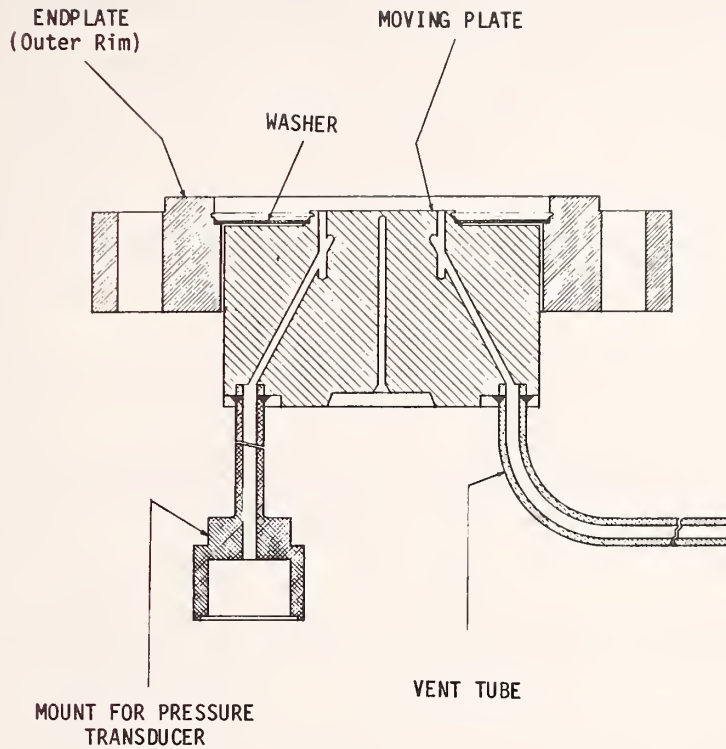


Figure 6. Drawing of endplate.

temperature was held at a constant 500°C. No changes in the measurement results were noticed between these new data and the data recorded earlier at 500°C. Also, on two of the measurement runs after the sample holder had cooled down to room temperature (25°C), the room temperature measurements were repeated again allowing a comparison to be made of the dielectric properties of the oil shale samples before and after retorting. For these particular samples, the before-and-after dielectric properties were almost identical.

Figure 7 is a plot of some of the measurement data obtained from four of the oil shale samples as a function of temperature at 500 MHz. This frequency was chosen because of the good data representation of the A samples at this point, plus it is typical of the other measurement frequencies. No explanation is given at this time for the marked increase in the dielectric values for the oil shale that occur in the 400 to 500°C range. This increase was also noted at all the other measurement frequencies. It is interesting to

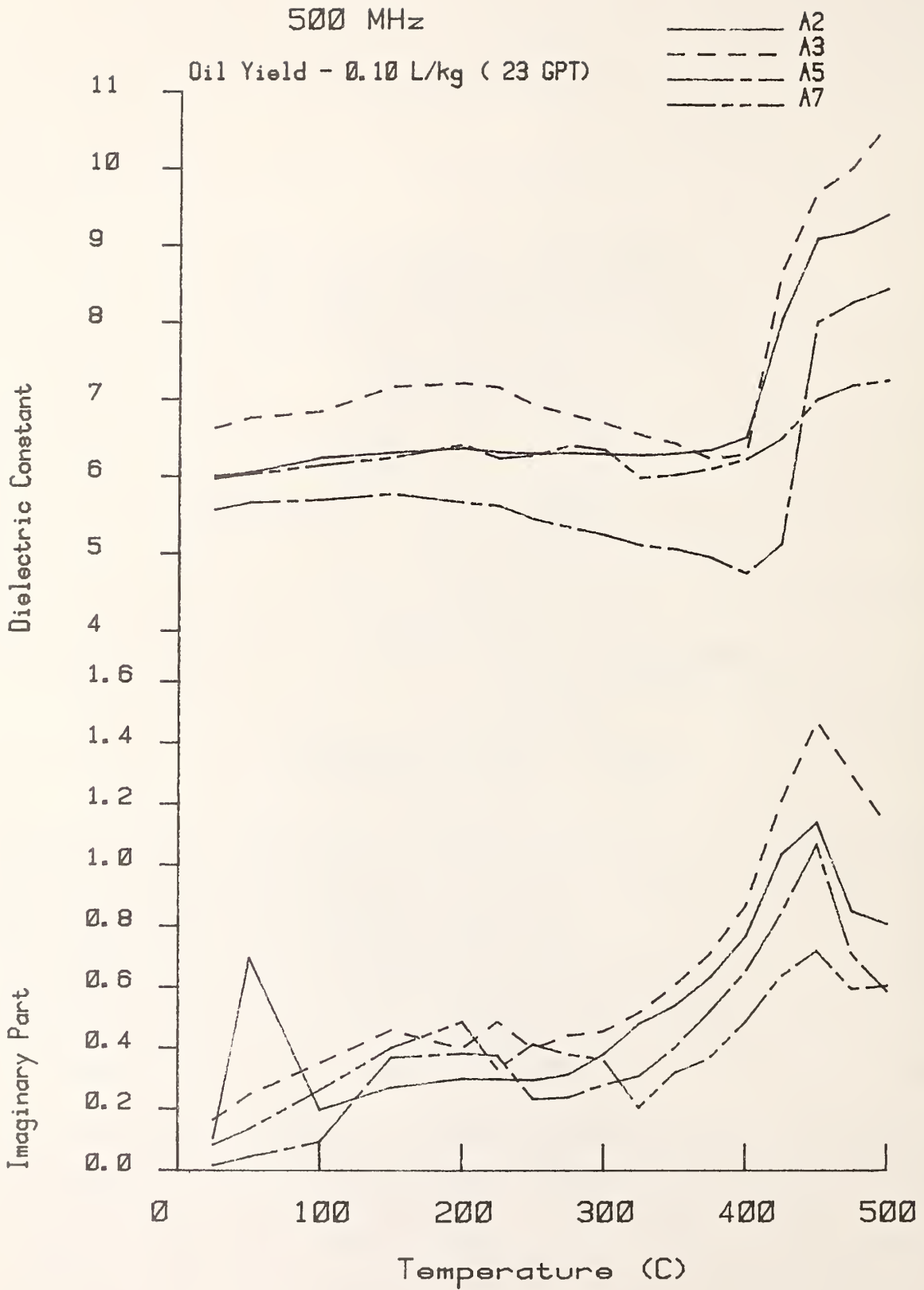


Figure 7. Plots of dielectric constant and imaginary part of four oil shale samples as a function of temperature at 500 MHz.

note that in an oil shale report by DuBow [5], pressure curves show that a drastic change in mechanical strength of oil shale samples occurs at around 380°C. In subsequent measurement runs, several modifications in the measurement procedure were tried that could possibly affect the high value dielectric properties at 400°C and above. These included:

1. reducing the endplate pressure from 454 kg (1000 lbs) to 181 kg (400 lbs),
2. increasing the endplate pressure to 816 kg (1800 lbs),
3. reducing the diameter of the oil shale sample from 2.520 cm (0.992 inch) to 2.222 cm (0.875 inch),
4. reducing the temperature heating profile from 1.6°C/min to 1.0°C/min, starting at 350°C.

In the final analysis, these modifications did not alter the measurement results. After the sample holder cool down, as previously reported, the values of the dielectric properties of the oil shale samples were almost identical to the original room temperature values.

3.1.2 Measurement Results of Additional Oil Shale Samples

3.1.2.1 Measurement Conditions

For the additional oil shale samples, three types of measurement conditions were investigated: 1) Measurement of the electrical permittivity (dielectric and attenuation constants) as a function of sample temperature with the sample holder operated in two different modes as a) vented, so as to relieve any developed pressure and b) unvented, so as to retain any developed pressure. 2) Measure the pressures developed during the unvented mode of operation as a function of the sample holder temperature. 3) With the sample held at a fixed temperature, determine the effect of an externally applied pressure on the electrical permittivity.

Under actual measurement conditions, items 2 and 3 were difficult to attain. The main problem encountered with item 2 resulted from the air-cooled pressure transducer that was obtained for this project. This transducer was designed for measuring gasses up to 500°C, but not fluids. A fluid has to be used to eliminate the compressible gas volume contained in the vent tube and the pressure transducer tube (see fig. 6) in order to externally monitor the pressure accurately. The transducer air cooling is insufficient to cool the fluids at 500°C and prevent damage to the pressure transducer. Ideally, the pressure transducer should be located next to the oil shale sample. In the case of item 3, this was simply a case of not being able to maintain a tight pressure seal over the whole temperature cycle. Once the oil shale sample began to expand with heating, the expansion allowed gasses to escape past the gold wire pressure seals. This condition occurred at about 225°C and continued until about 400°C when the system resealed, as the oil shale samples lost compressive strength and their released effluents. In the actual pressurized measurement runs, dry nitrogen was used to pressurize the sample to about 3447×10^3 Pa (500 psi) and was applied externally through the vent tube. Only two or three measurement runs were attempted since a problem still existed in holding a pressure seal over the 225°C to 400°C temperature range.

The remainder of the oil shale samples were investigated by measurement condition 1a. where the sample is vented so as to relieve any developed gas-liquid pressure. This was the condition used for measuring the A-1 oil shale samples described in section 3.1.1.

