Devices & Services Co.

D&S Technical Note 84-3

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Measurements on Low Emittance Materials with the Model AE Emissometer

Introduction

This technical note concerns the use of the D&S Emissometer Model AE for making production emittance measurements on low emittance materials, where it is desired to improve the repeatability and resolution of the measurement. The sources of measurement errors, including sample temperature, detector temperature and sample positioning are discussed. Test data are presented which provide quantitative information about these errors. Also discussed are increasing the resolution of the Model RD1 Voltmeter from 0.01 to 0.001 and improving the repeatability of the emittance measurement. Although a single procedure will not work in every case, guidelines are recommended for making emittance measurements in a production environment. It is often possible to simplify the measurement procedure while retaining the improved resolution.

This note does not consider those factors that determine how the AE measurements compare with normal or hemispherical emittance values obtained with other test methods. These factors include the wavelength dependence and directional dependence of the detector that cause deviations from the ideal detector response for surfaces that have variations in emittance with wavelength or direction. Only the repeatability of the measurements will be considered.

Background

The design of the Emissometer detector enables the net output to be nearly linear with emittance over a wide range of emittance values. Using two calibration standards maintained at the same temperature it is possible to scale the output of the detector to read emittance. However, if the sample temperature is not the same as the standards, the linear relationship between the detector output and emittance is no longer valid.

For this reason, the Emissometer was originally designed as a table top unit to measure the emittance of small samples of flat materials that could be easily placed on a flat heat sink. The heat sink assures that the standards and the samples will be at the same temperature (near ambient). However, some special techniques have been developed for making measurements of surfaces in place, surfaces of non thermally conductive materials and non flat surfaces. The following Technical Notes describe how to handle these special measurements.

TN 79-17 Emissivity Measurements for In-Place Surfaces and for Materials with Low Thermal Conductivity
TN 81-1 Use of the Emissometer for Semi-Transparent Materials Measurements

The AE also has been used in production environments to monitor batch or continuous plating and coating processes. Frequent calibration is inconvenient in this environment and it may be impossible to accurately control the sample temperature. Also in the case of low emittance materials it would be useful to be able to improve the 0.01 resolution of the instrument as it is supplied, to 0.001 if possible, and to be able to make more repeatable measurements.

Model AE measurement errors can be related to one of three possible causes: detector drift, due to thermal or mechanical changes; sample temperature; or sample positioning relative to the detector. Although these errors are usually small, they must be understood and reduced to an acceptable level in order to improve repeatability.

Detector Drift: As stated above, the detector output is approximately linear with emittance and is dependent on sample temperature. The equation below can be used to approximate the detector output.

$$D = K1\ (T_s^4 - T_d^4)\ E_s + K2 \tag{1}$$

where,

D - detector output
E_s - sample emittance
T_s - sample temperature
T_d - detector temperature
K1 - detector constant
K2 - offset

Since the detector temperature (T_d) is controlled at a constant level and assuming that the sample temperature (T_s) is constant,

$$D = K\ E_s + K2 \tag{2}$$

where, K - gain

This is the linear relationship between detector output and emittance. After the Emissometer is calibrated, the offset (K2) should be zero.

Independent of the sample temperature, there will be a minor drift in detector output due to changes in the temperature control point (T_d) of the detector. Although a resistive element controls the detector temperature, the control is not perfect. As the total heat loss from the detector decreases, the detector temperature will increase slightly. This results in a slight increase in the detector output. An increase in the heat load will cause a similar decrease in detector temperature and a decrease in the output. A change in the heat load on the detector can be caused by a change in ambient temperature, air velocity, sample temperature and even sample emittance. This temperature change will appear as a drift in gain.

Another source of drift in both gain and offset is mechanical change or degradation of the thermopile detector or housing. These changes along with the changes in ambient conditions as described above, usually occur gradually and are eliminated by routine calibration.

One source of drift in gain that is not eliminated by calibration is the change in detector temperature (T_d) that occurs when the detector is placed on a sample with low emittance. Normally the detector rests on the high emittance standard and the temperature reaches a steady level. When moved to the low emittance material the heat load is suddenly decreased, and the detector temperature moves up slowly. Upon returning the detector to the high emittance standard the output will overshoot and then slowly return to near the original level. This has only a small effect on the emittance readings, for example a 3% overshoot on the high emittance standard amounts to an error of only 0.003 in emittance for a sample with an emittance of 0.10.

The overshoot is smaller if the difference in emittance between the two materials is smaller. It is therefore possible to minimize the overshoot error by using standards with emittance values close to the samples to be measured.

Sample Temperature: The sample temperature obviously affects the detector gain (equation 1) and can also possibly cause a shift in the offset constant. If the change is gradual, routine calibration compensates for the sample temperature, as long as the standards are at the same temperature as the sample. Special procedures have been developed for measuring non-conductive materials and in-place surfaces for which the surface temperature cannot be controlled and may change during a measurement due to heating by the detector. Technical Note 79-17 describes techniques for making these measurements.

In a production environment these procedures may be too complicated and time consuming. It would be preferable to be able to correct for the surface temperature or reduce the size of the error.

Sample Positioning Relative to the detector: The output of the detector is also affected by the position of the sample with respect to the detector. Ideally the sample should be flat and in perfect contact with the circular port of the Emissometer. In practice this is rarely the case. If the sample is not flat or the Emissometer does not rest flatly against the sample, two factors can contribute to error in the reading. One is an error in the radiation heat transfer between the low and high emittance detector elements and the sample. Assuming that the calibration samples are flat and the Emissometer rests flatly against the standards during calibration, then the radiation view factors between the detector elements and the sample are the same for both standards. If the sample position is not the same the view factors will be different resulting in an error in the reading.

The other factor is a similar change in the conduction and convection heat transfer to the detector elements if the surface is uneven or out of position. If the sample is on the average closer to either the low or high emittance detector elements, the heat transfer between the sample and those detector elements will be greater causing an offset in the reading. The Emissometer is designed to minimize this imbalance, but the larger the deviation in the relationship between the detector/standard combination and the detector/sample combination, the larger the error.

Each of the factors contributing to the uncertainty in the AE detector output, although usually small, become important when attempting to increase the resolution of the instrument. The test data presented in the following section covers each of these sources of error.

Test Data

Detector Drift with Ambient Temperature: The test for drift with ambient temperature was conducted in a temperature controlled oven with a circulating fan. Three Emissometers were tested simultaneously. The units were calibrated at near room temperature and then the ambient temperature was changed. No further adjustments to the calibration were made. At each set point, the Emissometers were allowed to come to equilibrium with the surroundings. To make sure that steady state readings were obtained, data points were also taken at several set points as the oven was cooled from the maximum temperature of 115 F. The data is presented in Figure 1, showing indicated emittance versus ambient temperature.

Although three Emissometers were tested there was very little scatter in the data. Test points for all three units are plotted in the figure. The data shows that a change in ambient temperature primarily affects the detector gain and has almost no effect on the offset. The indicated emittance for the low emittance sample shows very little change and the slightly downward trend indicated can be attributed to the gain change. Equation (2) given above can be used to model the detector output. Using the two regression lines in the figure to represent the data, the gain and offset constants were calculated for the 115 F maximum test temperature. The gain decreased to a value of 0.88 times that at the 78 F calibration temperature, but the offset shifted by only 0.001 emittance units (well within the experimental uncertainty).

