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INSTRUCTIONS FOR OPERATING THE DIVIDED BAR APPARATUS FOR THERMAL CONDUCTIVITY MEASUREMENT AT THE GSC, CALGARY

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Executive Summary

The divided bar apparatus provides a simple and reliable method for measuring the thermal conductivity of geological samples. This report provides an overview of the theory behind the divided bar method, instructions for use of the divided bar, and treatment of data obtained from the apparatus. The data treatment methods cited in this report are based on software designed by Alan Jessop, and used at the Geological Survey of Canada. The Introduction provides some background on the Divided Bar project. This is followed by a section outlining the basic theory behind the divided bar technique, and a section describing the structure and mechanics of the apparatus. The next two sections provide an overview of general techniques for collecting data from the apparatus and performing calculations with that data. The following two sections indicate some of the assumptions and generalizations utilized in handling data obtained from the apparatus, after which follows a discussion of the nuances specific to measuring geological samples with the divided bar technique. The next section is a description of the methods used to calibrate the divided bar apparatus and the thermistors used in the apparatus. The final sections indicate the accuracy of the GSCC divided bar apparatus measurements as compared to previously conducted measurements, and list recommendations for greater accuracy in future projects using the GSCC divided bar. Appendix I provides a point form description of the method of operation of the divided bar apparatus and Appendix II describes the computer processing of resistance data acquired during sample measurement.

Acknowledgements

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Introduction

The divided bar is a steady-state measuring device which is capable of determining the thermal conductivity of a prepared sample within an accuracy of 2% (Jessop, 1990). The divided bar has become a preferred method of measuring the thermal conductivity of geological samples due to its relative simplicity of operation, the ease of sample preparation and the availability of samples. The Geological Survey of Canada at Calgary (GSCC) recently commissioned the manufacture of two divided bar apparatuses for the purpose of measuring the thermal conductivity of geological samples. The assembly of the apparatuses and all necessary hardware was completed in the Spring of 1996. However, to develop an operational system, it was necessary to calibrate and test the machines for accuracy, test the data acquisition software and develop procedures for sample preparation and data presentation. This latter work was completed between the months of May and September, 1996.

The primary objective of the project was to initiate the operation of the divided bars at the GSCC, and to determine the viability of their use. This study was conducted through the everyday operations involved in preparing the divided bar for use, and the actual use of the divided bar for measurement and calibration purposes. The bar was operated solely by the author, who was guided by the advice of Drs. Dale Issler and Alan Jessop. This report outlines the physical principles utilized in divided bar measurements, as well as providing an overview of the practical operation of the apparatus and the specific concerns which must be addressed in the testing of geological samples.

This project proved successful in preparing the divided bar apparatuses for use. All the needed extra equipment and resources have been obtained, the bar has been tested thoroughly, and recommendations on procedures for use of the divided bar have been documented

Basic Theory of the Divided Bar Method

The divided bar apparatus measures thermal conductivity by means of comparative temperature readings. A cylindrical bar of glass is split in the middle, and a sample of unknown conductivity is inserted. Each end of the bar is brought to a constant temperature, and thermal conductivity is determined by comparing the temperature difference across the sample to the temperature differences across each half of the glass bar. The temperature differences are calculated from temperature readings taken on both sides of each half of the glass bar. The temperature readings are calculated using four thermistors, which, for ease of measurement, are inserted into a brass disk. These temperature readings are converted into temperature differences across the sample and each of the glass disks. The temperature difference across the sample is then compared to the temperature differences across the glass disks, which have a known thermal conductivity. The result of these calculations is the thermal conductivity of the sample.

Structure and Mechanics of the Divided Bar

The glass bar itself is composed of two disks of identical composition. Glass is chosen as the bar material so that the thermal resistance of each half of the bar will be of the same magnitude as the thermal resistance of the sample being measured. Since these glass disks cannot be adapted to allow for the inclusion of temperature reading devices, brass disks are inserted on either side of each glass disk (see Figure 1). These disks are chosen to be brass because holes can be drilled easily into brass to form cavities for the insertion of thermistors. Brass also has a low thermal resistance, which prevents these extra disks from influencing measurements. The sample is inserted at the centre of the bar, between the two interior brass disks.

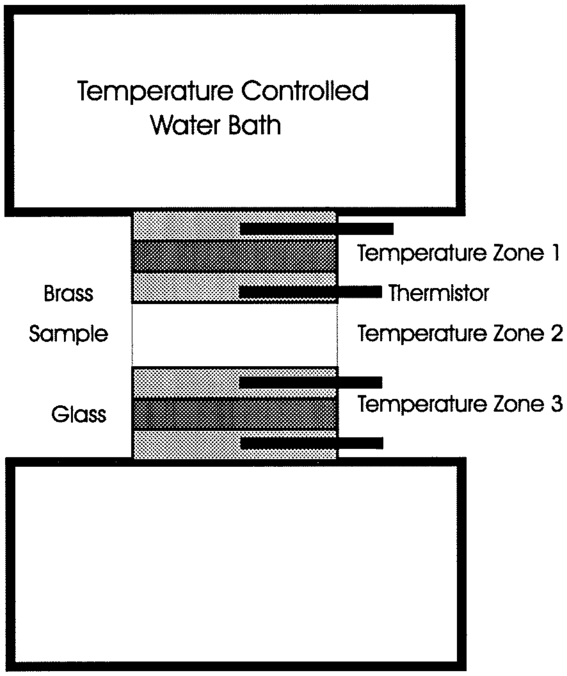

Figure 1: Schematic Diagram of the Divided Bar Apparatus

The ends of each half bar are kept at constant temperature by means of direct thermal contact with a circulating water bath. The baths are held with a constant 10°C temperature difference. The top bath is held at 5°C above room temperature; the bottom bath at 5°C below room temperature.