3.1.2.2 Preparation of Oil Shale Samples

As in the previous section, 3.1.1, the samples from table 1 were machined into disks 2.520 cm (0.992 inch) in diameter and 6.477 cm (0.255 inch) thick with the bedding planes of the samples kept parallel with the disk diameter. These samples were also measured at low frequencies in the Hartshorn Ward type dielectric sample holder at frequencies of 0.01, 0.02, 0.04, 0.1, 0.2, 0.4, 1.0, 2.0, 5.0, and 10.0 MHz at room temperature (25°C). To help establish a higher degree of confidence in the results of the dielectric measurements, more than one like sample was prepared and measured for each designated sample type (see table 1). As before, evacuation and purging of the samples preceded the heated measurement runs.

3.1.2.3 Measurement Results

A total of thirty-four oil shale samples were measured between the frequencies of 5 and 1,000 MHz in 5 MHz steps and at temperatures of 25°C (room temperature), 50°C, 100°C, 150°C, 200°C, and then in 25° steps up to 500°C. A desk-top calculator controlled the measurement process, computed the permittivity, analyzed the data, and produced the graphical results. A smoothing routine was applied to the data to reduce random variations and systematically induced spikes.

Figures 8-13 show plots of the dielectric constant and the imaginary part of the complex dielectric constant of all the designated samples from table 1 as a function of temperature at 500 MHz. Again, this frequency was chosen because of the good data representation of all the oil shale samples at this point, plus it was typical of the other measurement frequencies. Frequency plots are shown later on in the text for several oil shale samples. The figures are ordered in increasing oil yield richness from the leanest, L = 0.025 L/kg (6 gal/ton), to the richest, J = 0.338 L/kg (81 gal/ton). The values for the dielectric constant in the figures appear to increase from 25°C to about 200°C before decreasing in value and then increase rapidly, in most cases, starting around 400°C, as noted previously in figure 7 with the A samples. The increase in dielectric values up to 200°C probably results from the moisture being released from the oil shale samples. In reviewing figures 8-13, probably the most significant discovery is that the dielectric constant of the oil shale samples decrease as the oil yield increases. This phenomena is even more apparent in figure 14, which compares temperature plots of the dielectric constant of the lower yield A sample with those of the highest yield J sample as a function of frequency. One can see the marked difference in values for dielectric constant between the A and J samples which vary between 6.5 and 4.0 respectively at 25°C.

Figures 15-16 show frequency plots of the dielectric constant and the imaginary part as a function of temperature for oil shale samples C, G, D, I, and J. Figures 15 and 16 are given to show effects of frequency on the measurement data. With the exception of increased values of dielectric constant and imaginary part at 15 MHz, which is typical of dielectric measurements at lower frequencies, the frequency effects for the most part are minimal with the dielectric values remaining fairly constant above 100 MHz.

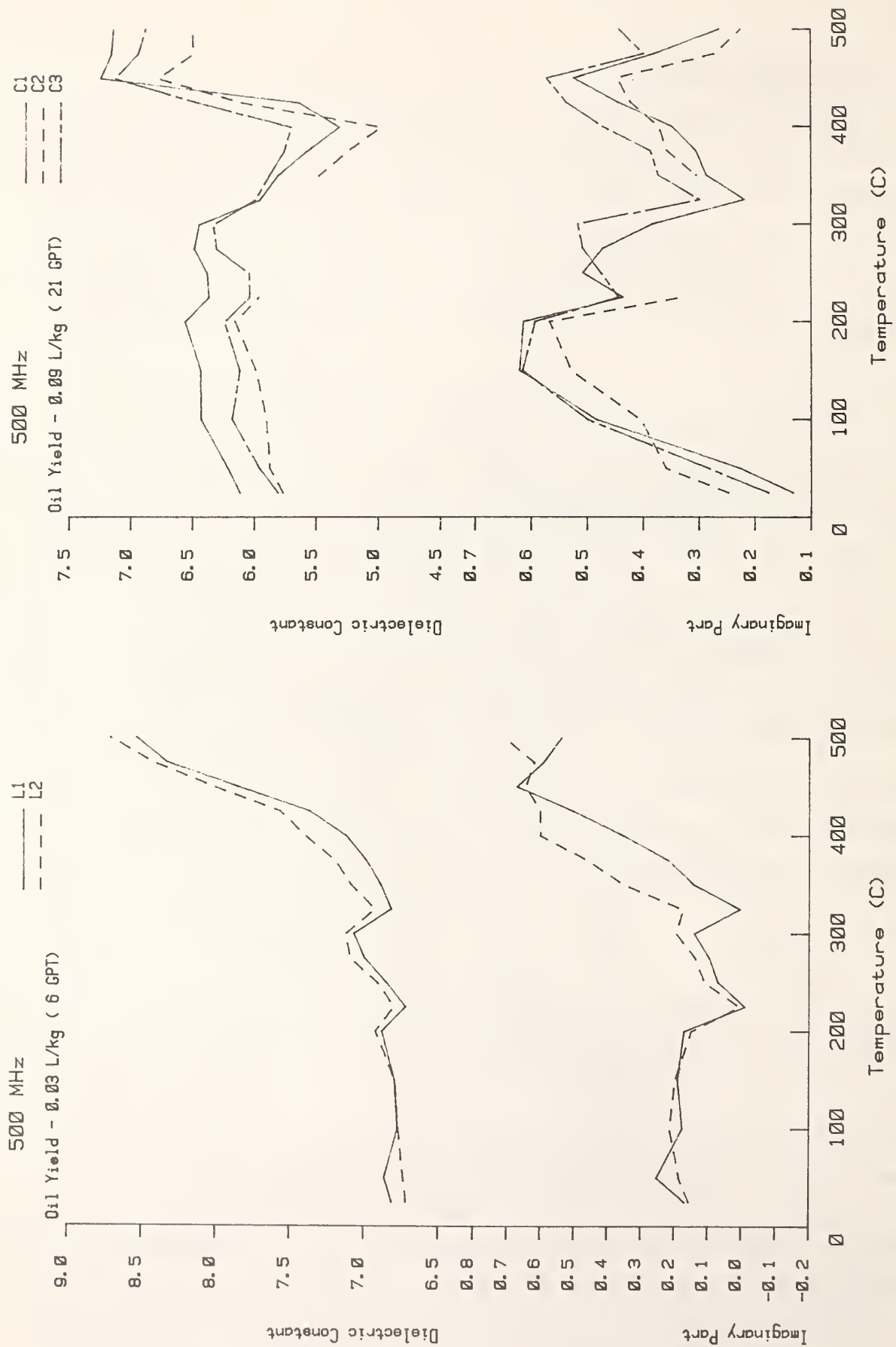


Figure 8. Plots of dielectric constant and imaginary part of designated samples L and C as a function of temperature at 500 MHz.

