

USER MANUAL FOR IMPEDANCE SPECTROSCOPY MEASUREMENTS AT ISIS

N J G Gardner, S Hull, D A Keen and P Berastegui

ISIS Facility, Rutherford Appleton Laboratory, Chilton, Didcot,
Oxon, OX11 0QX

Table of Contents

1 Introduction *

1.1 Impedance Spectroscopy *

1.2 Ionic Conductors *

2 Experimental Method *

2.1 Solartron 1260 Impedance Analyser *

2.2 Eurotherm 800/2400 Series Temperature Controllers *

2.3 Sample Environment *

2.3.1 Vacuum Furnace *

2.3.2 Tube Furnace *

2.3.3 High-Pressure High-Temperature Cell. *

2.3.4 Low temperature measurements using a Closed Cycle Refrigerator *

3 Solartron Control Program (SOLCON.EXE) *

3.1 Outline of Operation *

3.2 Composition of Command File *

3.2.1 Eurotherm commands *

3.2.2 Miscellaneous commands *

3.2.3 Example command files *

3.3 Solcon Operation *

3.4 Output Files *

3.4.1 The .log file *

3.4.2 The .ord file *

3.4.3 The .dat file *

3.4.4 The journal.txt file *

4 Data Analysis Programs *

4.1 Data Manipulation (SigmaPlot) *

4.2 Real Time Immittance Fitting Program *

4.2.1 Introduction *

4.2.2 Data File Transfer *

4.2.3 Running Immfit *

4.2.4 Immfit Options *

4.2.5 Immfit Operation *

4.2.6 Immfit Output files *

4.2.6.1 The .out file *

4.2.6.2 The .par file *

4.2.6.3 The .pow file *

4.2.6.4 The .tem file *

4.2.7 Importing output files into SigmaPlot *

4.3 Equivcrt program *

5 Archiving, backup and file retrieving systems *

5.1 Outline of archive operation *

5.2 Directory and file server systems *

5.3 Copying files onto Windows NT server and local hard drive *

5.4 Backup operation onto zip disk *

5.5 Remote access *

Acknowledgements *

References *

Appendices *

Appendix A: Eurotherm & GPIB Addressing *

Appendix B: Fixing Problems *

- (a) The Impedance and Admittance Run Numbers do not match *
- (b) Solcon crashes when the sweep finishes on the Solartron *
- (c) Solcon does not read a newly created command file *
- (d) Solcon sometimes will not read a new program *

1. **Introduction**

1. **Impedance Spectroscopy**

At the start of the twentieth century, measurements of the ionic conductivity of a sample were taken by means of constant voltage direct current (D.C.) measurements. However, this measurement technique has many limitations associated with it. To make accurate measurements, the electrode behaviour must be such that ions are not blocked (acting as a capacitor) and the sample resistance must be the major component of the measured impedance. In most electrical systems, there are a wide variety of processes that contribute to the

measured impedance. These difficulties make it desirable to obtain such conductivity measurements over a wide range of alternating current (A.C.) frequencies, where the effect of these other "components" is not simply ignored, but each component can be isolated and the sample impedance obtained. Impedance spectroscopy is the study of sample resistivity over a wide range of frequencies ranging from several mHz to a few MHz. Each individual spectrum provides information about the resistive, inductive or capacitive elements, which together constitute the electrical behaviour of the sample.

Impedance spectroscopy is frequently used in the study of electrochemical systems. Since the arrival of automated high quality frequency response analysis (FRA) devices (such as the Solartron 1260), impedance or admittance (generically called immittance) measurements can now be obtained with considerable ease. The ability to interface these systems with PCs enables the multitude of data to be recorded and analysed in a way that previously would have been impossible. These measurements are taken over a large frequency range and enable different circuit elements, which have different time constants, to be resolved and have been particularly useful for the study of ionic conduction, grain boundary impedance, electrode/electrode interface characteristics and also mixed conducting solid electrode materials.