The top water bath is secured at a fixed height, whereas the bottom water bath is mounted on a hydraulic piston. The bottom bath is lowered for the insertion and removal of samples, then raised and held at 250 kPa pressure for the actual measurement. This pressure ensures good thermal contact between the bar and the sample without threatening the integrities of the glass reference disks. Thermal contact is encouraged further by lubricating all contacting surfaces of the bar and the sample with a conductivity agent which in this case is glycerin. The method for testing samples using the divided bar apparatus is outlined in Appendix 1.

Collecting Data from the Divided Bar

An experimental run of the divided bar apparatus will generate, through appropriate hardware/software interfaces, a series of periodic resistance readings from each thermistor. Good results are obtained by taking one resistance measurement per thermistor per second.

Since the divided bar operates on steady state measurement principles, all temperatures must be stable when a measurement is made. Stable temperatures are achieved only if the bar is given sufficient time to equilibrate, typically between three and seven minutes. Temperature stability is determined by examining the temperatures calculated from each of the individual thermistor readings. For each thermistor, temperature is sufficiently close to equilibrium when fifteen successive calculated temperatures indicate a change of no more than 0.005°C, and have a standard deviation of no more than 0.005°C. The average of these fifteen temperatures is used in calculations as the equilibrium temperature.

Calculation of Thermal Conductivity

The result from each trial on the divided bar is four equilibrium temperature readings. These four temperatures must then be converted into three temperature differences: the temperature difference across the sample, and the temperature difference across each of the glass disks. The temperature difference, V_1, across the first glass disk is determined by subtracting the temperature reading taken on one side of the disk from the temperature reading taken on the other side of the disk. The temperature difference across the sample, V_2, and the temperature difference across the second glass disk, V_3, are calculated in the same manner.

In order to calculate the thermal conductivity of the sample, these temperature differences (V_1, V_2 and V_3) are inserted into the formula for thermal conductivity. The thermal conductivity relationship is based on the principle that temperature drop is proportional to thermal resistance. The basic equation (Jessop, 1990) is

$$d_g \ (V_2 / (V_1 + V_3)) = d_s \, (K_g / K_s) + R \, K_g \qquad (1)$$

Here, K_s is the thermal conductivity of the sample, d_s is the average thickness of the sample, d_g is the total thickness of the two glass disks, K_g is the thermal conductivity of the glass and R is an estimate of the total unwanted thermal resistance associated with the components of the divided bar. Solving for the thermal conductivity of the sample gives

$$K_s = d_s \, K_g / (d_g \, V_2 / (V_1 + V_3) - R \, K_g) \qquad (2)$$

Assumptions

When conducting thermal conductivity measurements, one must assume that heat flows only in the axial direction (Jessop, 1970). The possibility of lateral heat flow is not accounted for in the calculations, and thus every effort must be made to minimize non-axial heat flow. This heat flow is minimized by the basic structure of the divided bar: since the diameter of the water baths greatly exceeds the diameter of the bar, the temperature field around the bar conforms to axial heat flow through the bar. Heating the upper water bath to a greater temperature than the lower water bath prevents heat convection around the bar. To reduce perpendicular heat flow, the water baths are maintained at temperatures that will approximate room temperature when averaged. The bar is insulated with a Parafilm wrap during the period of measurement to further encourage only axial heat flow. Also, the entire apparatus is encased in a cabinet in order to maintain a constant environmental temperature, and to minimize any air currents.

There is also an assumption made in the estimation of the variable R. Generally, only one disk per sample is measured using the divided bar, but the divided bar method has been used for multi-disk measurements of conductivity as well. The multi-disk method involves taking temperature measurements on a set of disks of common composition and varying thickness. The results are plotted according to formula (1), and a value for the ratio K_g / K_s is determined from the linear slope of $\{dg\ V_2 / (V_1 + V_3)\}$ versus ds. The intercept gives a value for R, where

$$R \sim R_2 - (R_1 + R_3)\, d_s\, K_g / d_g\, K_s \qquad (3)$$

Here, R_1, R_2 and R_3 are the unwanted thermal resistances in each section. These resistances are due to the brass disks and to the various contacts involving the baths, glass standards, sample and brass disks.

The multi-disk technique is not employed at the GSCC for many reasons. The number of trials necessary is far greater than in single-disk measurement, and multi-disk calculations and plotting routines would demand sophisticated data treatment programs. As well, the composition of the geological samples in a multi-disk set is not identical. The inhomogeneities inherent in geological samples introduce scatter into the plotted results. The uncertainty due to compositional change could be reduced by repeatedly grinding and polishing one sample to a lesser and lesser thickness and measuring the conductivity for successively smaller sample sizes, although the amount of time demanded by such a process would be great.