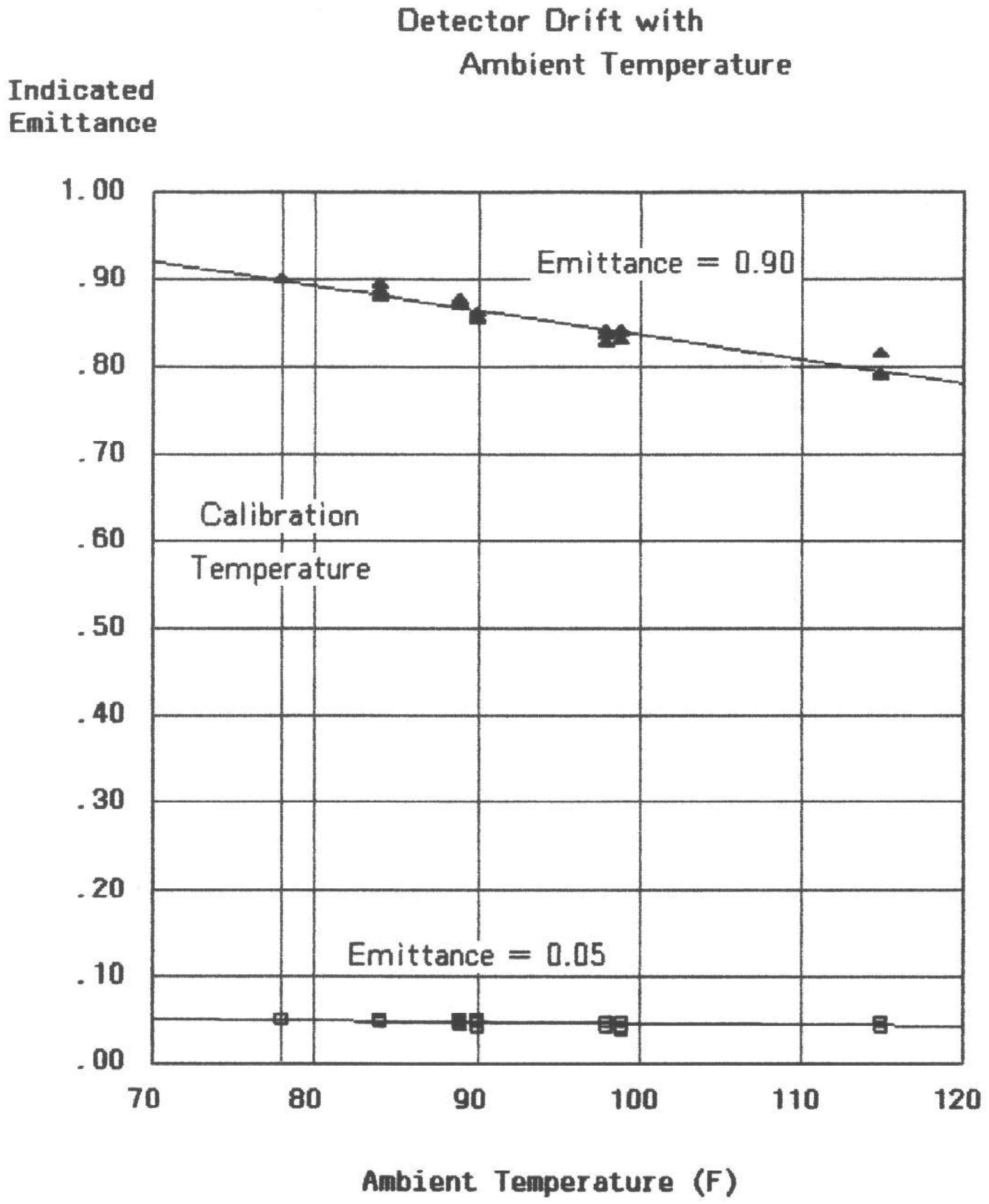


Figure 1. Detector Drift with Ambient Temperature

Output Overshoot: As described above, output overshoot occurs when the Emissometer is placed on a low emittance material for some time and then returned to the high emittance standard. It is caused by a slight increase in the detector temperature due to the decrease in heat loss and affects the detector gain. A test for output overshoot was devised using a 0.90 emittance material as the high emittance standard in one case and a sample of stainless steel with an emittance of 0.164 in the other. In both cases a 0.05 emittance material was used as the low emittance standard, and also as the low emittance test sample. The detector was allowed about 30 minutes on the high emittance material to reach a steady state condition. A gain adjustment was made on the voltmeter if necessary to adjust the reading to the emittance value of the standard. Then the detector was placed on the low emittance sample for various lengths of time, and then placed back on the high emittance standard. The peak reading on the high emittance standard occurs about 90 seconds later. The difference between the peak value and the emittance value set for the standard is the overshoot. The data is summarized in Figure 2. Note that the percent overshoot is the percentage of the high emittance standard in each case and not 0.01 emittance units.