The measurements, which are taken in the frequency domain, can be described analytically by using an equivalent circuit as a model. In many cases the circuit elements may be easily estimated, using simple graphical means. For example, a single semicircle observed in the complex impedance plot, with the high frequency measurements converging upon the origin, (see Figure 1) may be modelled by a resistor in parallel with a capacitance (Figure 2), where the resistance is obtained by extrapolating the low frequency measurements onto the real axis. Semicircles like this are frequently observed in impedance spectroscopy measurements and are caused by a combination of a sample resistance in parallel with a sample capacitance.

This manual describes the procedure for taking ionic conductivity measurements using the Solartron 1260 Frequency Response Analyser at high temperatures at both ambient and high pressures and also at low temperatures using a closed cycle refrigerator (CCR). The high-pressure high-temperature neutron diffraction/conductivity cell has been developed at ISIS for work on the binary silver and copper monohalides to study the relationship between crystal structure and dynamical properties. This work has been jointly funded by a SERC research grant and a development grant from ISIS. The vacuum furnace and the low temperature conductivity apparatus have been built by collaboration between researchers at Oxford University and at ISIS (N J G Gardner (ISIS and Clarendon Laboratory, Oxford), D A Keen (ISIS), S Hull (ISIS) and, for the low temperature measurements, R McDonald (Clarendon) and A Mihut (Clarendon)). The tube furnace cell has been developed by P Berastegui and S Hull at ISIS. The availability of the listed equipment is heavily dependent upon usage and cannot be guaranteed. Enquiries should be addressed to Dr S Hull

2. Ionic Conductors

Fast-ionic conductors are materials with high ionic conductivity in the solid state. These materials are of considerable commercial value because of their potential application in the construction of solid state batteries and fuel cells. This technological importance has provided the motivation for studies of both their structure and their dynamics, with the aim of enhancing our understanding of the conduction mechanism and determining important factors for the production of better ionic conductors. Structural studies have shown that these materials often exhibit extensive disordering of the mobile ions amongst interstitial sites in the crystal structure, providing further evidence of the ionic nature of the conduction. The extent of the observed disorder and the energy barriers associated with hopping between the interstitial sites can be calculated from the measured conductivity.

Impedance spectroscopy is a powerful technique for the study of these materials, because it enables the bulk ionic conductivity to be resolved from other resistive or capacitive elements within these conductors. A single crystal ionic sample would show a semicircle in an impedance plot (see Figure 1), resulting from the bulk resistance and the bulk capacitance of the crystal. This is modelled by an equivalent circuit of a resistor in parallel with a capacitor and is shown in Figure 2. Many of these fast-ionic compounds undergo several phase transitions as they are heated. These transitions would cause single crystals to split up, so they are not commonly used for these measurements. A sintered powder pellet consisting of fine grains of the sample is connected via blocking graphite electrodes or unreactive metal (e.g. silver or platinum) electrodes to an impedance analyser. Impedance spectroscopy provides information about some or all of the following properties: the bulk grain conductivity, the grain boundary conductivity and the capacitances associated with these conductivities or the double layer capacitance.

A typical spectrum consists of a (depressed) semicircle and a spike (see Figure 3). This is interpreted in terms of two circuit elements connected in series: the first is the double layer capacitance (producing the spike) and the second is related to the properties of the bulk grain - a resistance in parallel with a capacitance (producing the semicircle). The double layer capacitance is caused by the geometry of the measurement electrodes, whereas the bulk grain resistance and capacitance are intrinsic sample properties. A further semicircle may be seen which relates to the grain boundary resistance and capacitance. Thus the observed spectrum may be described by the equivalent circuit shown in Figure 4. The impedance for this circuit is:

$$Z_{total} = \frac{1}{j\omega C_{dl}} + \frac{1}{\frac{1}{R_b} + j\omega C_b} + \frac{1}{\frac{1}{R_{gb}} + j\omega C_{gb}},$$

where C_{dl} is the double layer capacitance, R_b is the bulk sample resistance, C_b is the

bulk sample capacitance, R_{gb} is the grain boundary resistance and C_{gb} is the grain boundary capacitance.