The variable R is estimated as a constant for the purpose of the single disk measuring technique. This estimation is based on the values of R determined from previously conducted multi-disk measurements. Generally, R is assigned a value of about 1.0×10^{-4} Km^2W^{-1} . A geological sample of median conductivity and thickness (a 10 mm disk of sandstone) will give the bar an overall thermal resistance of about 9.0×10^{-3} Km^2W^{-1}. Since the resistance R is only about 1% of the total thermal resistance across the bar, approximating R does not introduce a great error into calculation.

Dealing With Disparities between Sample Diameter and Bar Diameter

Ideally, the diameter of the bar should be matched exactly by the diameter of the sample. Unfortunately, in practical use, this rarely occurs. Considerations for the disparity in size between the bar and a sample can be factored into the calculations for the thermal conductivity of that sample.

The diameter difference between the sample and the bar will dictate the adjustment made to the calculation. If the sample is smaller in diameter than the bar, all heat flow is assumed to be directed through the sample. However, if the sample is larger in diameter than the bar, an approximation of heat flow must be made using a Bessel function, which solves the heat flow problem in cylindrical coordinates.

Geological Samples and the Divided Bar

Particular care must be taken in using the divided bar apparatus to measure the conductivity of a geological sample. Generally, the core samples analyzed in this study had received only preliminary preparation. The disks were cut from core plugs without careful measurement or additional finishing, and therefore were not immediately suitable for bar measurement.

In order to achieve good thermal contact, samples were polished so that their surfaces were flat to within 0.3 mm (no wedge shape), and parallel to within 0.03 mm throughout (no dome or concavity on the sample surface). These specifications ensure maximum surficial contact between the sample and the brass disks.

Every effort must be made to reproduce the *in situ* conditions of the sample. Low values for thermal conductivity are obtained if a sample is measured while dry. This is attributed to the fact that, in the subsurface, the pore spaces of the sample are saturated with groundwater, which has a thermal conductivity of about 0.6071 W/mK at 25°C (distilled water). Air at 25°C has a thermal conductivity of only 0.0026 W/mK, a difference which is especially significant for highly porous samples.

In order to recreate *in situ* groundwater conditions, samples were saturated with an appropriate pore fluid. This was achieved by placing the sample disk in a beaker of fluid. The beaker was then placed in a desiccator and air was evacuated for twelve hours, in a vacuum of at least 22 ft of water to remove any air present within the pore space of the disk. Then the beaker was exposed to atmospheric pressure for twelve hours, allowing the pore space to be permeated by the fluid present in the beaker.

The type of saturating fluid chosen depends on the mineralogical composition of the sample. Carbonates, quartzites and sandstones can generally be saturated in pure water. Due to reactions with water (e.g. swelling, flaking or deterioration), shales, which contain a high proportion of clay minerals, must be saturated in a different fluid. The GSCC is currently using brine mixtures, with concentrations and compositions determined by studying the groundwater of the samples' area and formation of origin. A 2% solution of KCl brine has been used successfully to saturate problematic rock types. Where this method is in doubt, mineral oil is used as a saturating fluid.

The porosity and pore fluid composition of a sample can greatly affect the thermal conductivity. Saturation of the sample allows the porosity and density of the sample to be accurately obtained. In order to calculate the porosity of the sample, both saturated and dry weights must be measured. Porosity (ϕ) is given by the formula

$$\phi = (\rho_s - \rho_d) / \rho_f \tag{4}$$

where ρ_s is the saturated density, ρ_d is the dry density and ρ_f is the fluid density, approximated to 1 g/cm^3 for water and brine solutions. The densities of the samples are determined by the formula

$$\rho = w / V \qquad (5)$$

where w is the mass and V is the volume of the sample. The saturated mass is the greatest mass recorded for the sample immediately upon removal from the saturation bath. The mass of the sample will decrease rapidly with time, as shown in figure 2, and an accurate approximation of the dry mass can be measured after the sample has been in a drying oven for about one week at about 80°C.