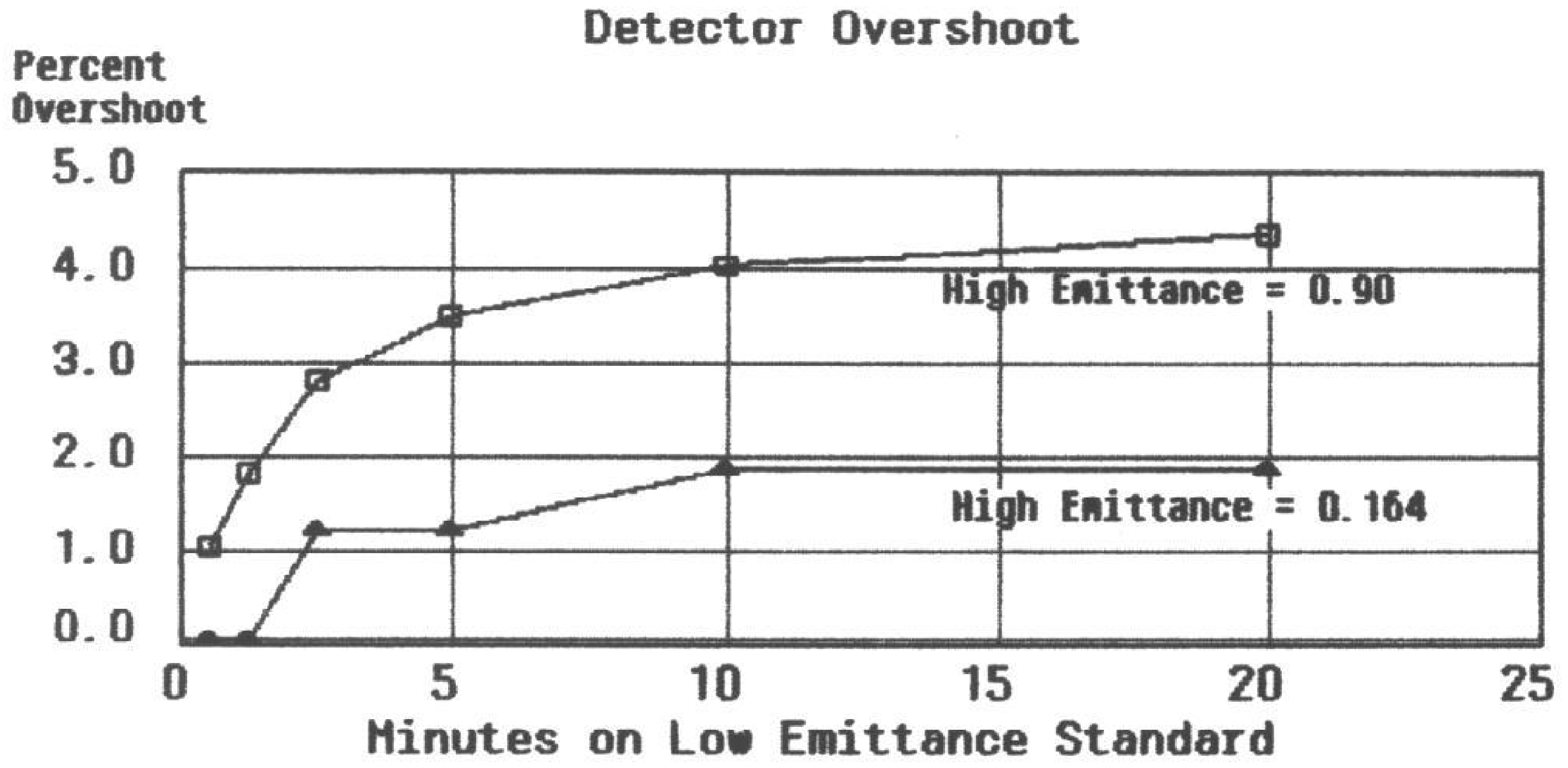


Figure 2. Detector Overshot versus High Emittance Standard Value

Because a measurement takes approximately 90 seconds with the Model AE, the overshoot cannot be limited to a value less than that indicated at about the two minute mark in the figure. Taking the steady state value for both the 0.90 and the 0.164 emittance standards as the worst case, the overshoot is decreased by better than a factor of two by using a lower emittance standard. If the material being measured has an emittance less than 0.20 the overshoot error can be reduced to less than two percent of the emittance value.

Using the lower emittance standard has the added advantage of reducing the temperature rise of the heat sink, thus improving the stability of the calibration. In still air the temperature rise of the heat sink is about 3 F with a 0.16 emittance standard and about 5 F with a 0.90 emittance standard.

Long term drift: Data for long term drift was taken under conditions typical of the way in which the Emissometer is normally used. The ambient temperatures were not controlled, but the unit was left on continuously. The Emissometer was warmed up for about an hour as described in the instructions and then calibrated on samples applied to the heat sink. The heat sink temperature was monitored during the test. Figure 3. summarizes the test data.

Over the test period the heat sink temperature drifted up significantly. This was in part due to an increase in the ambient temperature, but also due to heating by the detector. When the temperature reached a near steady level there was very little additional drift in the detector output, indicating that most of the drift is due to changes in the sample and ambient temperatures. Again the drift appears to be largely gain drift rather than offset. The test was continued for several days with little additional change in the emittance readings.

Sample Temperature: Sample temperature dependence was established using the same temperature controlled oven in this case to control the heat sink at a set temperature. Three Emissometers were set up and calibrated on a separate heat sink at room temperature. To get the data points, the Emissometer was placed in the oven on the test samples just long enough to reach a steady reading and then returned to the standard at room temperature. This is similar to a production test environment where the material or coating is processed at an elevated temperature and has not reached ambient conditions when the emittance is measured. The test results for the three detectors showed little variation and as with the ambient temperature test, the data for all three are plotted together in Figure 4. Also similar to the ambient temperature test, the sample temperature appears to affect only the gain and not the offset. Using equation (2) to model the detector output and the two regression lines as before, the data indicates a decrease in gain by a factor of 0.825 and a offset shift of only 0.009 at 111 F. It is interesting that the decrease in gain is greater when only the sample temperature is changed compared to when the ambient temperature and sample temperature both change in the same manner. This occurs because if the detector is also subjected to the higher ambient temperature, the increase in detector temperature compensates somewhat for the increase in sample temperature.