At higher temperatures, the measured impedance does not behave as described above. As the thermally activated ionic conduction causes the impedance to decrease dramatically, the frequency associated with each semicircle ($\omega \sim 1/RC$) becomes considerably larger. Thus, one would only expect to observe these features at the highest of frequencies ($>1\text{MHz}$), which unfortunately are also the noisiest measurements. However, if the sample is in good contact with the electrodes, then the measured impedance over a wide range of frequencies is a good approximation to the resistance of the bulk of the sample. The grain boundary resistance at these temperatures becomes very small, as would be expected for an annealed sample.

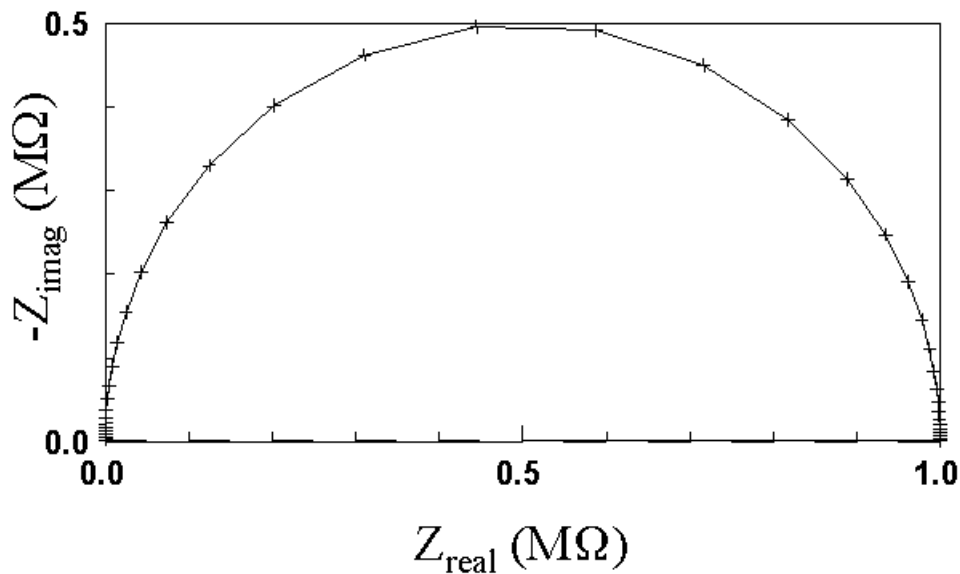


Figure 1. A simulation of the semicircle obtained for an impedance plot of a $1\text{ M}\Omega$ resistor in parallel with a 10 pF capacitor (an idealised ionic conductor). The crosses represent simulated ionic conductivity measurements from 0.1 Hz to 10 MHz at regular intervals in $\log(\text{frequency})$. There are 60 of these measurements over the whole frequency range.

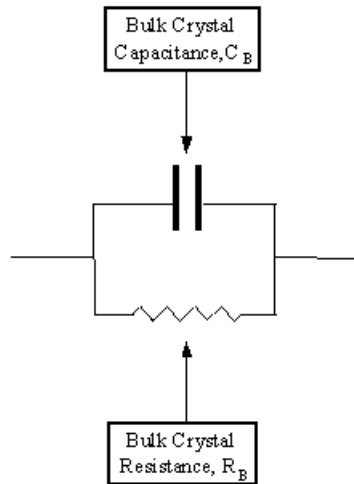


Figure 2. The equivalent circuit for an idealised ionic conductor, consisting of a single sample capacitance in parallel with the bulk crystal resistance.