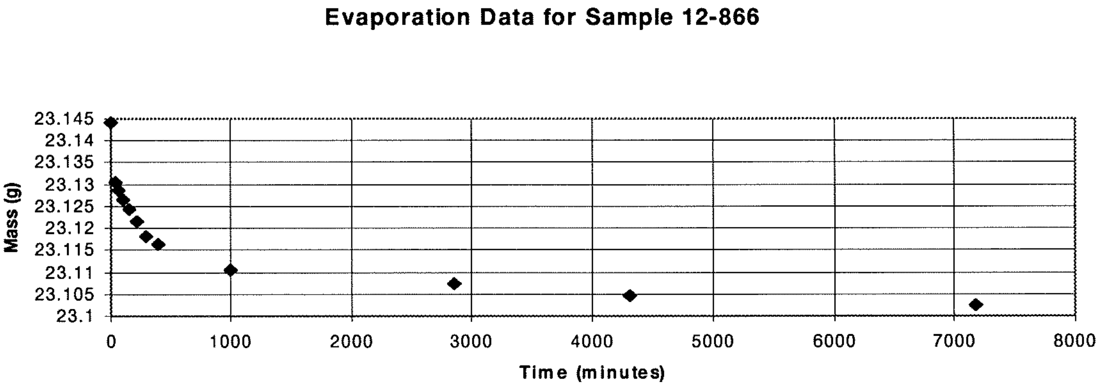

Figure 2: Mass loss of a Saturated Sample Disk as a Function of Time

The volume used in these calculations is determined by substituting the measured thickness and diameter of the sample into the equation for the volume of a cylinder. Sample disks are never perfect cylinders, so the dimensions for a sample are best obtained by averaging measurements made on several different points on the disk. This averaging, as well as the instrument uncertainty of the calipers, introduces uncertainty into the calculated volume. This volume uncertainty is propagated into the density and porosity values. Samples which have been glued with epoxy are subject to inaccurate density and porosity measurements, due in part to gaps in the sample not filled with epoxy, and due in part to the epoxy itself.

Calibrations

I. Calibration of the Divided Bar

Calibration tests must be run in order to determine the thermal conductivity of the bar. The thermal conductivity of glass varies depending upon its composition, therefore the calibration routine must be repeated with every change of half-bar. The edges of the glass half-bars have been bevelled in an attempt to minimize the damage caused by any uneven pressure exerted on the glass, but the half-bars will still become chipped during regular use. Recalibration may be necessary to account for damaged glass disks.

The calibration routine is a reversal of the routine for measuring conductivity. A sample of known conductivity is inserted into the divided bar, and the corresponding bar resistance is determined using the following manipulation of formula (1):

$$K_g = d_g V_2 / \{(V_1 + V_3)((d_s / K_s) + R)\} \qquad (6)$$

where the variables are as outlined previously.

The calibration tests are valid only when conducted at the temperature at which the bar is generally held. The sample used in calibration is chosen generally to be fused quartz (vitreous silica), due to the precise data available regarding its thermal conductivity. Calibration tests are performed on samples of varying thickness in order to better determine the conductivity of the bar. Calibration results are considered valid if the calibrated glass disks can be used to measure the thermal conductivity of fused quartz to within two percent of the value reported in the CRC Handbook.

II. Calibration of the Thermistors

Thermistors are extremely well suited for use with the divided bar, owing to their small size and their high sensitivity. The thermistors used for divided bar purposes have a nominal resistance of about 10 kΩ at 25°C. Due to possible resistance drift over time, it is required that thermistors be recalibrated periodically. A comparative method of calibration is done using the divided bar apparatus.

A brass disk of about 5 mm thickness containing evenly spaced drilled holes about its circumference is inserted into the bar. The thermistors which require calibration are then placed in the holes of the brass disk. Both water baths are maintained at the same temperature, within the accuracy of the baths' temperature control. The entire system is then allowed to reach an equilibrium temperature, where resistance readings are taken by each thermistor simultaneously. Three trials are conducted at temperature intervals varying from 10 to 30°C. For each trial, individual thermistor resistances are averaged.

In order to determine the temperature at which the trial was conducted, these average resistances are converted to a temperature, T, by using the formula

$$T = \{B / (\ln(A) - \ln(R))\} + C \qquad (7)$$

where R is the measured resistance and A, B, and C are constants determined in the initial thermistor calibration. Since the system is measured at a stable temperature, this average temperature is the constant temperature detected by the thermistor. Completely random thermistor drift is assumed, and the temperature of the entire system is found by averaging the temperature reading from each of the thermistors present.

Calibration drift is accounted for by appending a linear correction onto a manipulation of formula (7). This correction is determined by plotting the mean deviation of one thermistor's temperature reading against the system temperature, as in figure 3. The formula of a least-squares fit linear trendline to this plot is added to formula (7) in order to account for thermistor calibration drift.

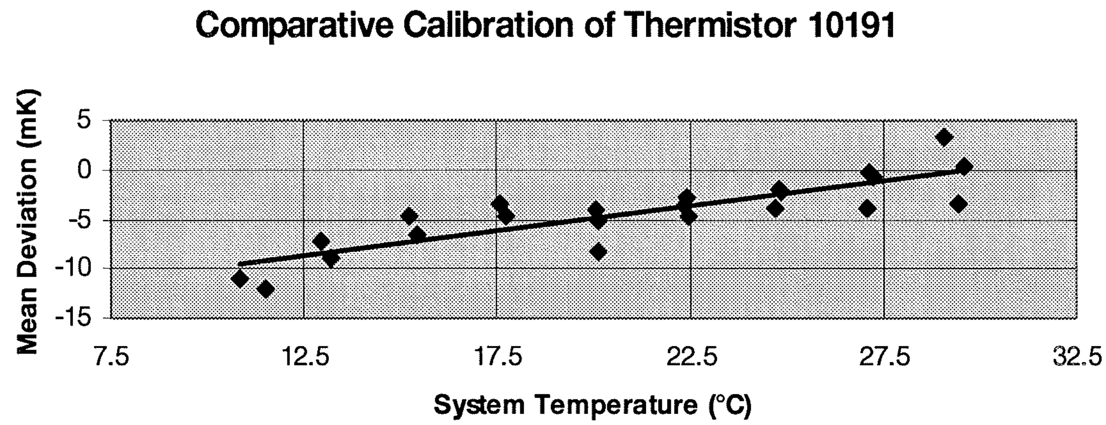

Figure 3: Thermistor 10191: Mean Deviation from Average Temperature as a Function of System Temperature