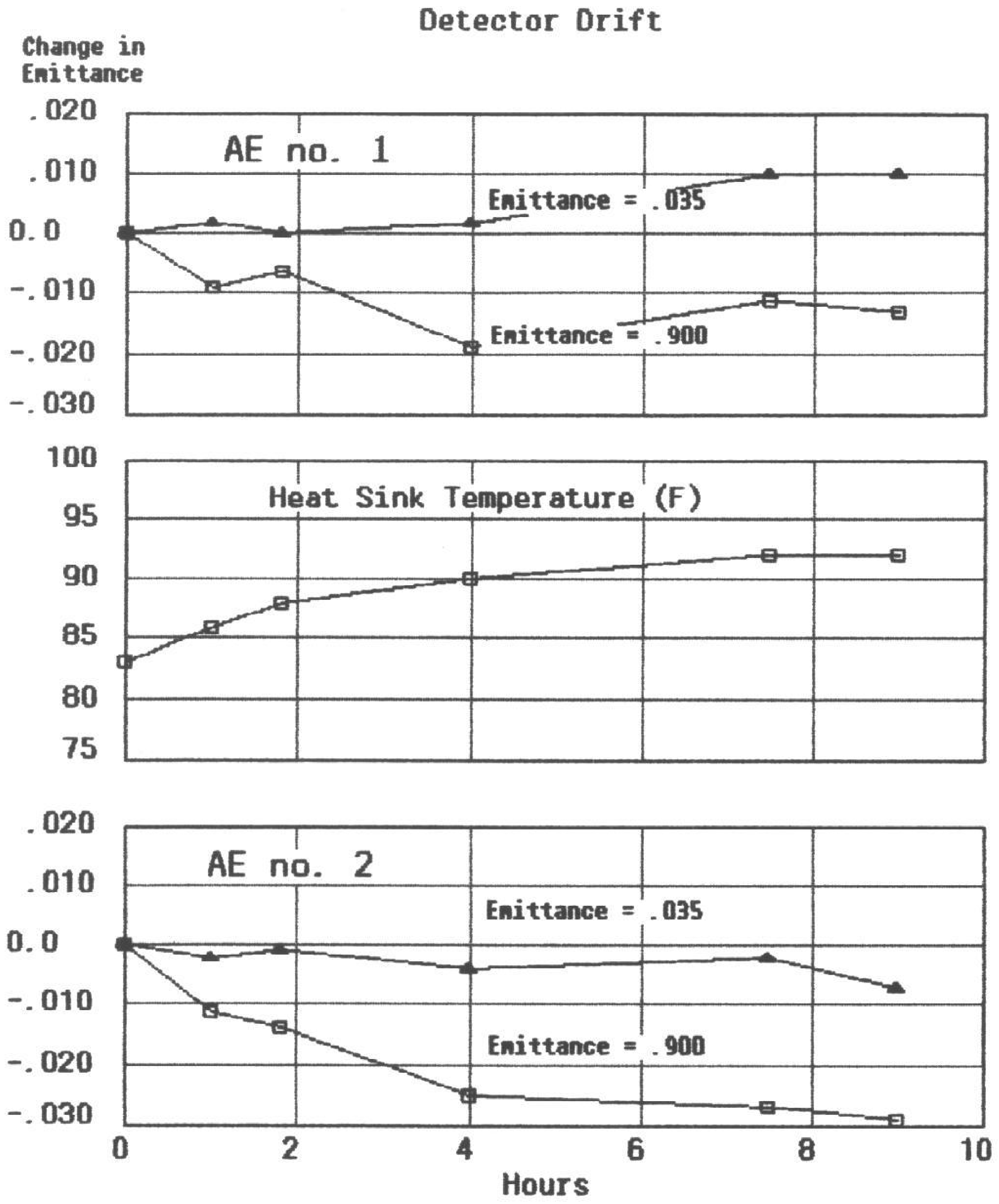


Figure 3. Long Term Detector Drift

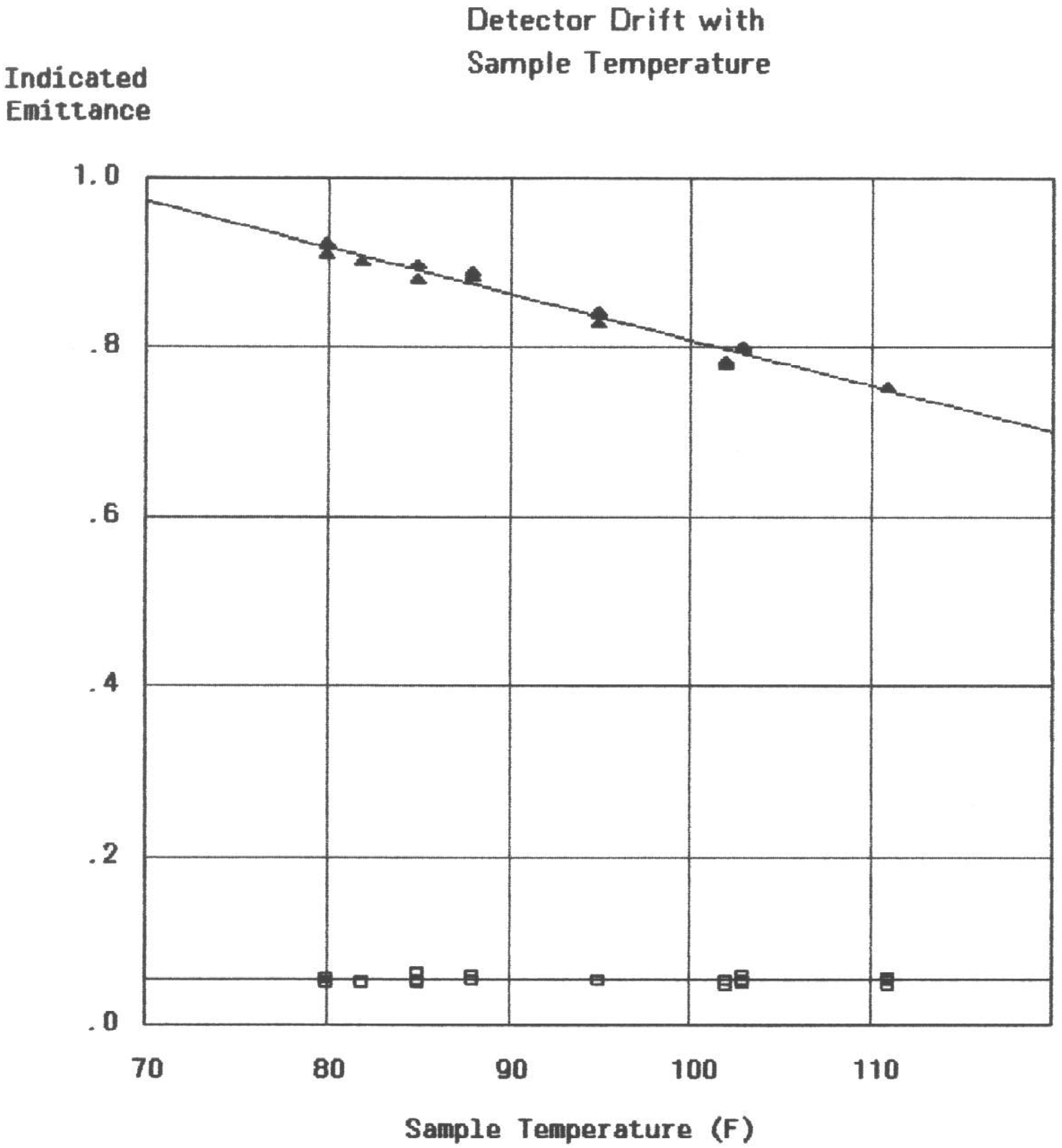


Figure 4. Detector Output as a Function of Sample Temperature

Sample Positioning: Two tests for sample positioning errors were conducted. One utilized a very flat sample with an error introduced by tilting the detector up on one side. A reference point and azimuth relative to the detector elements was established to determine if the position of the elements affect the tilt error. The azimuth angle is defined in Figure 5. The Emissometer was tilted in a vertical plane passing through the azimuth line. Three samples were tested to determine if the error is a function of emittance.

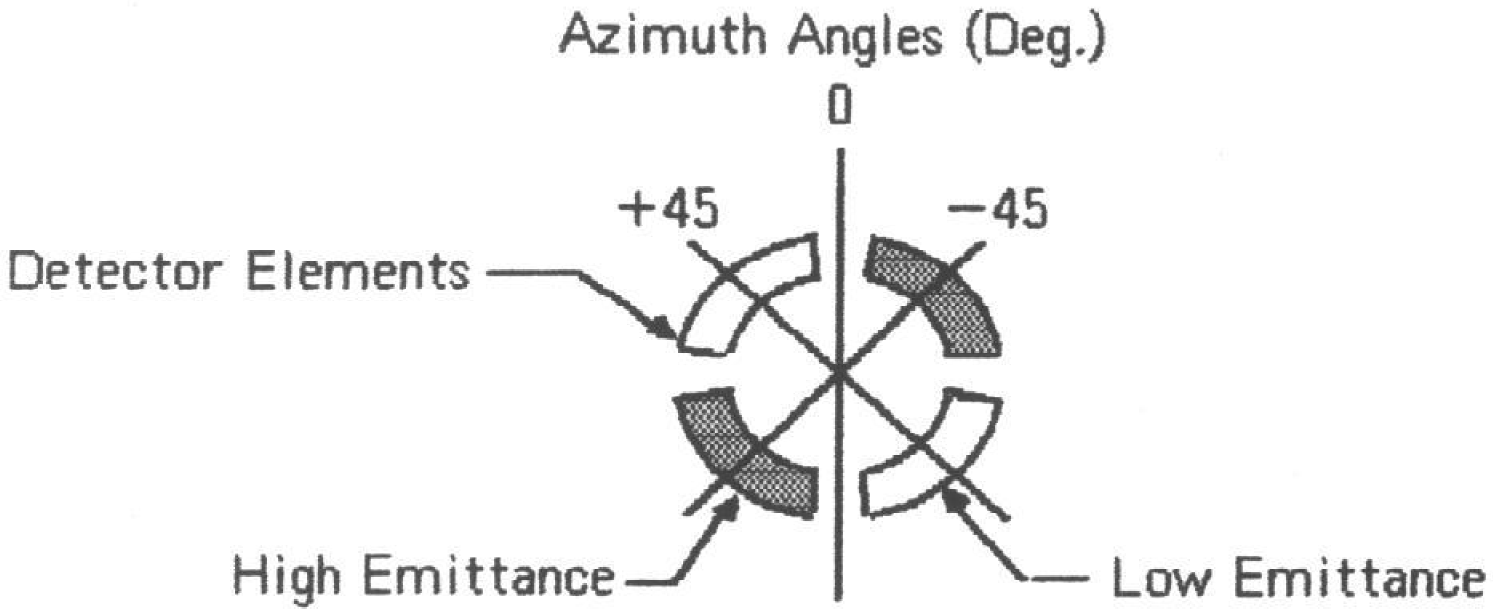


Figure 5. Azimuth Angles for Tilt Error Measurements

The data shown in Figure 6 indicates some dependence on azimuth, but is inconclusive, possibly due to uncertainty in the data. It was anticipated that this would determine what part of the error was due to differences in radiation view factors, and what part was due to the imbalance in the conduction and convection heat transfer to the detector elements.