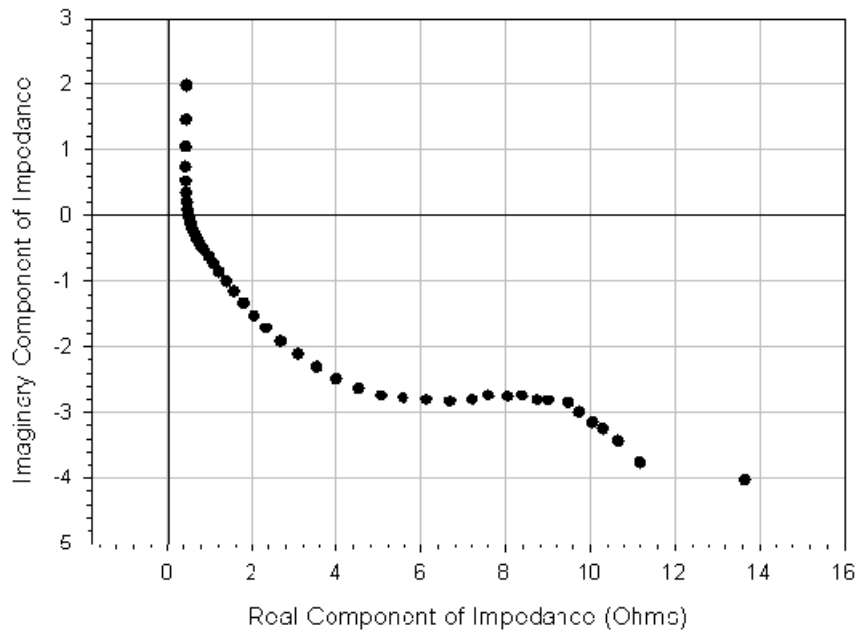


Figure 3. The impedance plot from copper iodide at $\sim 350^\circ\text{C}$ and 1 GPa. The partial semicircle and the beginning of a spike are clearly visible. The frequency range for the data shown is 0.1 Hz to 100 KHz.

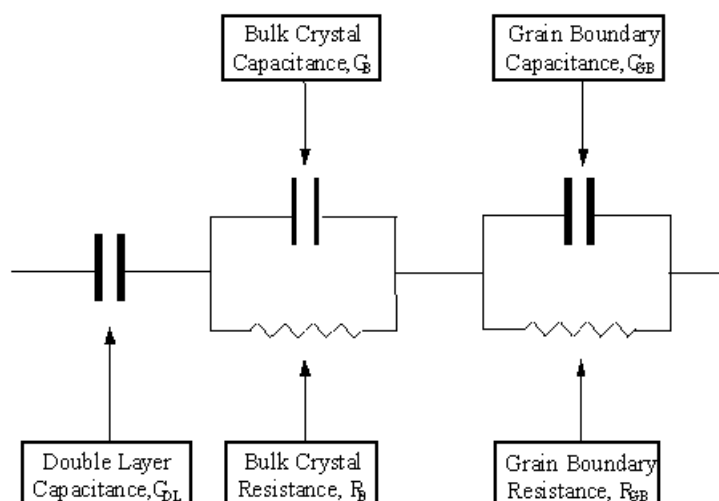


Figure 4. The Equivalent Circuit for a Polycrystalline Sample with Blocking Electrodes, showing the effect of grain boundaries and the double layer capacitance.

1. **Experimental Method**

A schematic diagram of the measuring apparatus for impedance spectroscopy experiments is shown in Figure 5. This consists of a furnace/pressure cell with two electrical contacts for measurement, the Solartron 1260 Impedance Analyser, which takes the immittance measurement, and a PC which directly controls the Solartron and monitors the temperature via a Eurotherm 800 or 2400 series temperature controller.