Comparison With Previous Thermal Conductivity Measurements

The findings of this study confirm that the divided bar apparatus provides a viable method of measuring the thermal conductivities of geological samples. In an ongoing project, various geological and geothermal data have been measured for sites across Canada and compiled into the Bordat data collection. The Bordat files include thermal conductivity measurements conducted using a divided bar apparatus which had previously been in operation at the Geological Survey of Canada, Ottawa. Thermal conductivity measurements of Bordat samples were repeated using the GSCC divided bar apparatus, and GSCC results averaged only three percent different from the established values. These results are depicted in figure 4.

The divided bar's practicality for measuring geological samples is confirmed not only by this accuracy, but by the suitability of geological samples for measurement in the divided bar. Although samples must generally be lapped prior to testing, the amount of time dedicated to preparing samples is small compared to the time demanded by measurements. The saturation of samples prior to testing approximates *in situ* conditions, minimizing the uncertainties introduced by measuring terrestrial properties in a laboratory setting. Great quantities of depth-specific samples can be processed through the apparatus, making divided bar data extremely useful in determining terrestrial heat flow.

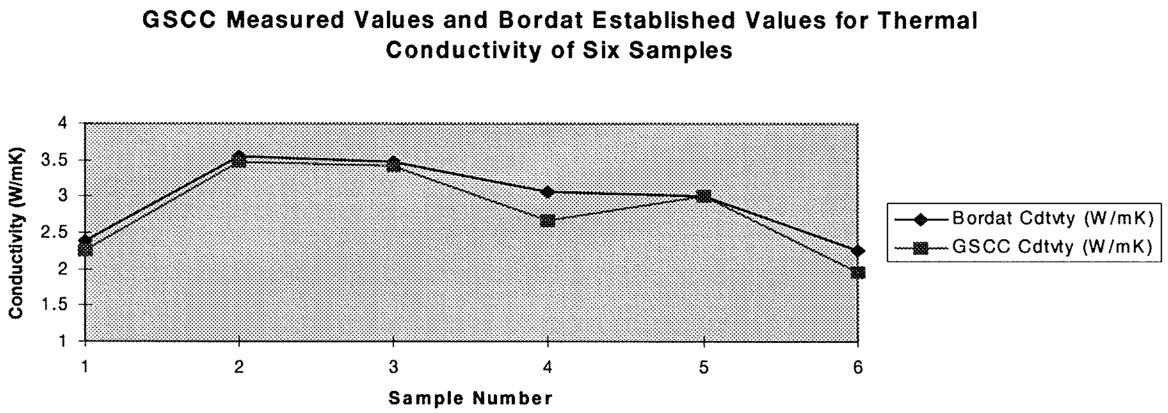

Figure 4: Comparison of GSCC Summer 1996 Measured Values for Thermal Conductivity to Previously Measured Values

Recommendations

Future technicians working with the divided bar apparatus should consider the following suggestions:

- The saturation of all samples in brine solution. Employing a saturation fluid similar in composition to the sample's indigenous pore fluid would better approximate *in situ* conditions than saturation in pure water. Samples which may deteriorate due to the brine solution's ionic content should be saturated in a light oil of know thermal conductivity and specific gravity.

- The measurement of known standards of thermal conductivity using the apparatus. Currently, the calibration disks of fused quartz are the only thermal standards available. Estimates of the measurement accuracy have been made by comparing the values obtained for geological samples to the previously documented values for the same samples. These previously measured values, however, are substantiated by only one measurement per sample, conducted using another divided bar apparatus. A genuine measurement of accuracy should be made using a rock standard of documented composition and conductivity.

- Employment of the multi-disk measurement technique to verify resistance estimates. The resistance constants estimated by GSCC experimenters are based on multi-disk measurements conducted using a different apparatus. The validity of these estimates could be tested by conducting multi-disk measurements on the GSCC apparatuses.

References

Jessop, A. M., 1970. The Effect of Environment on Divided Bar Measurements. In: Tectonophysics, 10. Elsevier Publishing Company, Amsterdam, pp. 39-49.

Jessop, A. M., 1990. Thermal Geophysics. Elsevier Publishing Company, Amsterdam. 2.7: 36-38

Appendix 1: Experimental Method of the Divided Bar Apparatus

- A suitably prepared sample is selected for testing.
- The lower water bath is lowered.
- Contacting surfaces of the baths, glass disks and brass disks are covered with a thin film of glycerin.
- Surfaces and edges of the sample are covered with a thin film of glycerin.
- With the bottom water bath lowered, the disks and sample are carefully stacked, care being taken to ensure that the centres of the disks are aligned with one another.
- The bottom water bath is raised until the upper disk of the bar makes contact with the top water bath.
- The bar is inspected to ensure that full surficial contact has been made between all disks.
- Thermistors are inserted into the holes in the brass disks.
- Parafilm is wrapped around the bar.
- Thermistor readings are taken during a ten minute test period.
- The bottom water bath is lowered and all disks are removed from the apparatus.
- Thermal contacts are completely broken.
- The sample is reinserted in the same manner.
- Five trials are conducted on each sample.