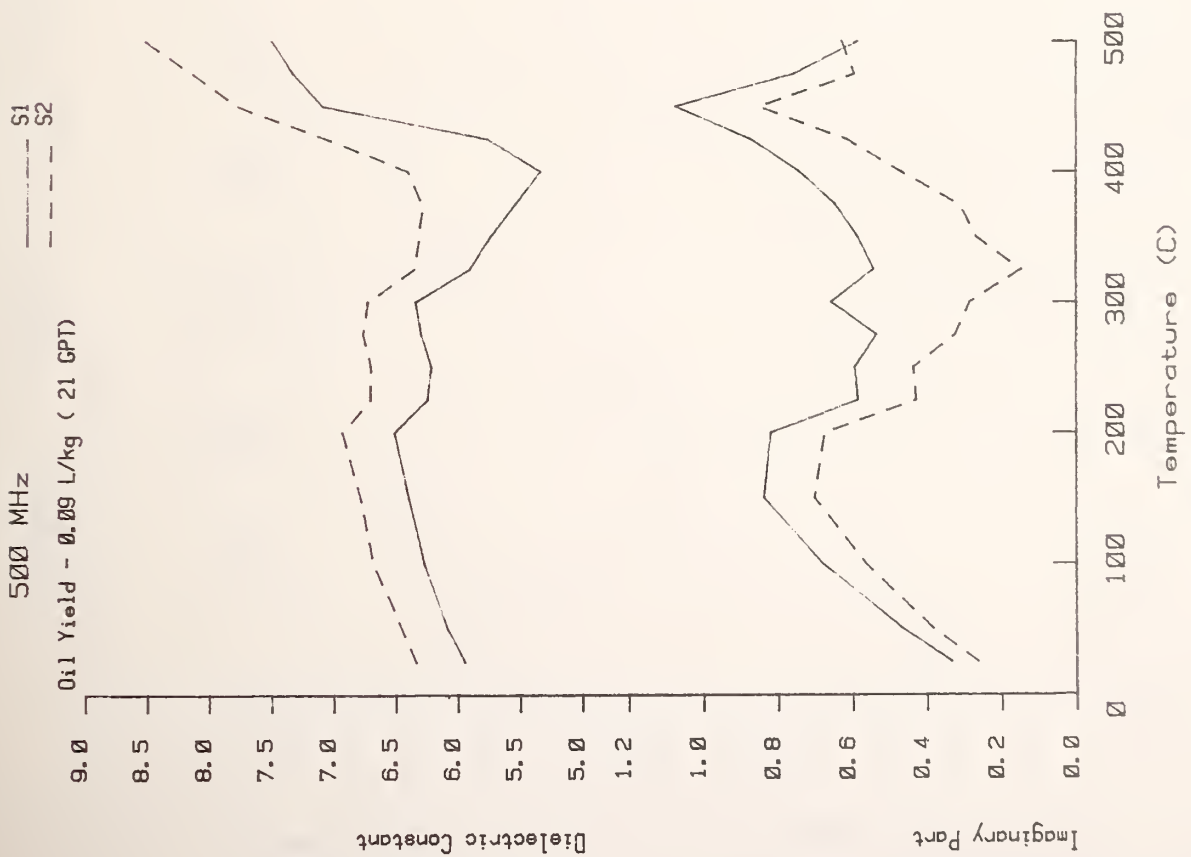
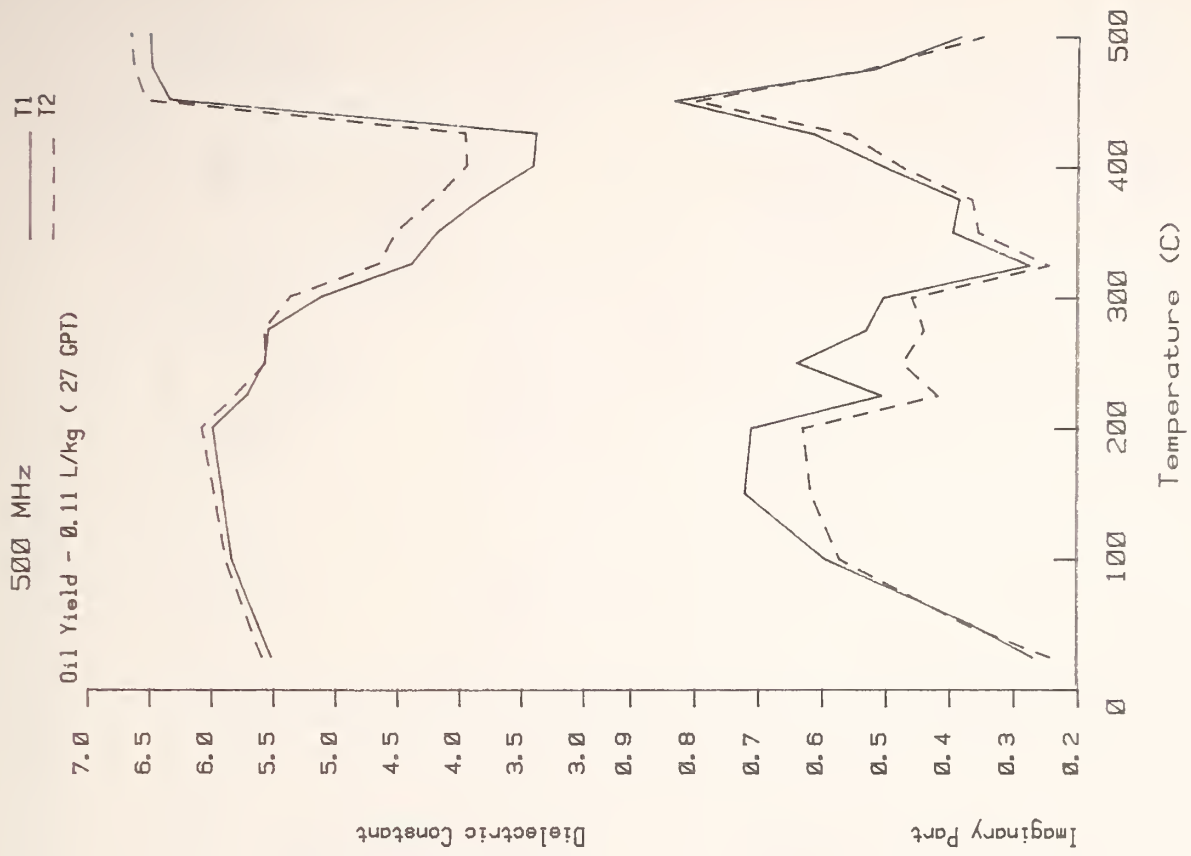


Figure 9. Plots of dielectric constant and imaginary part of designated samples S and T as a function of temperature at 500 MHz.

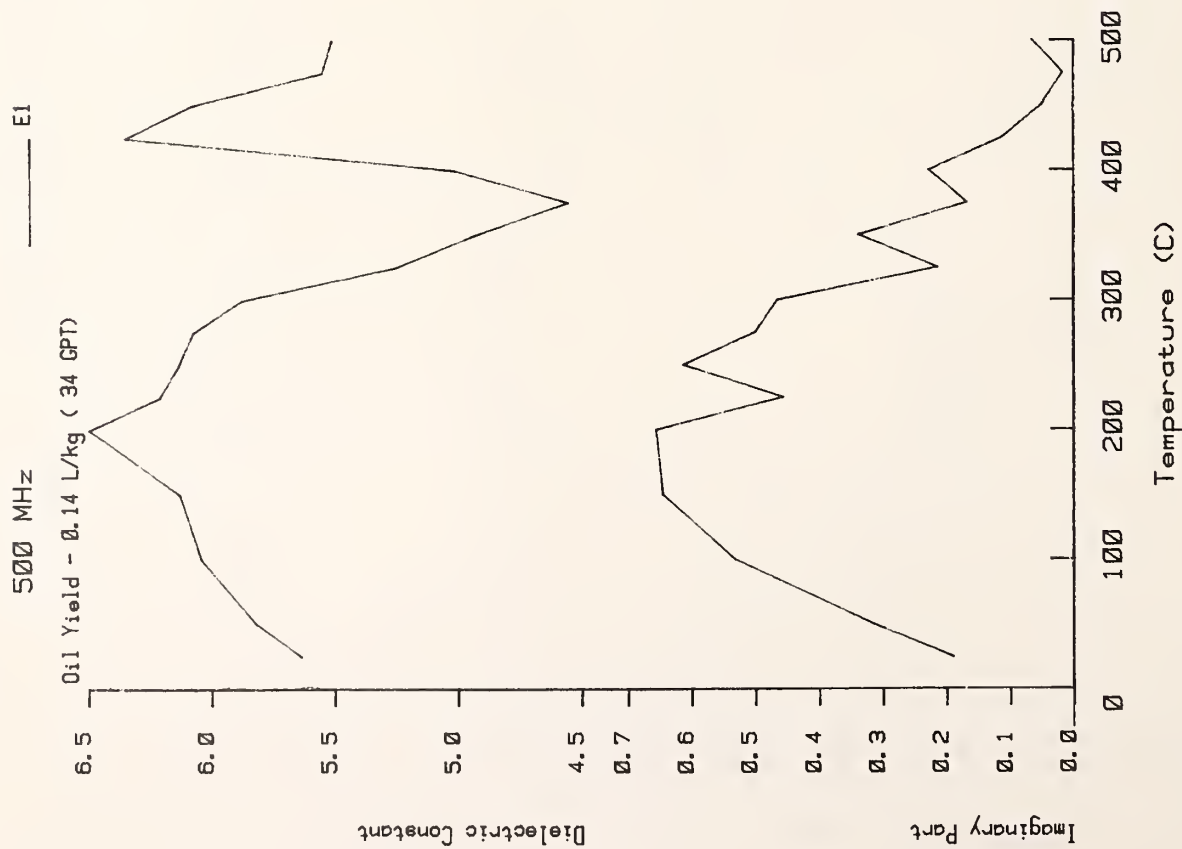
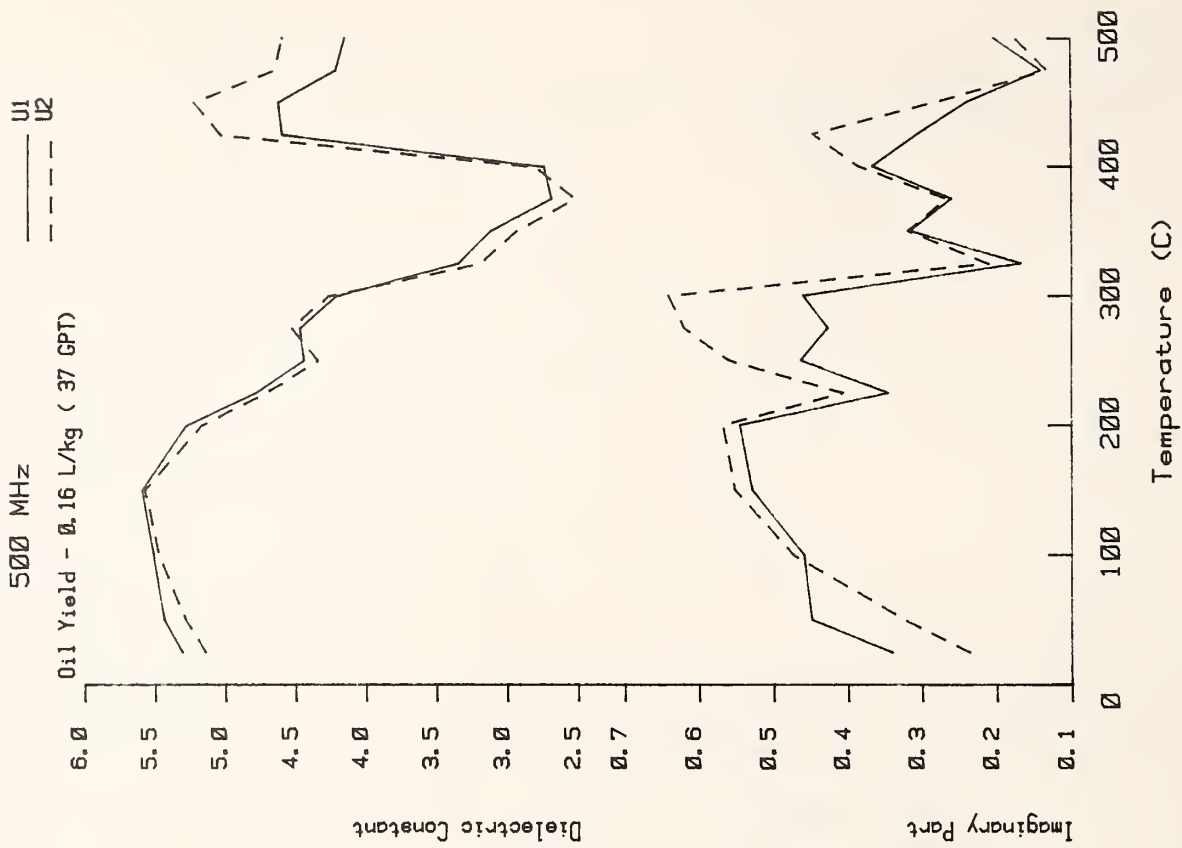


Figure 10. Plots of dielectric constant and imaginary part of designated samples E and U as a function of temperature at 500 MHz.