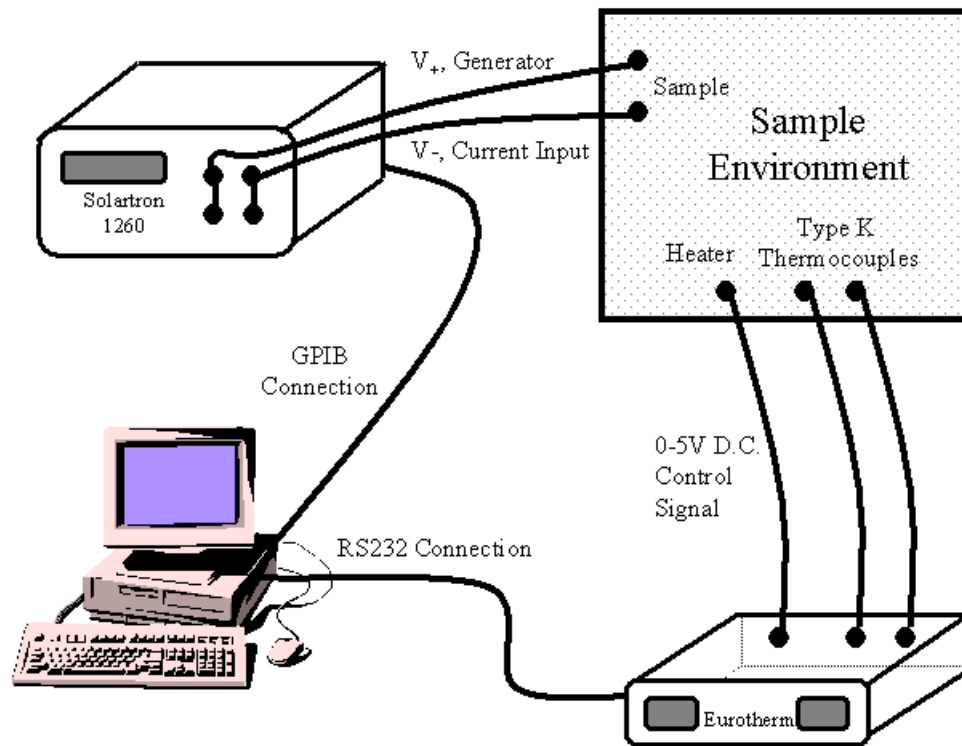


Figure 5. An overview of the measurement process. The Solartron makes the immittance measurement and sends the data back to the computer, whilst the Eurotherm controls the sample temperature and sends the data to the computer.

1. Solartron 1260 Impedance Analyser

The Solartron 1260 Impedance analyser or Frequency Response Analyser (FRA) measures the immittance over a frequency range of $10\mu\text{Hz}$ to 32MHz , via measurement integration, and auto integration, of the analyser input, for harmonic noise rejection. The generated voltage may be varied from 0V to 3V (up to 10MHz) or 1V (above 10MHz). The minimum measuring current is $6\mu\text{A}$ and the maximum is 60mA . The precision of measurements over this frequency range is shown in Figure 6. The instrument can be programmed to automatically "sweep" through a series of measurements and then to send these results to a PC via a GPIB-Type II interface.

The Solartron is capable of taking four point measurements, though for normal use, two point measurements have been found to be adequate, with the generator and V_+ inputs and the current and V_- inputs connected together, respectively.