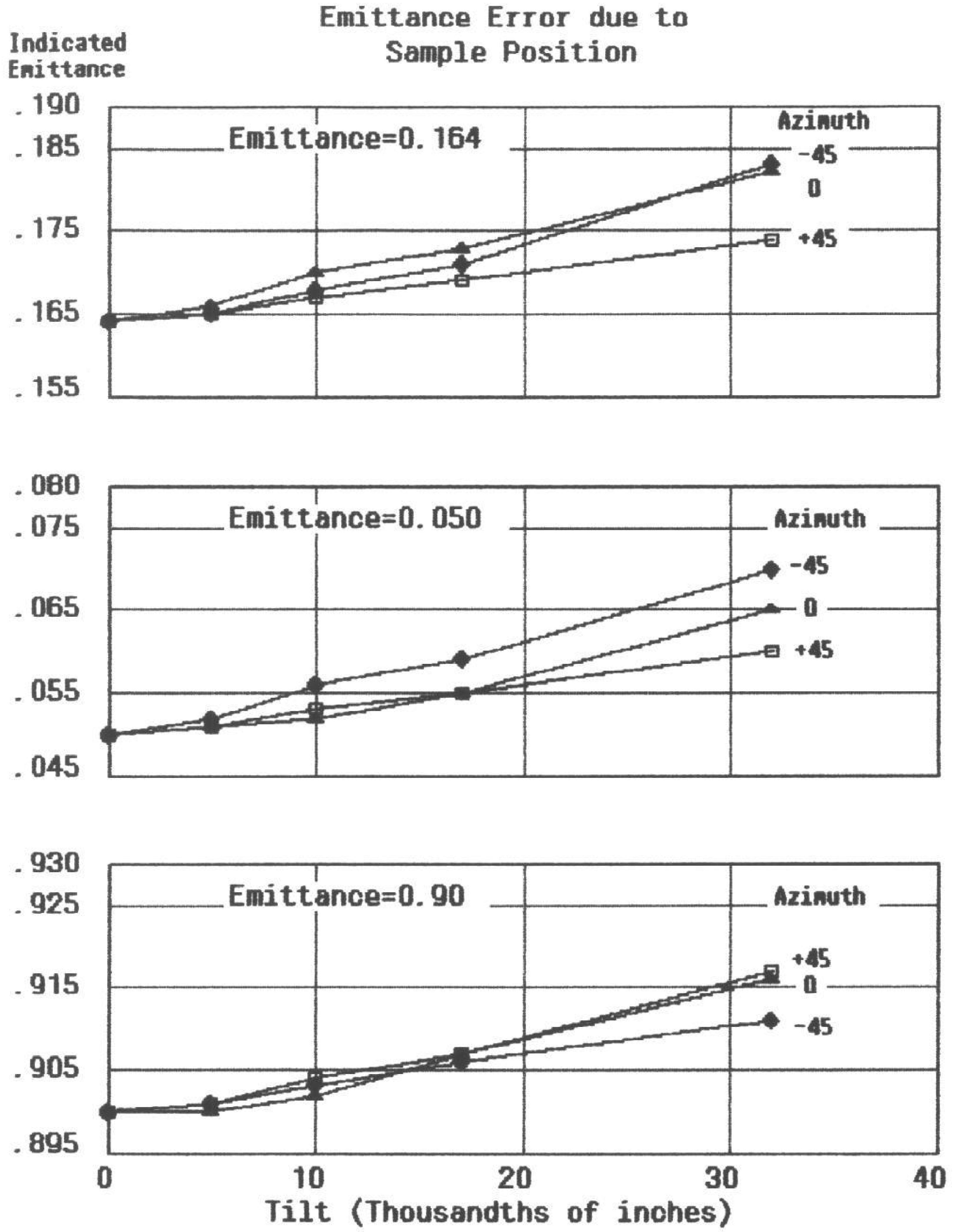
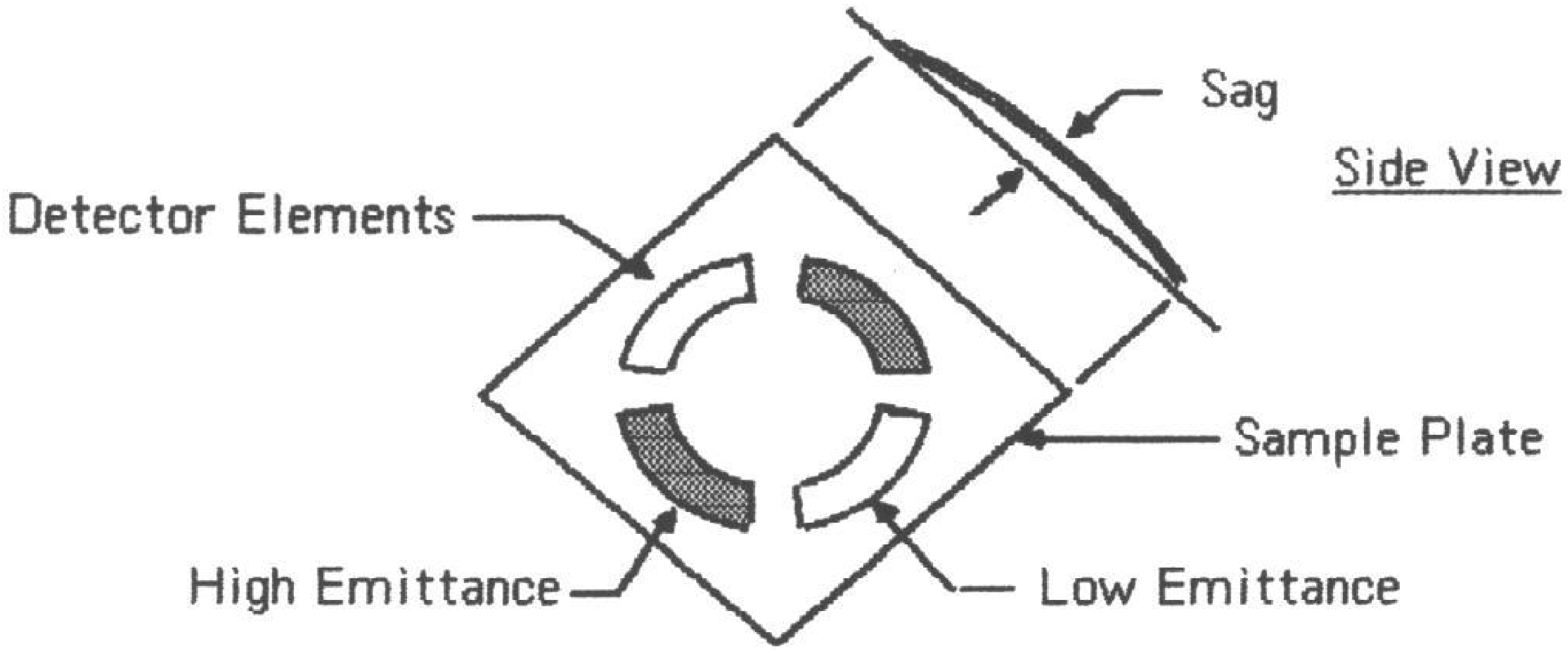
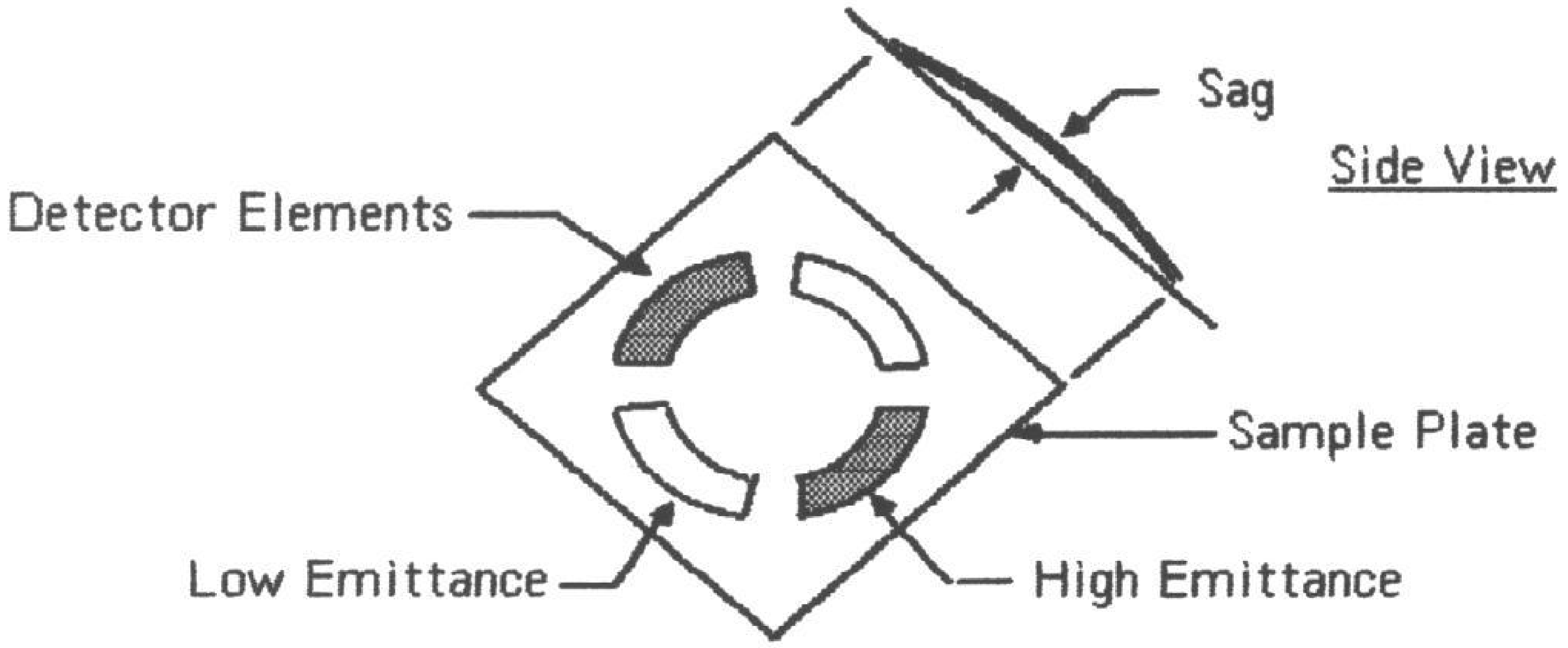