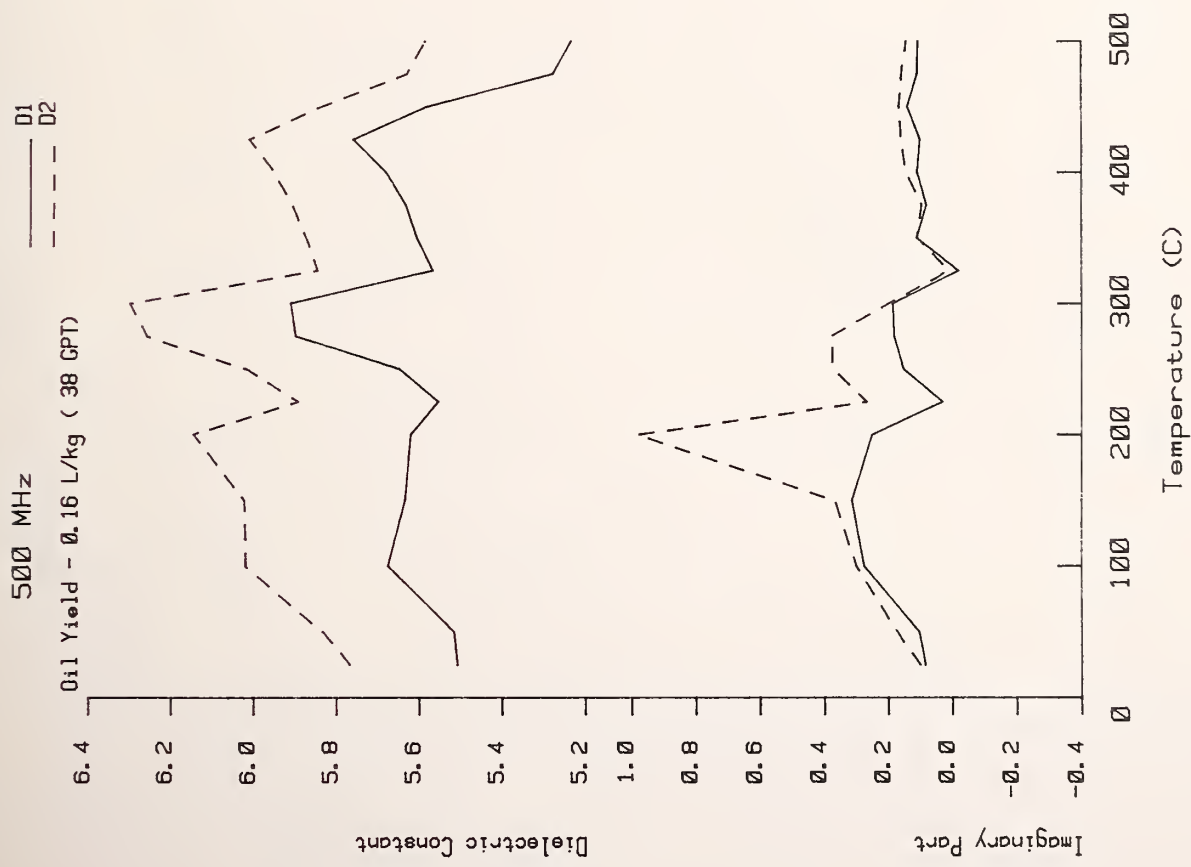
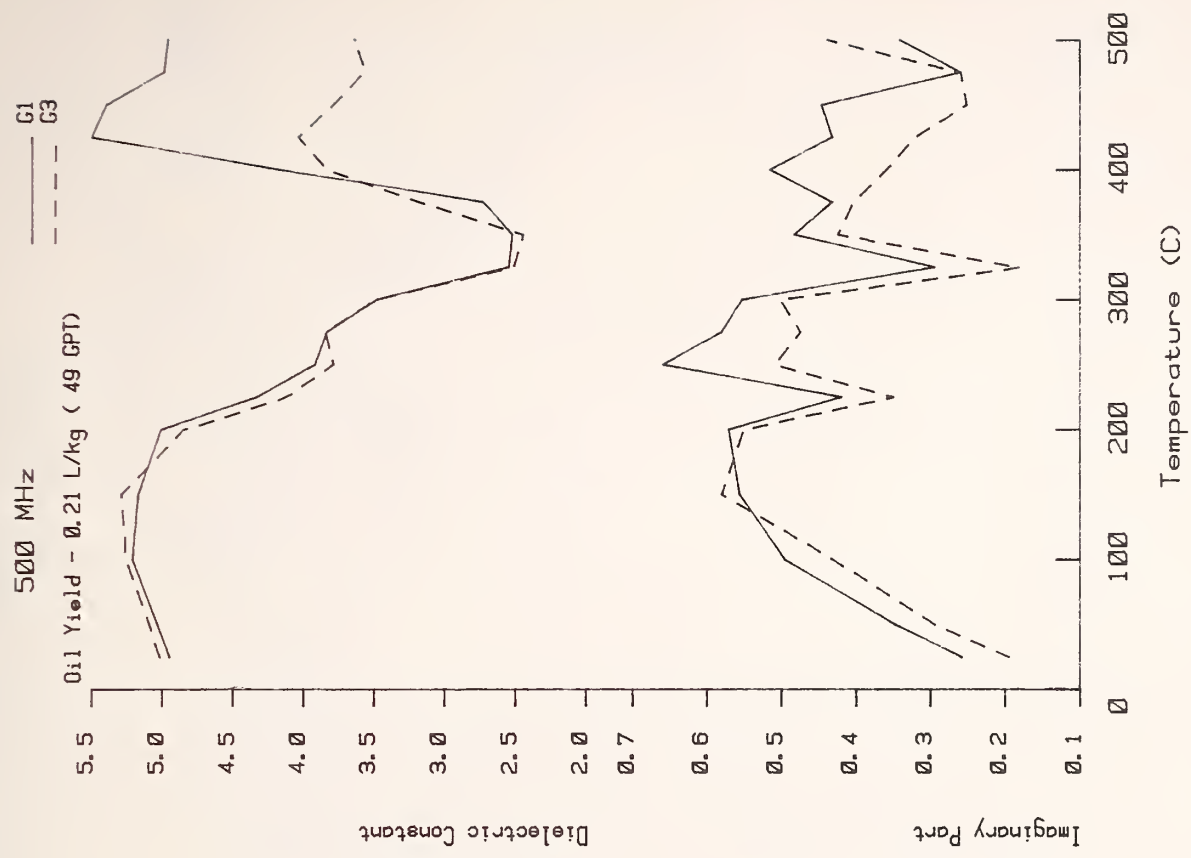


Figure 11. Plots of dielectric constant and imaginary part of designated samples D and G as a function of temperature at 500 MHz.