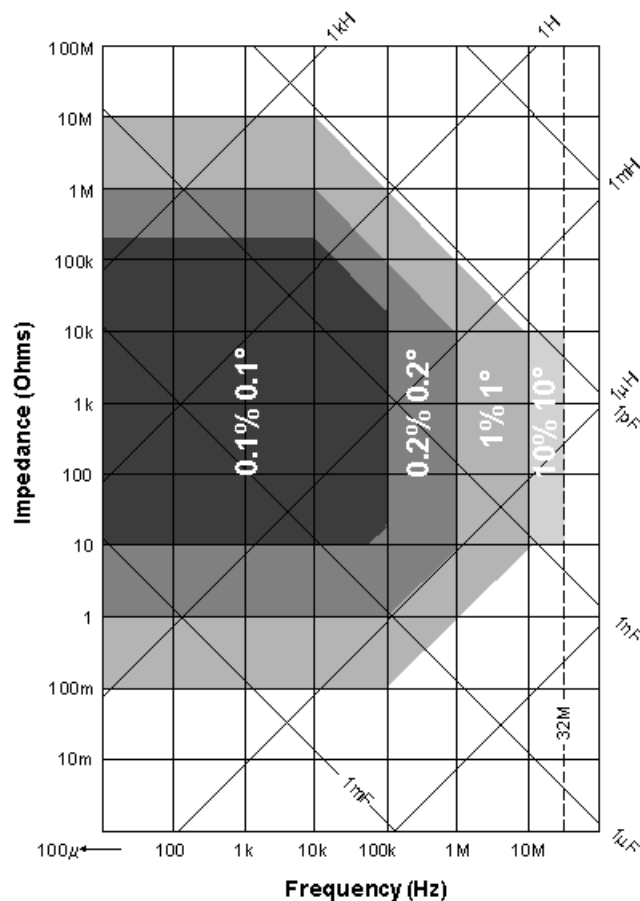


Figure 6. The sensitivity of Solartron FRA measurements at different frequencies (reproduced by permission from Solartron Instruments).

2. Eurotherm 800/2400 Series Temperature Controllers

The Eurotherm 800/2400 series communicating controllers provide two way asynchronous serial communication with a computer. They have the ability to supply a "power" control signal to stabilise the temperature to a specified level or even a specified rate of ramping. The Eurotherm specifies the power output in a manner defined by three parameters: the proportional band, the integral time and the differential time (see manual). Each Eurotherm is given a specific address, so the computer may be linked to several temperature controllers via the same RS232 connection and the addresses for various sample environments are shown in Table 1. Precise details about the addressing and interfacing of these Eurotherm temperature controllers with a PC are included in Appendix A. The control signal may have two forms: time-proportional or voltage-proportional. The time proportional signal is used in the high stability vacuum furnace, but voltage proportional signals are used for the tube furnace and the high-pressure high-temperature cell.

The Eurotherm may be instructed to set a particular temperature and control the sample environment such that this temperature is maintained until a new instruction is sent. Alternatively, the Eurotherm can be programmed to perform

a continual ramping at a specified rate up to a certain temperature, to dwell there, and then to ramp down in temperature at another (possibly different) rate.

The Eurotherm is able to send regular measurements of the temperature and the power output to the PC which are used to log the sample environment's performance.

Sample Environment	Eurotherm Address		
	0.1	0.2	0.3
Vacuum Furnace	Bottom T/C	Top T/C	N/A
Tube Furnace	Sample 1 T/C	Sample 2 T/C	Furnace T/C
High P Furnace	Bottom T/C	Top T/C	Load (t)
CCR	N/A	N/A	CCR T/C

Table 1. Thermocouple (T/C) arrangement for the various sample environments

3. Sample Environment

The four different sample environment apparatus available are listed in Table 2, which describes the temperature range provided by each apparatus and any other relevant factors that may help determine which apparatus to use for a particular experiment. The operation of each of these different sample environments is outlined in the paragraphs below.

Sample Environment	Temperature Range	Sample Dimensions Diameter, Length (mm)	Advantages	Disadvantages
Vacuum Furnace	RT-430°C	7-9, 5-15	High Stability (i.e. near to melting pt).	Slow heating cooling rate Susceptible to corrosion.
Tube Furnace	RT-1000 °C	7-11, 5-7	Fast Response. Inert platinum contacts & wires.	Less stability
High Pressure Furnace	RT-730 °C	4, 2.1-2.8	Measurement at pressure (<1.2 GPa)	Measurement circuit more complex & possible sample corrosion
CCR	10K-RT	4,5-7	Low T	N/A

Table 2. The temperature and pressure range obtainable using the four different sample environments.

1. Vacuum Furnace

A schematic of the vacuum furnace is shown in Figure 7. It consists of two blocks of stainless steel, each heated independently by means of two electric cartridge heaters (rated 60 V [A.C.], 1 A) and an A.C. power supply (120 W, 1 A). The sample is in contact with graphite electrodes, which in turn are in contact with copper blocks, which are electrically isolated from the metal blocks by means of a layer of pyrophillite. The signal is carried via copper blocks and copper wire through a Jaeger vacuum connection into coaxial wire connected to the Solartron FRA. The stainless steel blocks are supported by three threaded rods, which also support the heat shields. Nuts on the threaded rods can be tightened and so ensure that the sample is in good contact with the graphite.