Figure 6. Emittance Error versus Tilt

A second set of data was taken to determine the effect of sample curvature, that being the most common problem with test samples. A number of identical samples were prepared on three inch square samples that had relatively uniform curvature across the surface. Fourteen of these samples with a range of curvature were selected for testing. In every case the samples showed a minimum and maximum emittance reading depending on the position of the detector elements relative to the bow or sag in the sample. Figure 7. shows the position of the minimum and maximum readings.



Orientation for Minimum Output



Orientation for Maximum Output

Figure 7. Position of the Minimum and Maximum Readings for a Bowed Sample

With the high emittance detector elements in line with the direction of the sag, these elements are closer to the surface and therefore the heat loss by conduction and convection is larger than that from the low emittance elements. The high emittance elements are the low temperature side of the thermopile detector therefore the output in this orientation is a maximum. The opposite is true when the detector is rotated by 90 degrees.

The difference between the minimum and maximum readings is plotted in Figure 8. The sag plotted on the horizontal axis was determined as shown in Figure 7, using a straight edge and feeler gages. A more commonly used measure of sag is the sag per inch of width. This can be determined by dividing the sag values in the figure by 3 since a three inch square sample was used.

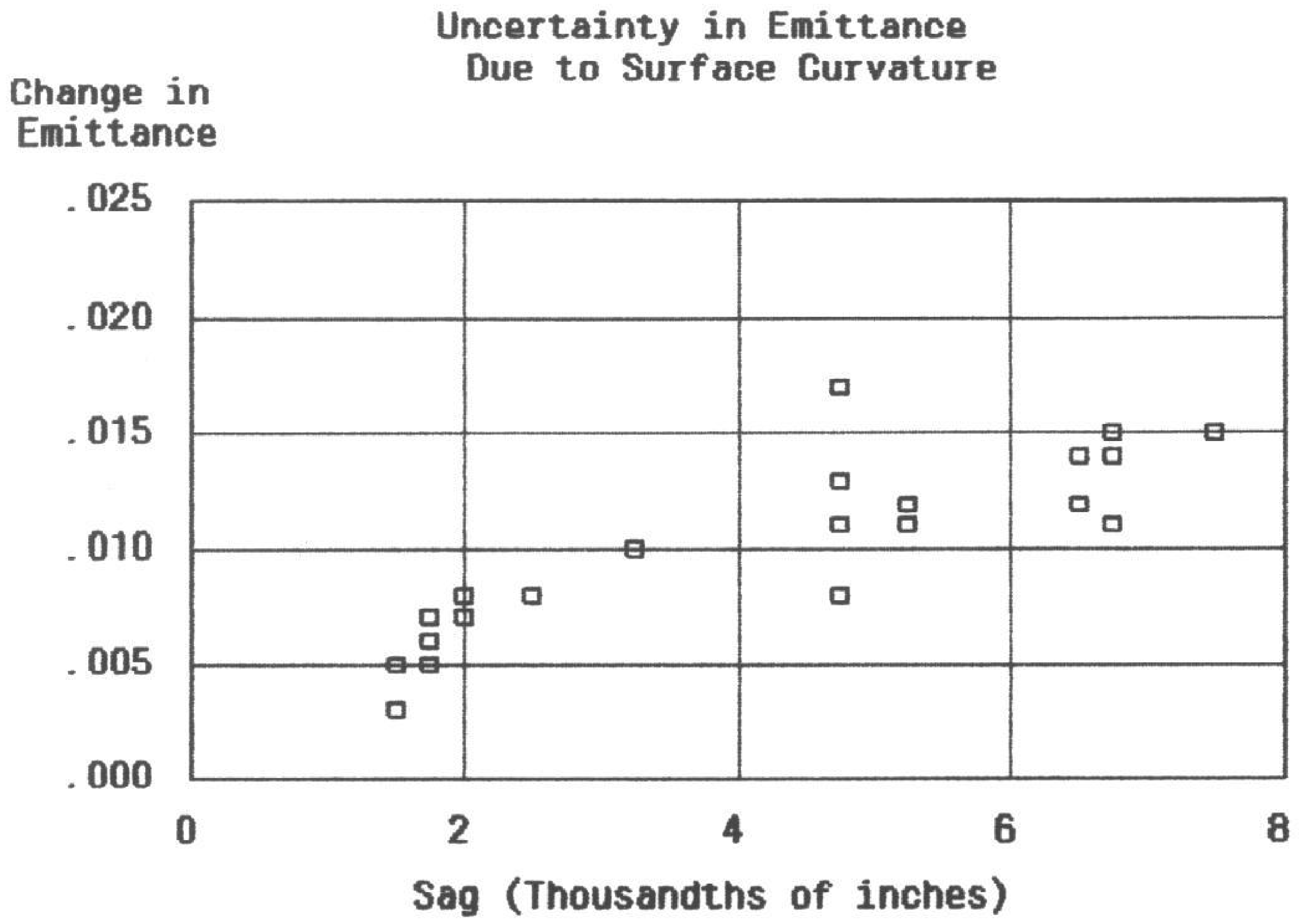


Figure 8. Emittance Error versus Sample Curvature

Conclusions and Recommendations

Based on the measurements outlined above, guidelines have been developed for the measurement of low emittance materials. If desired the resolution of the Model RD1 voltmeter can be changed to produce an emittance reading to 0.001 units. This is accomplished by increasing the gain by a factor of ten. It is also possible to adjust the millivolt scale to display 0 to 2.000 millivolts rather than 0 to 20.00 millivolts as the unit is supplied. Refer to the following section for instructions on making the changes to the voltmeter. It should be noted that even though the voltmeter will display the emittance to 0.001 the actual repeatability of the measurement will depend on many factors.