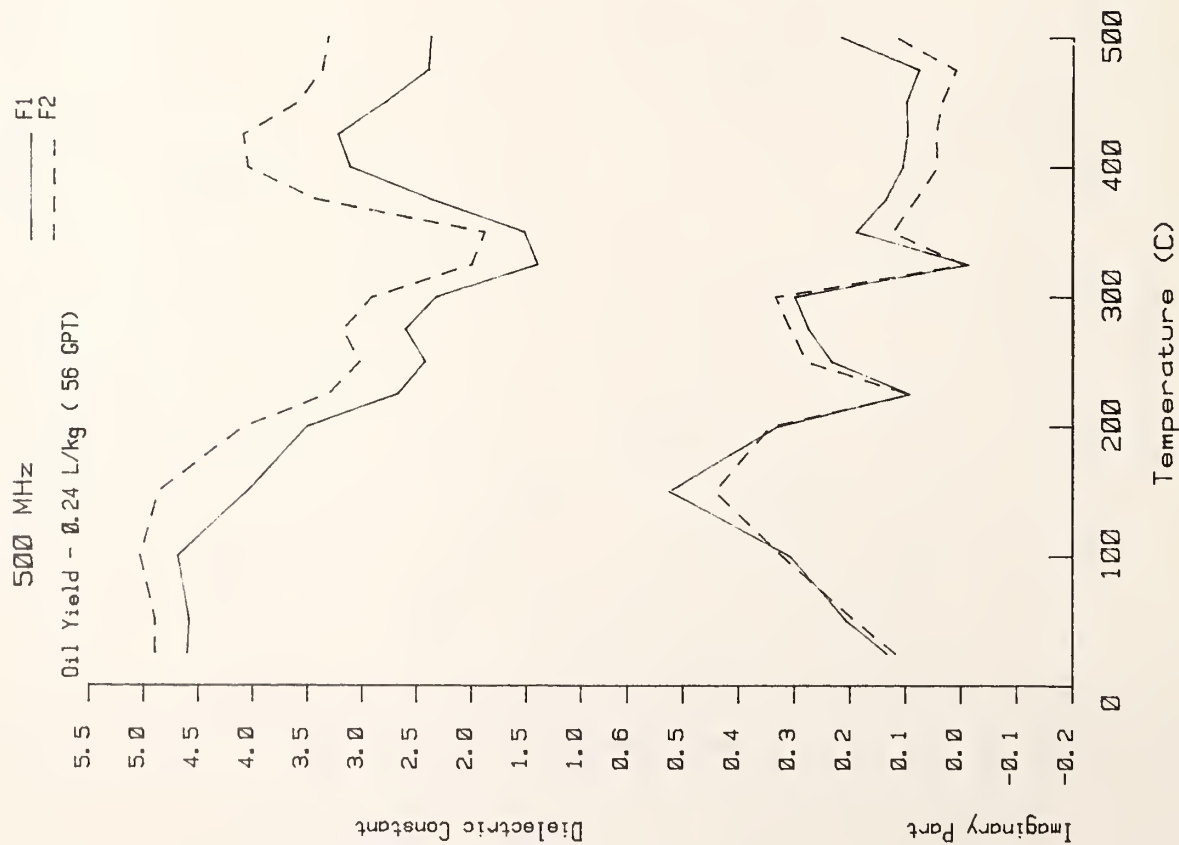
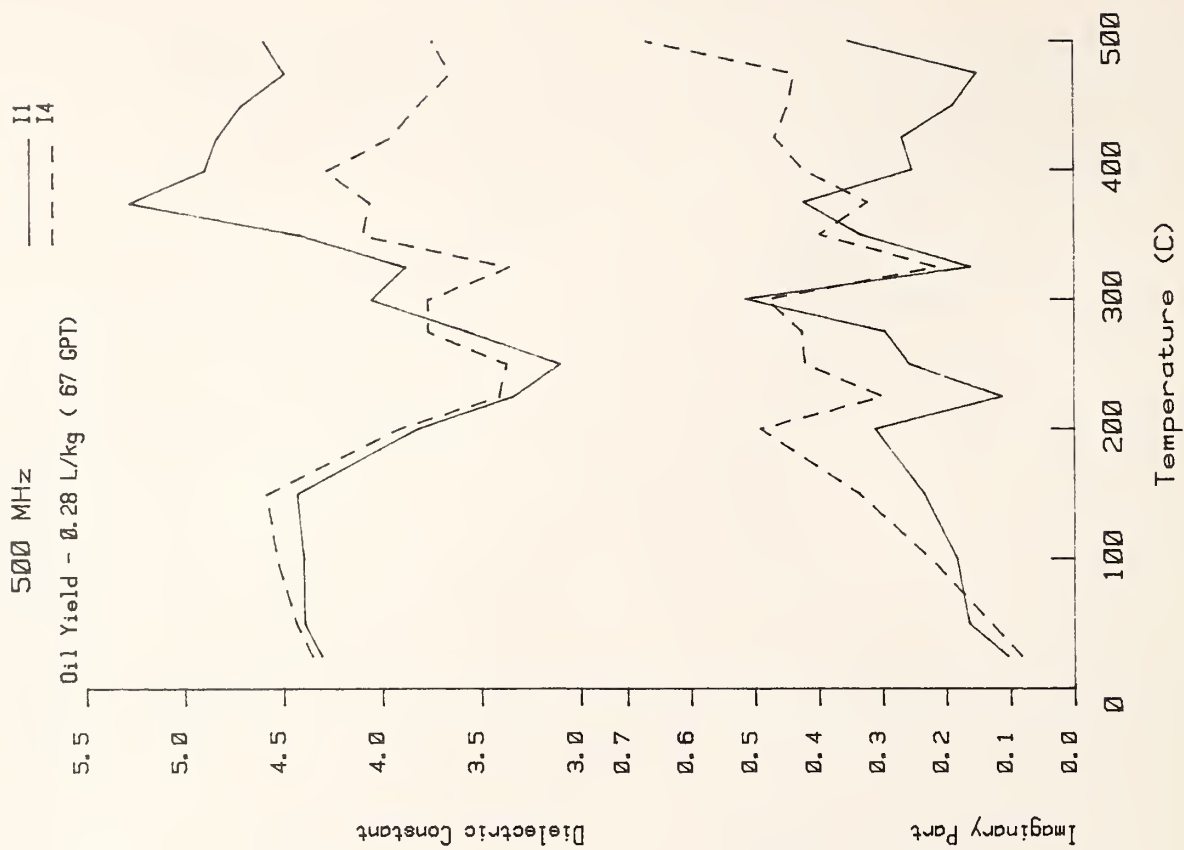


Figure 12. Plots of dielectric constant and imaginary part of designated samples F and I as a function of temperature at 500 MHz.

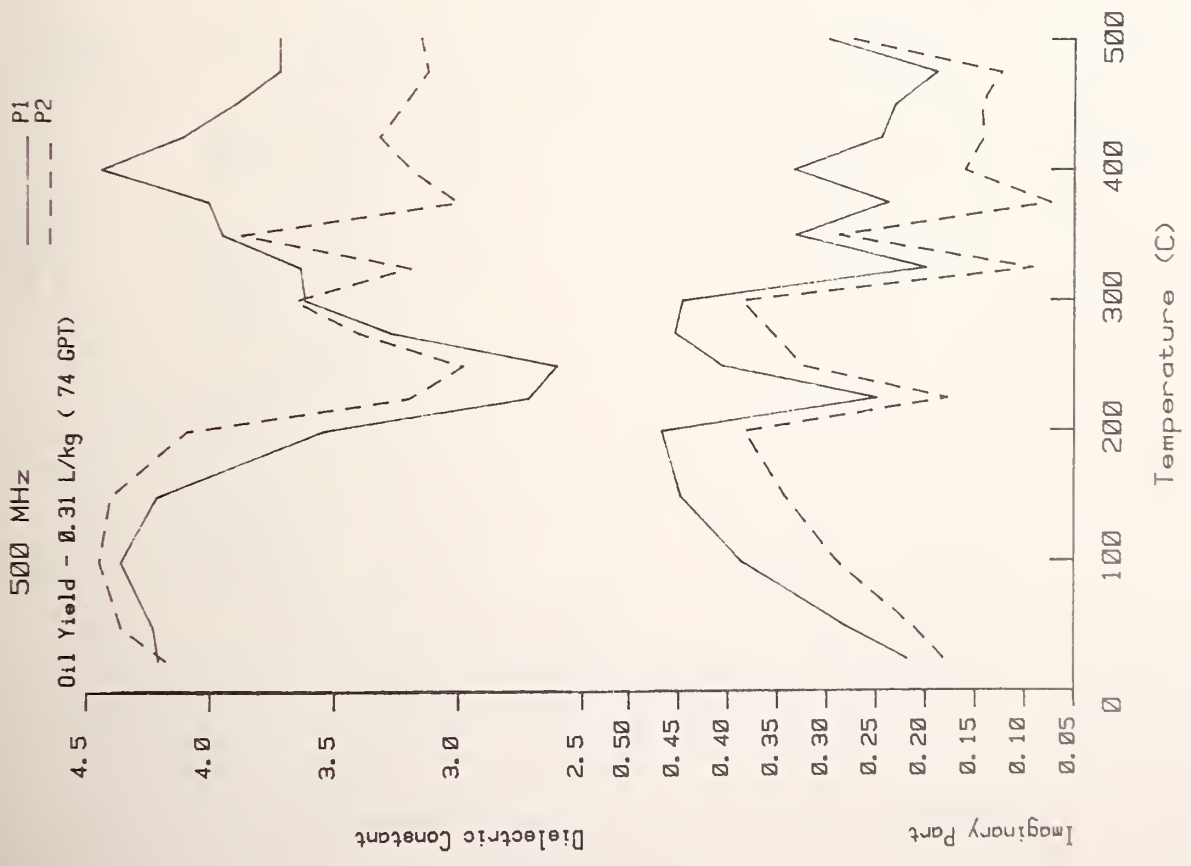
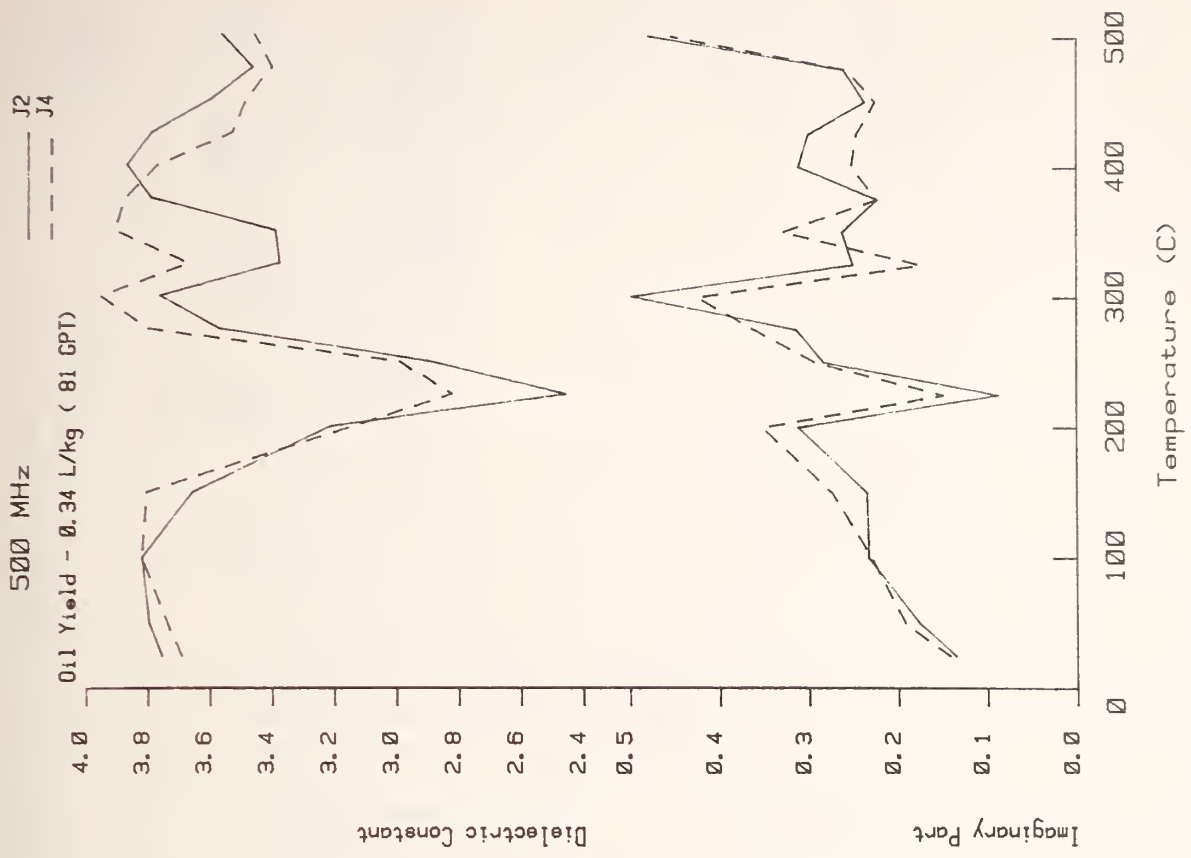