Two thermocouples are situated in the centre of the stainless steel blocks and measure the temperature of the pyrophilite disks near to the sample. Two Eurotherm 2400 temperature controllers maintain a constant temperature in the top and bottom blocks independently, using a time proportional 0-9V (on/off) signal.

This furnace heats a relatively large thermal mass (~ 1 kg) and, consequently, is very stable (to within $\frac{1}{4}$ K at high temperature). However, this thermal stability comes at a price. The furnace takes a long time to cool (~12 hours), making rapid measurements impossible. A further disadvantage of this design is that it is vulnerable to attack by corrosive vapours from the sample.

To load the vacuum furnace, the following steps should be observed, referring to Figure 7:

- Lift up the furnace using a crane.
- Undo the wires holding heat shields 2 & 3 and remove the heat shields.
- Unscrew the screws attached to the inner heat shield.
- Slide the inner heat shield down, so that the sample area is clear.
- Unscrew the nuts holding the upper stainless steel block onto the sample.
- Lift up this block and move the nuts below it up, supporting it in this raised position.
- Ensure that there are pieces of pyrophilite inset into the grooves in each stainless steel block. (The pyrophilite in the top stainless steel piece may be loose and, if this is the case, it should be placed onto the copper block after the sample assembly has been assembled.)
- Carefully place a piece of graphite on the centre of the bottom copper block, using tweezers.
- Make a pellet using a standard size die and the hydraulic press near Polaris. The sample is ground into a powder and carefully loaded into the die using a spatula. Pressure is applied to the die to form the pellet, which is pushed out of the die, using the press.
- Carefully place the sample pellet (dimensions: ~8mm diameter, ~8mm length) onto the graphite electrode.
- Carefully place another graphite electrode on top of the sample.
- Put the upper copper block on top of the graphite, using the copper rod to hold the whole sample assembly together.

- Unscrew the nuts holding up the upper stainless steel block.
- Slowly allow the stainless steel block to drop down so that the sample assembly is held into place.
- Tighten the nuts above the stainless steel block.
- Check the connections and the sample resistance using a digital multimeter (DMM).
- Replace the inner heat shield
- Replace the two outer heat shields.
- Lower the vacuum furnace into the vacuum tank.
- Connect this to a vacuum pump and evacuate.
- Test the connections once more (each heater circuit should have a value of $\sim 110\ \Omega$. Pins 7 & 12 are connected to the upper heater circuit, pins 3 & 6 to the lower heater circuit and pins 9 & 10 to the sample), checking for short circuits to each other and ground.
- Connect the Jaeger connector
- Start the experiment!