The guidelines for the measurement of low emittance materials are divided into Calibration and Measurement sections. It is assumed that if the material cannot be placed on the heat sink that it will be measured at a temperature as near as possible to the ambient temperature, and the calibration samples will be maintained at near ambient.

1. Calibration: The calibration procedure remains largely unchanged except that to minimize the overshoot error as described, a standard sample with a lower emittance value is recommended. Devices and Services can supply a stainless steel material with an emittance between 0.15 and 0.20. If another material is used make sure that the sample is as flat as possible. A Nickel plated stainless steel standard with an emittance of 0.05 is now being supplied with Emissometers and is recommended for use as the low emittance standard. These standards can be purchased in pairs from D&S. When the

detector is resting on the sample it must be free to rest flat against the surface. To make a complete gain and offset calibration adjustment, proceed as follows.

Gain and Offset Calibration:

1. Put the high emittance working standard on one end of the heat sink and the low emittance working standard on the other end. To insure that the two standards remain at the same temperature, good heat transfer must be maintained between the standards and the heat sink. This is accomplished by putting several drops of water on the heat sink before putting the standards down. Replenish the water as necessary. Make sure that there is enough water to fill the gap between the standards and the heat sink, but not so much that water flows up onto the samples. Leave space around the heat sink to allow free air flow so that the heat sink temperature will be as close to ambient as possible.

2. The RD1 should be switched to the "variable" position. Place the Emissometer on the high emittance working standard and wait 90 seconds for the reading on the RD1 to become steady. Adjust the variable gain knob on the RD1 so that the reading matches the emittance of the standard.
3. Move the Emissometer to the low emittance working standard and wait for the reading to become steady (90 seconds). Adjust the offset trimmer on the <u>Emissometer</u> until the reading is equal to the emittance of the standard.
4. Place the Emissometer back on the high emittance standard and repeat steps 1 and 2. Repeat the gain set/offset adjust procedure until you can move the Emissometer from the high to the low emittance standard and get the correct emittance readings without adjusting either the gain or the offset. Using a high emittance standard in the range of 0.10 to 0.20 will require a few more iterations than with the usual 0.90 high emittance standard.

This complete calibration procedure should be repeated as often as possible for best results. While the instrument is in use it should be left resting on the heat sink on the high emittance standard. If the reading on the high emittance standard drifts the calibration should be repeated. If it is not possible to calibrate frequently, for example in a production environment, follow the gain adjustment procedure as described below, between the periodic complete calibrations. The test data shows that the primary sources of drift in the detector output cause changes in gain rather than offset.

Gain Adjustment:

1. Return the AE detector to the high emittance standard between measurements. Allow about two minutes between measurements so that a steady reading of the emittance of the standard is obtained.
2. Check the standard emittance reading just prior to the next measurement. If the reading has drifted from the proper value use the gain adjust potentiometer on the RD1 so that the reading matches the emittance of the standard.

If the gain adjust procedure is used, it should be verified that no unexpected drifts in offset are occurring. Test the procedure by checking the low emittance standard periodically during an extended time period in the production environment without adjusting the detector offset.

2. Measurement of Emittance: The procedure for the emittance measurement remains unchanged. To get the most repeatable results the sample must be flat and be at the same temperature as the standards.

Sample Temperature: If the sample can be placed on the heat sink the temperature problem is avoided and no special consideration is required. If not the sample should be as close as possible to room temperature. The emittance reading can be corrected for the sample temperature as follows.

$$\text{Indicated Emittance} = (1 - 0.0058\ \Delta T) * (\text{Actual Emittance}) \qquad (3)$$

Where ΔT - Difference between the sample temperature and the ambient temperature or the temperature of the calibration standards. If the sample is cooler than ambient then ΔT is negative.

This equation is derived from the data in Figure 4. For temperature differences greater than 10 to 20 F, a correction curve should be developed for the particular application.

Some heating of the sample will occur during the measurement. The resulting error is usually negligible for metallic materials and can be ignored. The extent of the measurement error can be estimated by monitoring the detector output while it rests on the sample for two minutes as described in Technical Note 79-17. The transient response should be traced back to the moment that the detector was placed on the sample.

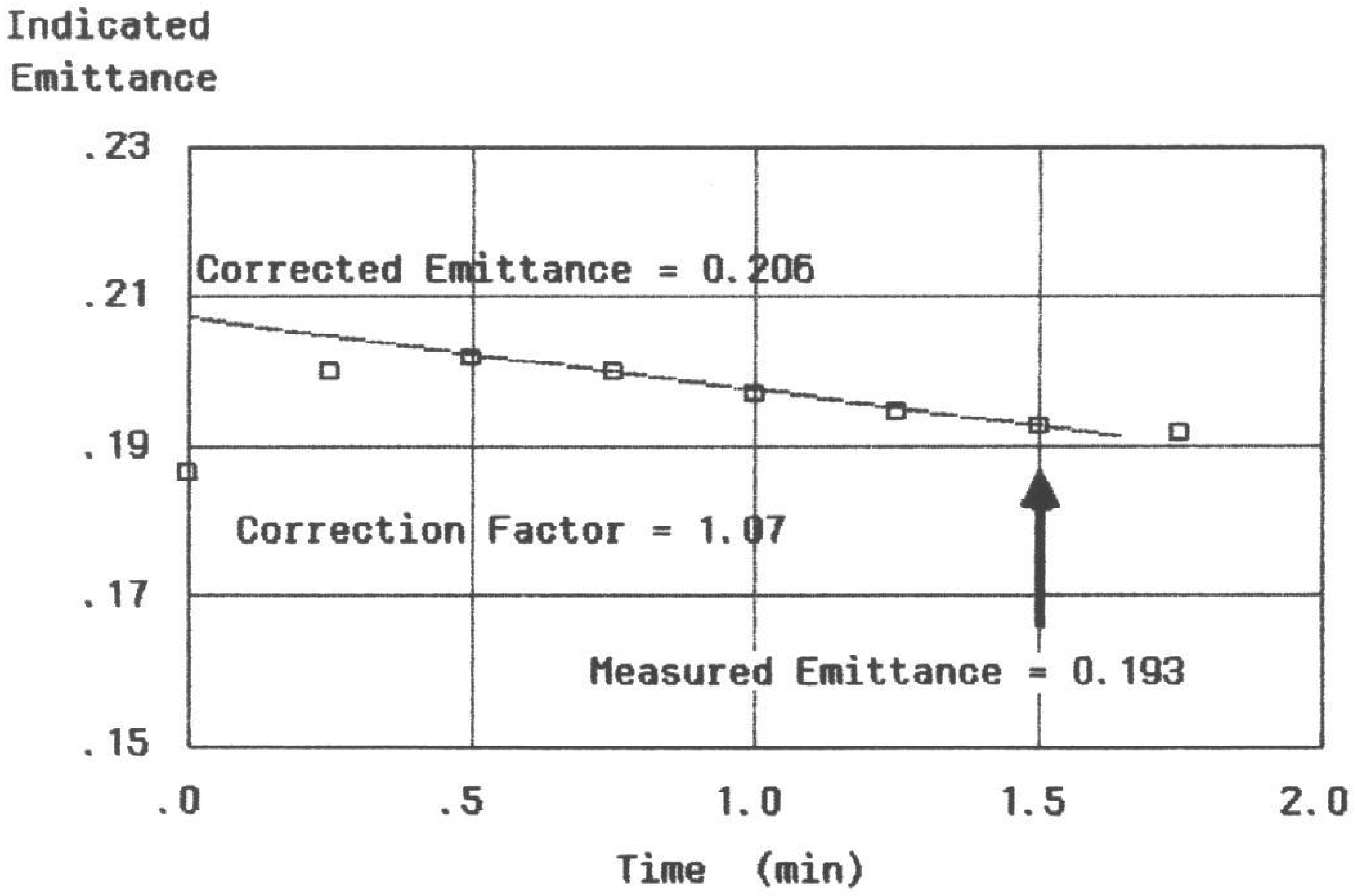


Figure 9. Transient Response on a Nonconductive Material

Once the error is determined for the particular set of conditions, a correction can be applied to succeeding measurements on the same material assuming that the emittance is nearly the same. For production measurements select a time of one to one and a half minutes at which a reading should be taken. Determine a multiplicative correction from data such as that shown in Figure 9 taken under conditions that will be duplicated in the production environment.