Appendix 2: Treatment of Divided Bar Output Data Using GSCC Software

The GSCC uses DB, a FORTRAN program designed by Dr. Alan Jessop to convert the divided bar output data from resistance readings to thermal conductivity. With sufficient data inputs, the DB program will also calculate the porosity and density of a sample, using the methods cited earlier in this report. Thermistor resistance readings from the divided bar apparatus are compiled into data files by the LabTech Notebook software. You can find this package on the hard drive of the divided bar computer (C:\NB). In order to gather data for a ten minute trial, you must set both the Number of Iterations and the Stage Duration (both found by accessing first Setup, then Blocks, then Normal) to a value of 600. You must also provide a new name for the output data file (i.e. the file containing the 600 thermistor resistance readings made throughout the trial), which is done by accessing first Setup, then Files, and entering a Data File Name.

A LabTech Notebook file of resistance readings should look similar to the following, which is an excerpt from a file generated from the test of a sample from Kapuskasing, Well 12-0:

```
"Dry Conductivity Measurement"
"Thermistors:  A, 3, 4, 5"
"Positions:  1, 2, 3, 4"
"The date is  1-08-1997"
"one" "two" "three" "four" "five" "six" "seven" "eight"
"ohms" "ohms" "ohms" "ohms" "ohms" "ohms" "ohms" "ohms"
  7493.1  9126.3 10329.7  11690.8   8863.5   9387.9   9614.9 9900.0
  7492.4  9124.3 10328.4  11691.0   8862.7   9387.6   9614.6 9900.0
  7492.1  9122.5 10327.0  11691.3   8862.3   9387.2   9614.5 9900.0
  7491.5  9118.8 10324.3  11691.2   8861.5   9386.5   9615.4 9900.0
  7491.2  9116.8 10323.0  11691.0   8861.1   9386.8   9615.8 9900.0
```

The file which results from a full trial run will, of course, include 600 lines of resistance readings. You should note that generally the resistances recorded in the first four columns will correspond to the trial you are conducting on the left-side apparatus, and the last four columns will correspond to the trial you are conducting on the right-side apparatus. For cases (such as the one above) where you are only using one apparatus, four of the thermistors will simply indicate the ambient air temperature.

In order to utilize the DB program, you must move the LabTech Notebook output file (the above file was named "12-866a.dat" to reflect the well number, depth of sample, and trial number) to a directory containing the DB program. You will find this directory on the hard drive of the divided bar computer as C:\DIVBAR.

The DIVBAR directory also must contain the data files BARCALIB.DAT and THERMIST.DAT. DB will access the THERMIST.DAT file when it is converting the LabTech measured resistances to actual temperatures: THERMIST.DAT lists the A, B, C coefficients, and the linear correction terms D and E for each of the thermistors. DB will access BARCALIB.DAT to find values for the diameter, thickness and conductivity of the bar, for the estimations of thermal resistances due to the contacts and the metal, and for the temperature stability determination requirements. You must record any change in thermistor calibration, bar calibration, bar dimension, unwanted resistance estimate, or temperature stability requirement in these files.

When you run the DB program, you will be prompted to input information about the experimental setup and the sample characteristics. All of the possible program inputs are listed below:

```
 This version of the program is for use with
thermistors and the ISPG automatic data system
This program is for single-disc samples only
Program for card-image input not yet available
Program for disc sets not yet available
proceed? (y/n) ...

Enter date today (nn aaa nn) ...

Enter number of bar in use ...

Enter output name (printer = prn) ...

Bar Dimensions and Calibrations from file
                           Bar No                  1
                           Bar Diameter            31.740
                           Bar Thickness           6.300
                           Conductivity            1.200
                           Contact Resistance      0.300
                           Metal Resistance  0.100
Criteria for temperature equilibrium
                     Beginning to end change 0.005
               Std.dev. of ten temperatures  0.005
```

```
   New thermistor calibrations (y/n) ...
   Number of thermistor in place 1 ...
   Number of thermistor in place 2 ...
   Number of thermistor in place 3 ...
   Number of thermistor in place 4 ...

   Do you want conductivity analysis (y/n) ...
   Do you want calibration analysis (y/n) ...
   Do you want diameter correction (y/n) ...
   Do you want density (y/n) ...
   Do you want porosity (y/n) ...


   State of discs (wet/dry) ...
   Initials (3) of operator ...

Enter sample data:

   Measurement date (nn aaa nn) ...
   Depth ...
   metres or feet (m/f) ...
   diameter (in mm) ...
   disc thickness (in mm) ...
   dry weight (in g) ...
   wet weight (in g) ...

   comment on the sample (30 char) ...
   Enter name of bar readings file ...
```

The 'Number of the Bar in Use' refers to the line of the BARCALIB.DAT file to which DB will look for the bar information. Entering 1 directs DB to the first line of data, 2 to the second, and so on.