Figure 13. Plots of dielectric constant and imaginary part of designated samples P and J as a function of temperature at 500 MHz.

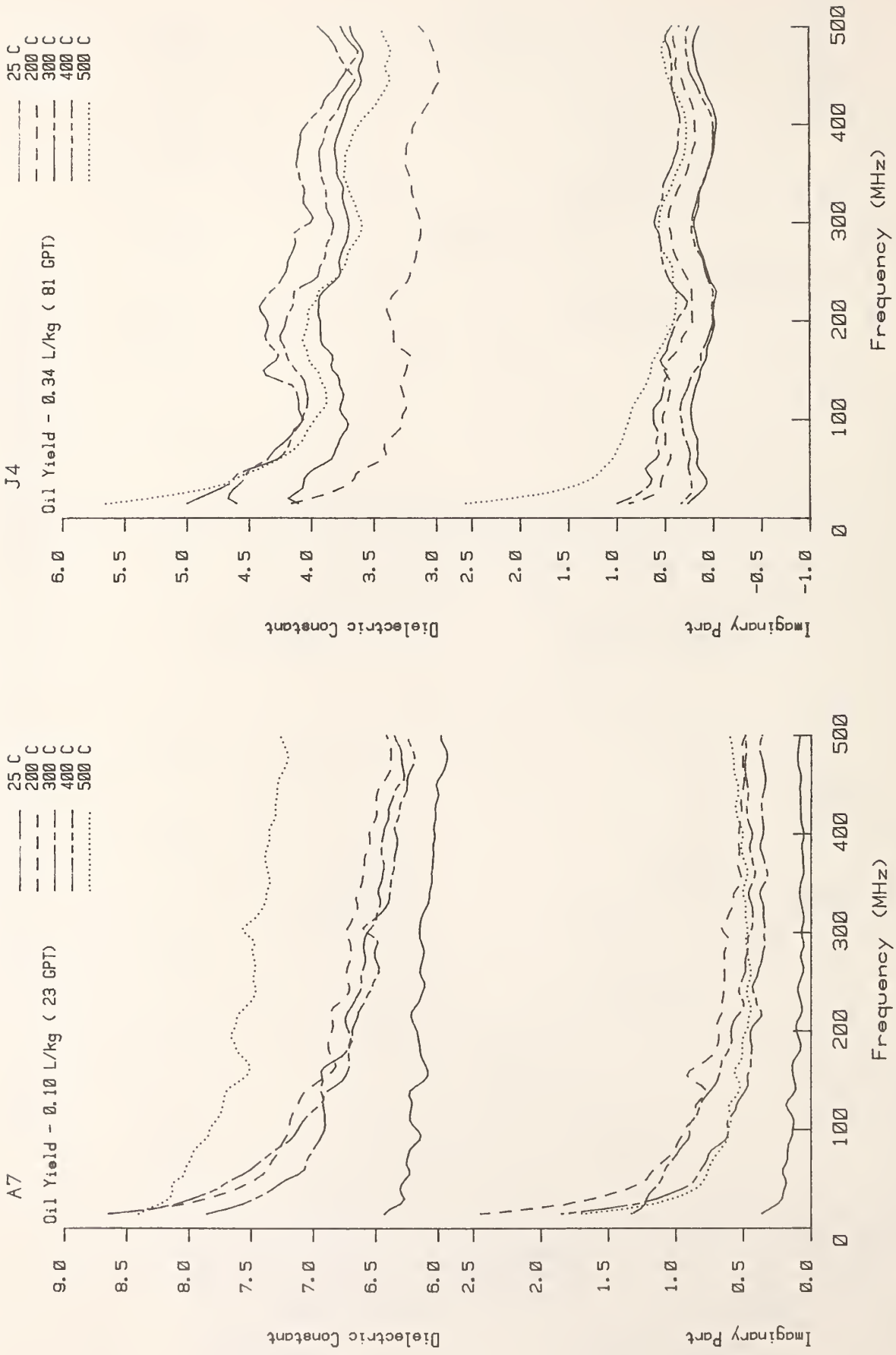


Figure 14. Comparison of temperature plots of dielectric constant and imaginary part of the lower yield A sample with those of the highest yield J sample as a function of frequency.

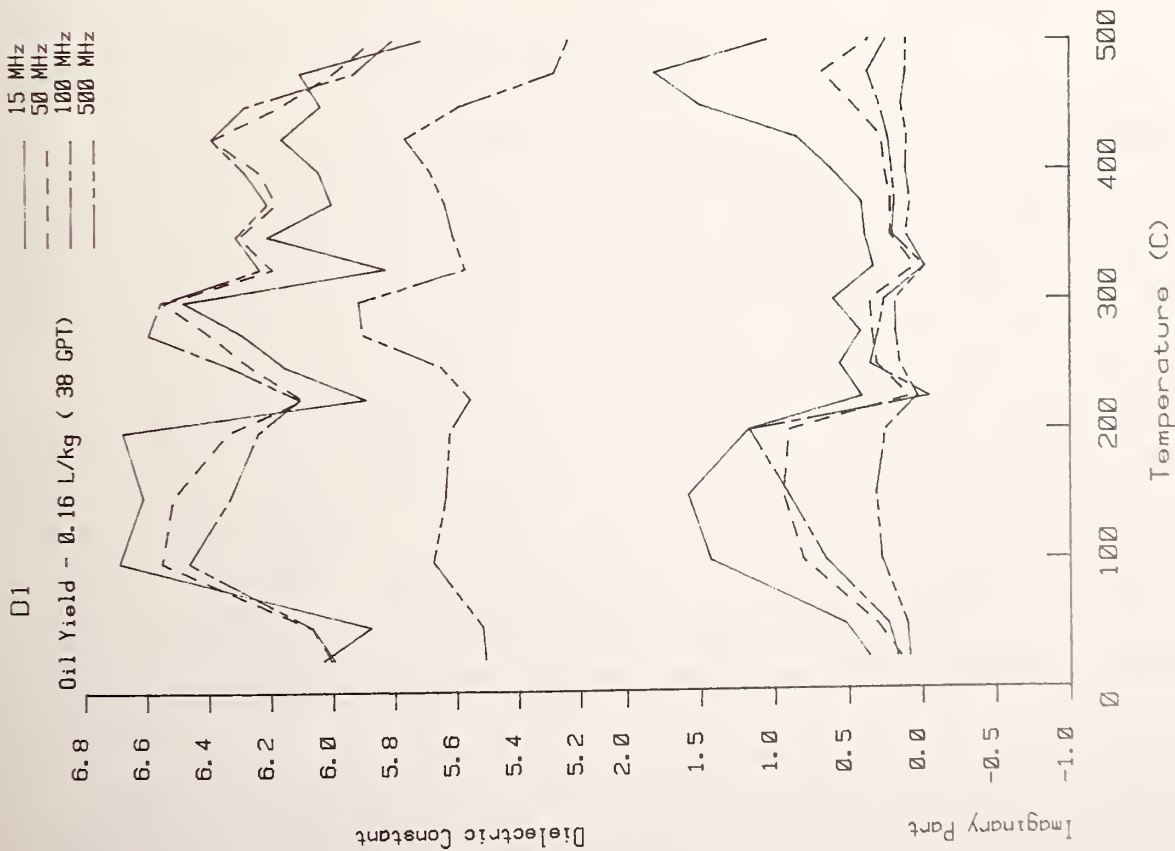
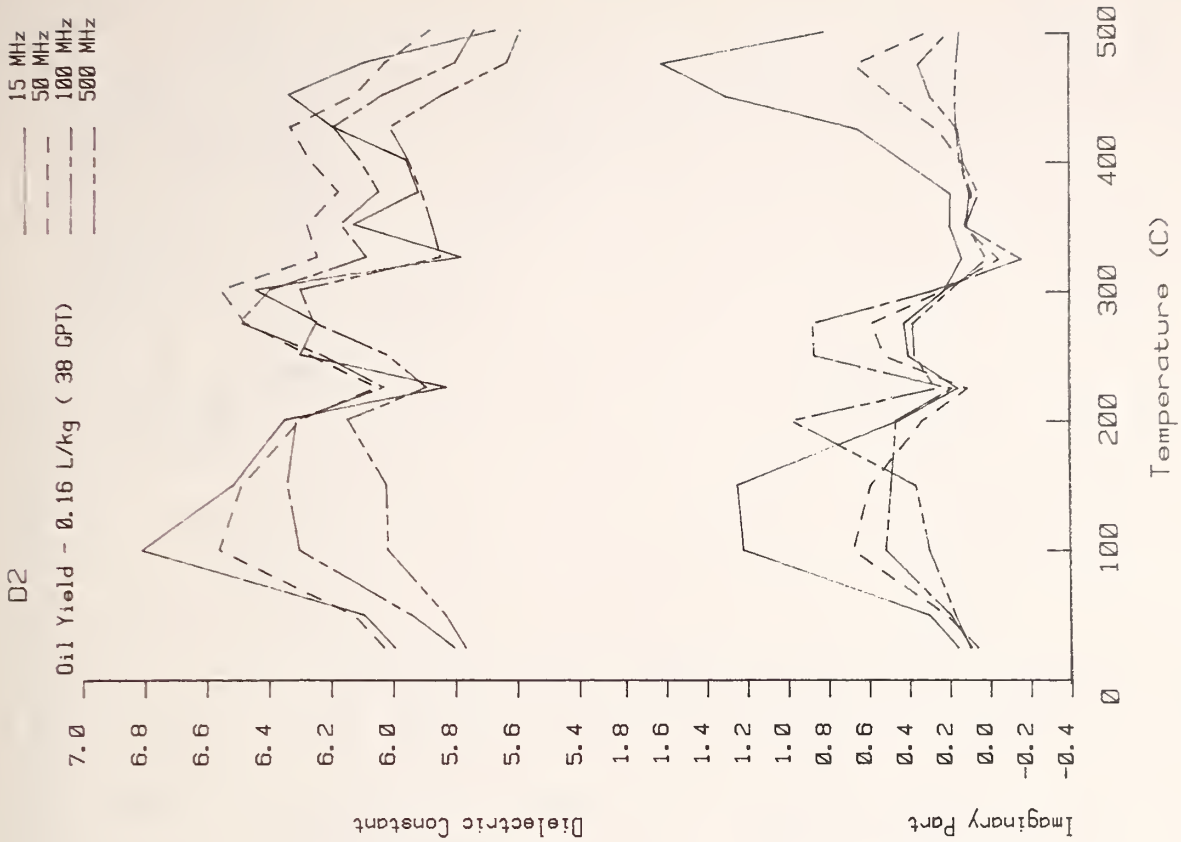