Sample Flatness: For a film or thin metallic material that can be placed on the heat sink, the material will either conform to the flat surface of the heat sink or the Emissometer can be pushed down and held to flatten the material against the heat sink. If the material cannot be placed on the heat sink and is not flat to within about 1 thousandth of an inch per inch of width the reading must be corrected for the sample curvature. If the material is not flat and does not have a predictable curvature, it is not feasible to correct the reading. In general the readings will be slightly higher. To determine a correction where the material has a predictable curvature, a sample of the material must be tested in the non flat condition and then flattened against the heat sink and measured. No universal correction can be applied.

If the material cannot be flattened, apply a piece of ordinary aluminum foil to the surface and similarly to the heat sink and determine how the emittance of the foil is affected by the curvature. Based on the data in Figure 6, the error is relatively uniform over the entire range of emittance values and should be almost identical for the foil and the low emittance material to be measured. The correction in this case will be a constant subtracted from the indicated emittance, rather than a scale factor. Select a convenient orientation or azimuth angle for the Emissometer and mark the Emissometer case so that for the production measurements the orientation relative to the curvature of the material can be reproduced. If the surface curvature is predictable, an adapter can be made as described in Technical Note 81-2, or an adapter based on the Model AE-AD1 Adapter described in Technical Note 84-2 can be obtained from Devices and Services Company. The adapter port can be made to match the surface curvature, assuring that the detector orientation is maintained and preventing any possible errors due to air movement between the Emissometer sample port and the sample.

Alteration of the RD1 Scaling Digital Voltmeter

All that is required to change the variable scaling range of the RD1 to display emittance (or reflectance with the Alphatometer) to 0.001 is to patch in a 470 ohm resistor to increase the gain by a factor of ten. The location of the resistor is shown in Figure 10 below. Make sure that the voltmeter is powered off when soldering in the resistor.

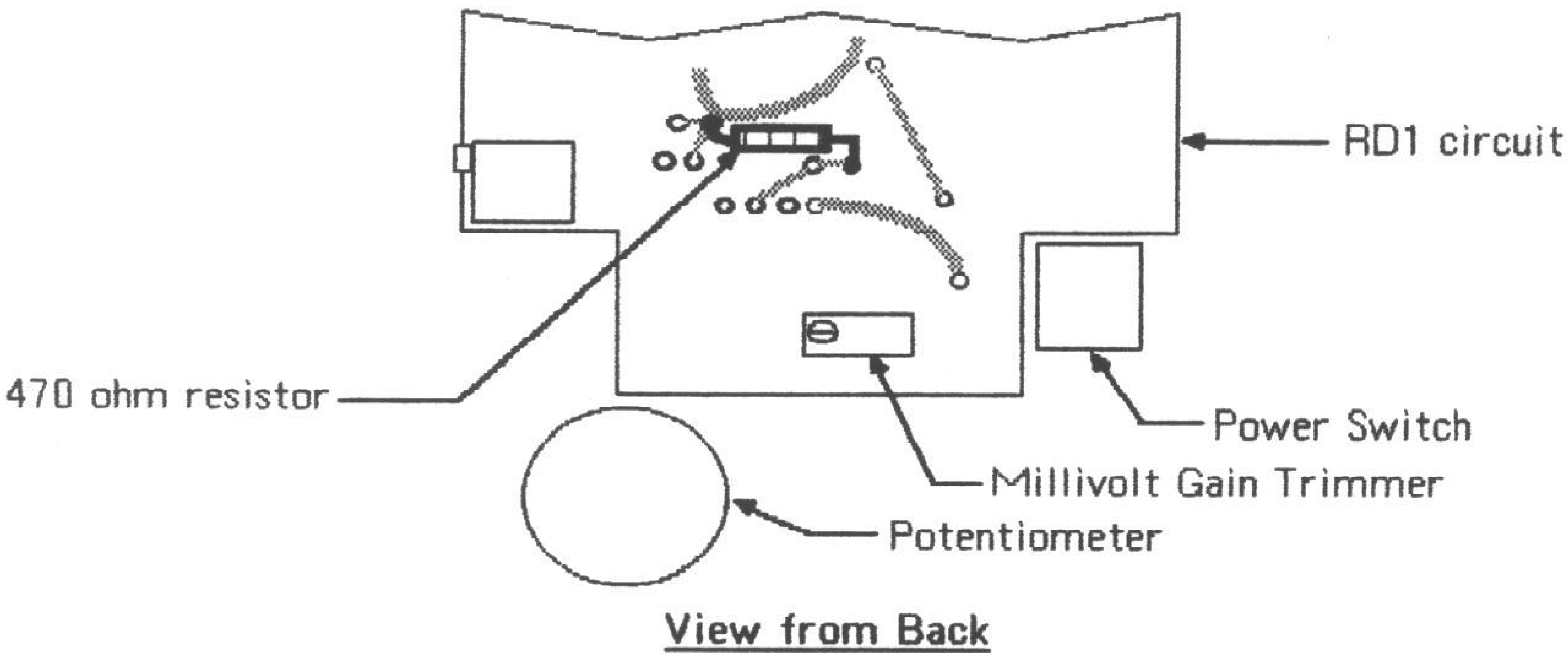


Figure 10. Patch to the RD1 Voltmeter for 10X Gain

It is not possible to move the decimal point without removing the entire board therefore this is not recommended. Also at the high gain level with the resistor installed, the zero offset becomes more critical. The offset will drift more noticeably and should be checked frequently. The major source of drift is the change in battery voltage as the battery ages. It is recommended that if the gain change is made, the optional battery charger/adapter and NICAD battery be installed. With the adapter plugged in the voltage is regulated by the battery, and the NICAD battery has a more constant discharge voltage than primary batteries.

The patched in resistor also affects the gain on the fixed gain or millivolt scale. If it is required that the millivolt scale read out properly it is possible to adjust the millivolt gain trimmer shown in the figure to produce a 0 to 2.000 millivolt full scale range. A millivolt source and a precision voltmeter are required to make the adjustment.

A shaft locking nut can also be supplied for the RD1 that will prevent inadvertently changing the gain setting. This nut is installed by removing the gain adjust knob and the hex nut on the front panel and replacing the hex nut with the locking nut. When installing the locking nut the potentiometer must be supported on the inside of the case to prevent stressing the connections to the printed circuit board. The shaft lock is not recommended for use with the Model 1A Alphatometer.

The following parts are available from Devices & Services Co.

RD1 AC Option: Includes AC Adapter and 9V NICAD Battery

RD1 Shaft Lock

470 Ohm resistor for 10X gain

AE Replacement Heat Sink, milled flat

AE-AD1 Adapter for the AE Emissometer to reduce the sample size to about 1.5 inches.