The complete output name that you specify will become the name and designator of the DB output file. Generally, it's helpful for purposes of grouping to give the DB output file a name that is similar to the LabTech Notebook output file (example: LabTech Notebook file name 12-866A.DAT and DB file name 12-866.DAT). Note that giving the files *exactly* the same name will cause the program to terminate.

DB next lists the specified bar constants that it has retrieved from the BARCALIB.DAT file. This allows you to check that the apparatus that you have specified is indeed the apparatus that you are using. If you need to change one or more of the bar constants, the DB program will allow you to do so. You can either make temporary changes (BARCALIB.DAT is not changed) or permanent changes (the new data overwrites the previous data in BARCALIB.DAT). DB will prompt you to indicate whether or not you would like the changes saved.

DB will also list and allow changes to the temperature equilibrium criteria. Good results are achieved by using the default values of 0.005°C beginning to end change and 0.005°C standard deviation, but thin samples (~6 mm or less) may not equilibrate to this precision in a reasonable time period. For these types of samples, you may want to relax these temperature equilibrium constraints somewhat (both values can be increased to 0.010°C without great risk to data integrity.)

The DB program can be used to enter new thermistor calibrations directly into the THERMIST.DAT file. The program will prompt you to enter the new constants corresponding to the thermistor number which you have specified, and will overwrite the existing THERMIST.DAT information with this new information.

The program will ask you next to specify the number of the thermistor in Place 1, 2, 3 and 4. Place 1 refers to the topmost thermistor placement in the apparatus (i.e. the warmest temperature region), and so on, with Place 4 referring to the lowest thermistor placement on the apparatus (i.e. the coolest temperature region). The numbers that you enter will describe both the line of the THERMIST.DAT file to which DB will look for calibration constants and the column of the LabTech Notebook output file to which DB will look for resistance readings. Specifying thermistor 1 in Place 1 means that the thermistor occupying the topmost position on the apparatus corresponds to calibration constants which can be found on Row 1 of the THERMIST.DAT file, and resistance readings which can be found in Column 1 of the LabTech Notebook output file. Note that thermistor x always represents row x of the THERMIST.DAT file and the xth column of numbers of the LabTech Notebook output file.

If you are using both apparatuses simultaneously, you will need to run each LabTech Notebook output file through the DB program twice: once to calculate thermal conductivity from the sample in the left-side bar, and once to calculate thermal conductivity in the right-side bar. Make sure that you indicate the correct bar data for each apparatus. It is also very important to correctly enter the thermistor information. Generally, the right-side bar will have thermistors 1, 2, 3 and 4 in positions 1, 2, 3 and 4, and the left-side bar will have thermistors 5, 6, 7 and 8 in positions 1, 2, 3 and 4. You will probably find it helpful to indicate the thermistor ordering and positioning in the header of the LabTech Notebook output file. This is especially important for files that may not be processed immediately, or files that may need to be reprocessed at a later date. You should also record the dates and values of each recalibration (for both bars and thermistors), so that the calibration information relevant to the date of testing can be applied to any trial runs that may be processed after the original trial.

Next, DB will ask if you would like conductivity analysis, and you should answer Yes. You have only one other option in the DB program, and that is to use DB for bar calibration purposes. The DB calibration routine, however, is in a state of development, and you can generally achieve precise calibration values by measuring the conductivity of fused quartz samples on an uncalibrated bar, and back calculating the glass bar resistance (this is done by manipulating the BARCALIB.DAT Kg value until the DB calculated value for the thermal conductivity of fused quartz samples falls within about one half of one percent of the CRC handbook value. See figure A2.1 for a plot of thermal conductivities of fused quartz at various temperatures).

The program will next ask if you would like diameter correction. Answering Yes calls a subroutine which uses an appropriate Bessel function to approximate heat flow through the sample. Diameter correction does not discernibly slow the operating time of the program, and it increases the accuracy of the measured thermal conductivity. Based on these factors, there is really no reason not to apply a diameter correction to every sample.

If you have taken suitable wet and dry mass measurements on a sample, DB will calculate from these values both wet and dry densities and a porosity. The next questions that DB presents are regarding these measurements. You will later be asked for the measured mass values of the sample.

The next two questions are for informational purposes only. DB will indicate in the printout of results both the state of the sample (i.e. wet or dry) and the operator's initials.