Figure 15. Frequency plots of dielectric constant and imaginary part of designated like samples D1 and D2 as a function of temperature.

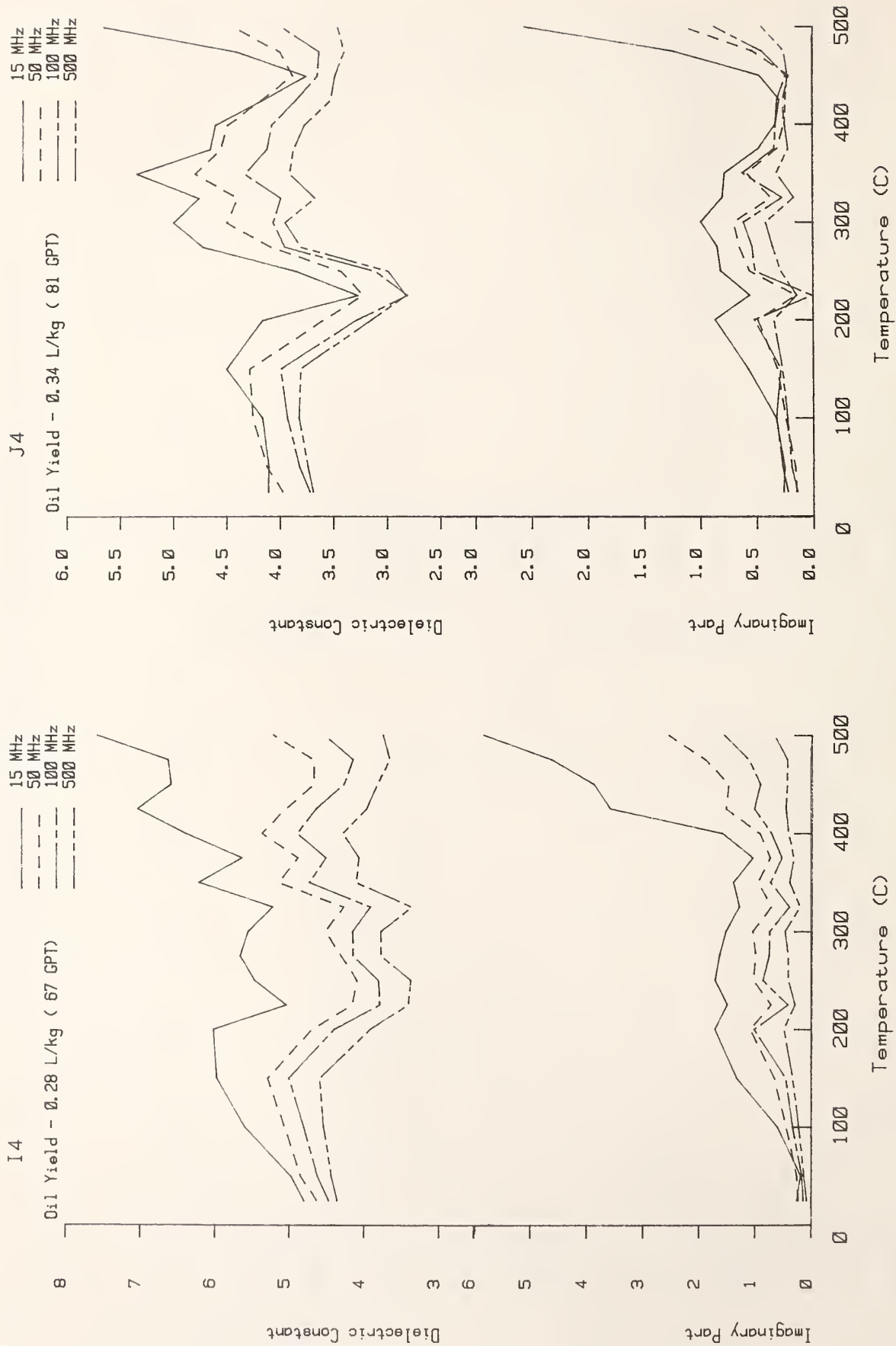


Figure 16. Frequency plots of dielectric constant and imaginary part of designated samples I and J as a function of temperature.

Figure 17 is given to point out some of the measurement problems encountered with some of the oil shale samples in the temperature range between about 150°C and 325°C as shown by the gaps in the data plots. For some reason, this temperature range appeared to be the most critical and troublesome for the sample holder. In this region, some of the values for the dielectric constant turned out to be quite low and, in a few cases, some dielectric constant values were less than 1, which is not possible. A thorough investigation of the sample holder revealed that the threaded portion of the center conductor (figure 2) had loosened slightly. Once this was tightened, some of the measurement data improved somewhat. Other measures were taken to clean all parts of the sample holder more thoroughly and frequently to remove the residual tar-like substance deposited from the shale oil. Increased amounts of this residual material from the shale oil was given off by the higher yield samples which created more of a problem with coking and plugging of the vent holes in the endplate by this tar-like substance. The cleaning of the sample holder after each measurement run seemed to improve the measurement results to a certain degree. In spite of the improved measurement results, there still existed a few gaps in the data plots.

3.1.3 Gas and Liquid Sample Collection

Chemical changes occurring during the heating of oil shale can be investigated by examining the gas and liquid products formed as a function of temperature during the heating process. Toward this end, gas and liquid samples were collected by the LETC at the NBS on designated oil shale samples E and S while they were being measured in the high-temperature sample holder. Gasses were sampled at the same time interval as the dielectric measurements were being taken by the NBS. Gas collection was made difficult by the loss of the pressure seal in the sample holder due to the shale expansion during heating. However, on a subsequent measurement run, data was collected using a thinner sample of oil shale and a spring arrangement to reduce the effect of shale expansion on the pressure seals. On this particular run, a tight pressure seal was maintained over the whole temperature cycle.