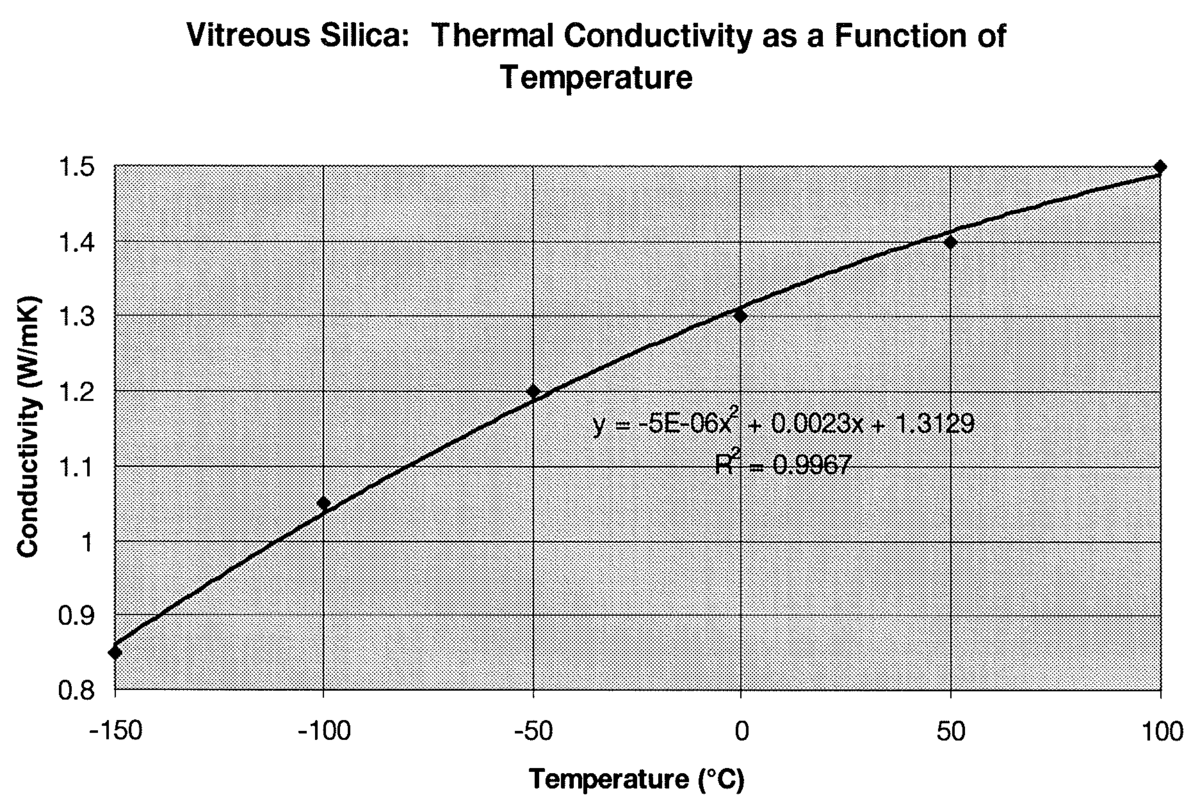

Figure A2.1: Thermal Conductivity as a Function of Temperature for Fused Quartz

The sample data section next requests information regarding the specific sample which is being measured. You should indicate the date of measurement for general informational purposes. DB also asks you to indicate the depth from which the sample was taken. Enter the depth in whatever units you have (i.e. metres or feet.) The next question asks in what units the depth has been measured. If you have supplied a depth in feet, DB will calculate the depth in metres. If you supply a depth in metres, the corresponding depth in feet will not be calculated.

DB next asks for sample data which will be used in calculations: the diameter of the sample will be used in diameter correction and volume calculation, the thickness will be used in thermal conductivity and volume calculation, and the weights will be used along with the volume in determining the densities and porosity.

Next, you are provided with an opportunity to comment on the sample. This is once again for informational purposes only. You will find that comments here are helpful for relating any peculiarities of the sample which may influence the conductivity measurement. These include (but are not limited to): doming, wedging, chips, epoxy fill in cracks, enclosure in plastic casing and saturation fluid.

The name of the bar readings file will be the name of the LabTech Notebook output file which corresponds to all of the information that you have provided above.

Once DB has been directed to the file of resistance readings, it will convert the resistances to temperatures as it searches chronologically down the file for temperature stability meeting your specified requirements. When temperature stability has been achieved (the overall change and the standard deviation of fifteen sequential temperatures are within your specified limits for each of the thermistors), DB will average the fifteen stable temperature readings into one temperature for each of the four thermistors. DB then uses these temperatures to calculate the temperature difference across each of the glass disks and the sample, and from this calculates the thermal conductivity of the sample.

DB will print, both to the screen and to the output file, the following information:

- The line at which stable temperatures were reached: this value will vary depending on the stability of water temperature in the baths (which increases as the baths are allowed to equilibrate longer at any given temperature), the thickness of the sample, and your specified temperature stability conditions.
- The depth: this is where the conversion from feet to metres, if necessary, will be indicated.
- The conductivity: the conductivity, calculated as outlined above, for the sample being tested. Conductivities are stated in units of W/mK.

DB will next ask if you would like to process another sample with no changes. If you answer No, the program will terminate. If you answer Yes, the program will return you to the point in the program where you are required to input the sample data. For organizational purposes, you will find it most convenient to generate one DB output file for each sample. This one DB output file will include the data from each of the five trial runs (i.e. each of the five LabTech Notebook output files).

You should now assess you results. This can be done using any relevant analysis. Good results have been achieved consistently by calculating the average of the conductivity from the five trials, and the absolute percent difference of each individual conductivity from this average. If you find that the average of the five absolute percent differences is greater than 2%, throw out the point that is the most different from the average and perform the calculations again using only four points. If you find that you cannot achieve an average absolute percent difference of less than 2% with 3 points or more, you should retest the sample (conductivities based on only two measurements may tend to be unreliable.)