The collection system used for the oil and liquid samples is the apparatus shown in figure 18. The system was operated at or below atmospheric

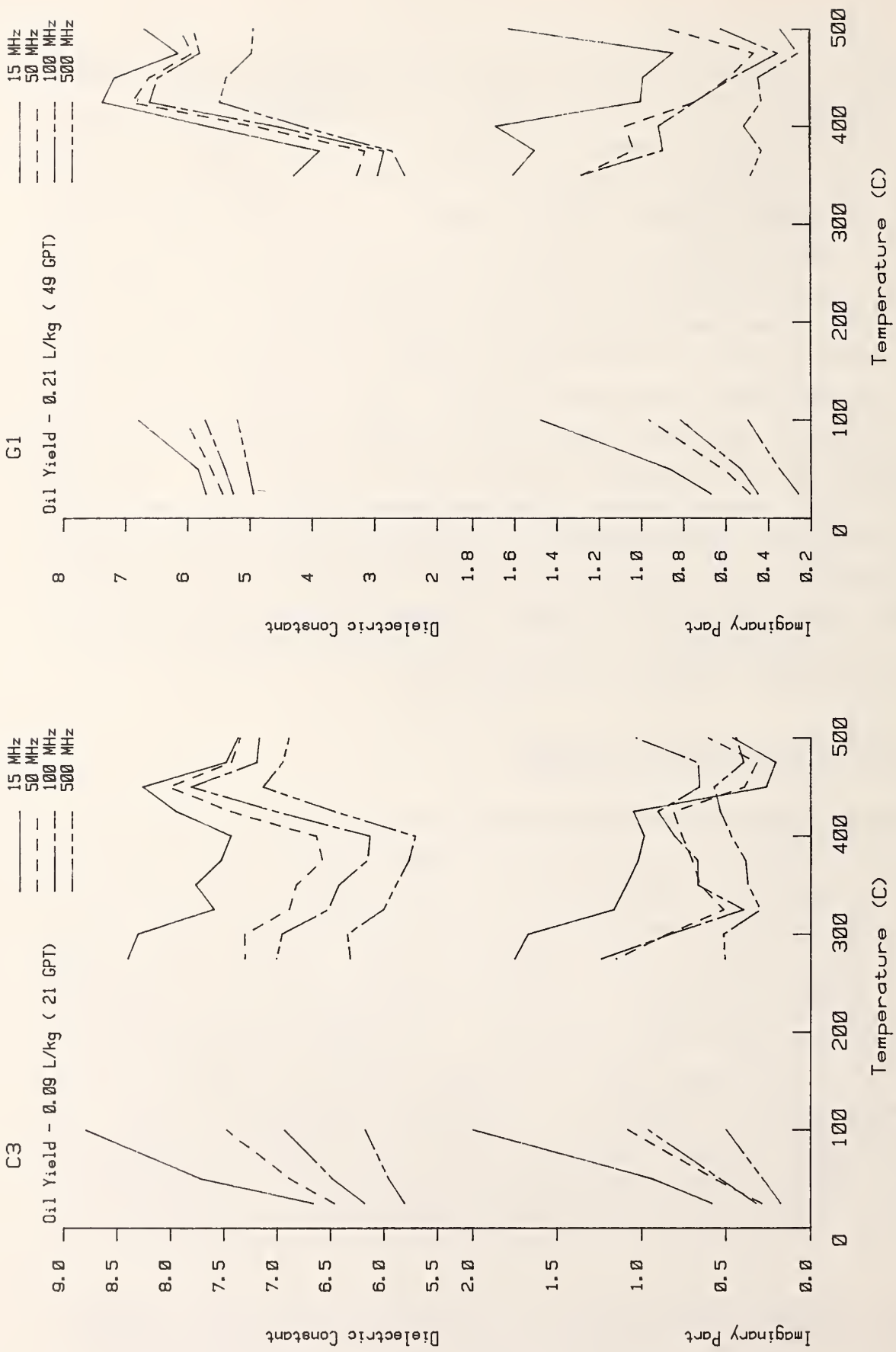


Figure 17. Frequency plots of dielectric constant and imaginary part of designated samples C and G as a function of temperature.

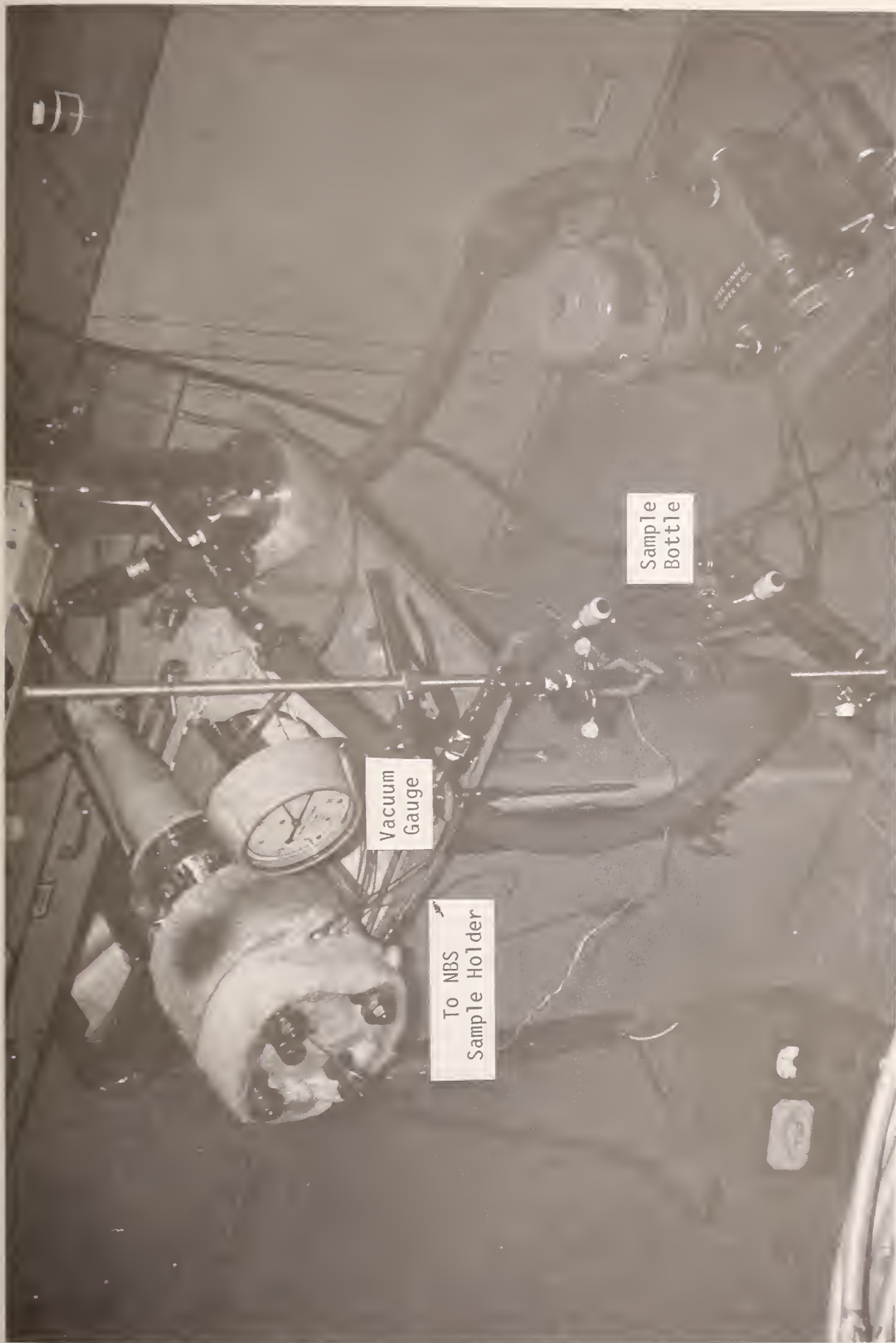


Figure 18. Gas and liquid collection system.

pressure because the volume of gas produced is small and so that use could be made of existing gas sample collection bottles and a gas analysis spectrometer. The Pyrex gas collection bottles were pre-evacuated to approximately 6.66 Pa (50 microns of mercury) and were connected onto the collection system with a short length of surgical tubing. The scheme of operation that was used for collecting the gas and liquid samples and their analysis is described in a report [6] by the LETC. At this reporting, no attempt has been made to intercompare the gas analysis results with the dielectric measurement results.

4. DATA SUMMARY

Making accurate permittivity measurements on oil shale samples at elevated temperatures is no simple task. While heating can change the physical properties and dimensions of the sample holder, it can also cause the oil shale to expand even more, creating problems with pressure seals in maintaining good electrical contact and in making accurate measurements over the whole temperature range difficult to achieve. Under actual measurement conditions, the unvented and pressurized conditions were difficult to attain with the present sample holder design. However, for the small amount of data that was available for analyzing, there doesn't appear to be very much difference between these data and the data collected for like samples in the vented condition.

The measurement uncertainty for the permittivity measurements on the oil shale samples varied from about 9 percent at 15 MHz for the values of dielectric constant down to about 2 1/4 percent at 500 MHz, while the uncertainty in the imaginary part was more than 200 percent at all frequencies.

5. CONCLUSIONS

A description of the high-temperature sample holder characterization is given along with the measurement procedure and the sample preparation. A measurement plot of measured and standard values of glass helps to validate the accuracy of the sample holder at elevated temperatures up to 500°C.

From the experiments performed on the oil shale samples, density and water content still appear to be two major factors that influence the permittivity of oil shale. Moreover, the results indicate that the dielectric

constant of the shale is not strongly influenced by the release of the shale oil at elevated temperatures as previously suspected. However, it does appear that the mechanical strength of oil shale does affect the dielectric values at about 400°C and above.

6. ACKNOWLEDGMENT

The authors wish to thank Raymond Montoya for his excellent job in preparing the high-temperature sample holder and making the elevated temperature measurements.

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11. ABSTRACT <i>(A 200-word or less factual summary of most significant information. If document includes a significant bibliography or literature survey, mention it here)</i> A high-temperature sample holder designed by the National Bureau of Standards was used to determine the dielectric properties of approximately 40 oil shale samples as functions of temperature and frequency. A description of the sample holder characterization is given along with the measurement procedure and the sample preparation. Experimental results are given for different oil shale samples, varying in yield from 0.024 L/kg (6 gal/ton) to 0.338 L/kg (81 gal/ton), that were measured at room temperature (25 °C) up to 500 °C and within the frequency range of 5 - 1000 MHz.			
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