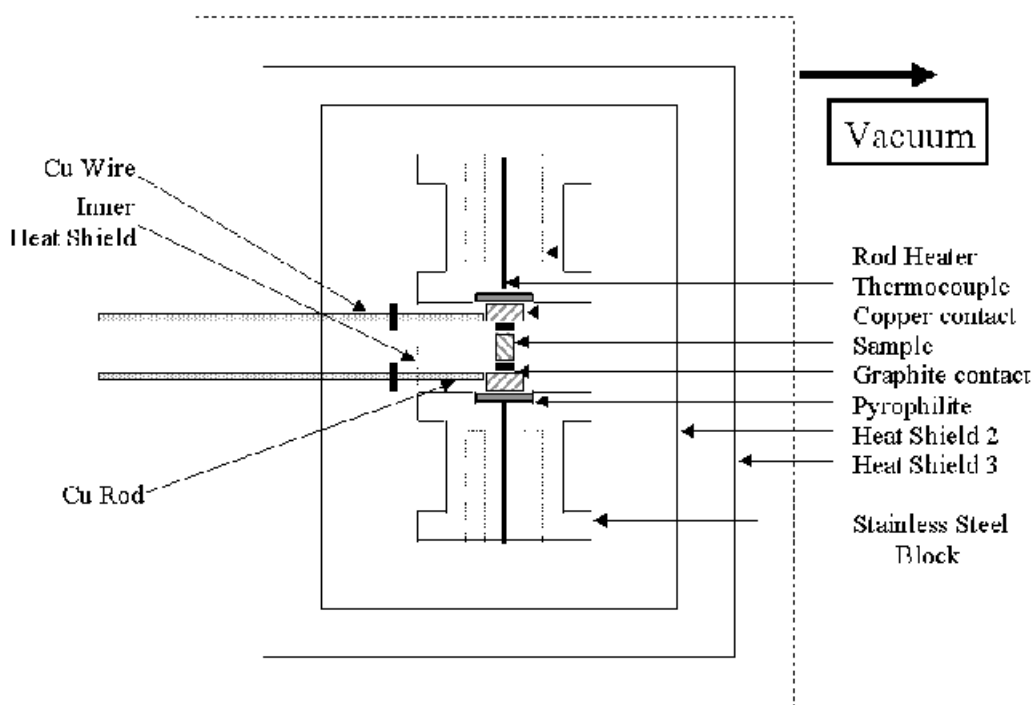


Figure 7. A schematic diagram of the vacuum furnace, showing the position of the sample in the furnace.

2. Tube Furnace

Measurements at higher temperature can be carried out using the cell shown in Figure 8. It consists of a long boron nitride rod with a removable hollow end piece of the same material, in which the sample and contact electrodes are placed. The maximum working temperature of this type of boron nitride is 1000°C in an inert atmosphere. The end piece has been shaped like a shoe for easy access to the sample position and provides a support to one of the electrodes. One of the platinum electrodes is thus attached to the base while the other has been welded to a platinum cup fitted to a boron nitride rod running along the centre of the long boron nitride rod. A spring at the other end provides the pressure needed to keep the sample in position and a screw is used to compress the spring. The platinum electrodes are welded to platinum wires and the points of contact strengthened by cement glue. The two platinum wires of 60 cm in length and two type K thermocouples are fitted inside four alumina tubes that have been inserted in holes drilled along the boron nitride rod. A cylindrical tube of boron nitride can be slid over the end piece and provides a protection for the sample, while a platinum foil wrapped along the inside provides also a protection for the boron nitride in case of failure of the spring at high temperature. The cell is attached to a Jaeger vacuum connector, to which the electrical contact to the Eurotherm and Solartron is plugged in, and it can be inserted inside a thick walled quartz tube and supported by screws to a metallic clamp around the quartz tube.

In order to protect the platinum electrodes, graphite electrodes are used and placed between the sample and platinum electrodes. The graphite electrodes act as inert blocking electrodes, causing the double layer capacitance which is observed by a spike in the impedance pattern. The use of graphite is motivated by being chemically inert without degrading the electrical properties of the sample and by providing a protection to the more expensive platinum electrodes. Depending on the exact position of the cell inside the furnace, the temperature difference between the sample and furnace heating zone is normally less than 15°C.

This set-up provides good electrical isolation with no conduction route other than through the sample and the furnace heating components do not show any severe effect on the impedance spectra.

In order to carry out an experiment, the following instructions should be observed:

- Hold the cell vertically, using a clamp and stand.
- Unscrew the spring tightening screw.
- Place a pellet (3-7mm long, 6-8mm diameter) (pressure dies of different diameters are available) below the rod providing the pressure and between two graphite electrodes in contact with the platinum electrodes. Move the rod upwards if necessary.
- Apply pressure by tightening the screw until the spring is totally compressed.
- Select the thermocouple closest to the sample for measurement of the sample temperature.
- Place the protecting cylindrical tube around the end piece.

- Place the cell inside the quartz tube and attach to the clamp using the set of screws.
- Evacuate the sample (or use a gas flow if desired).
- Plug in the Jaeger contact to the tube cell.
- Begin the experiment!

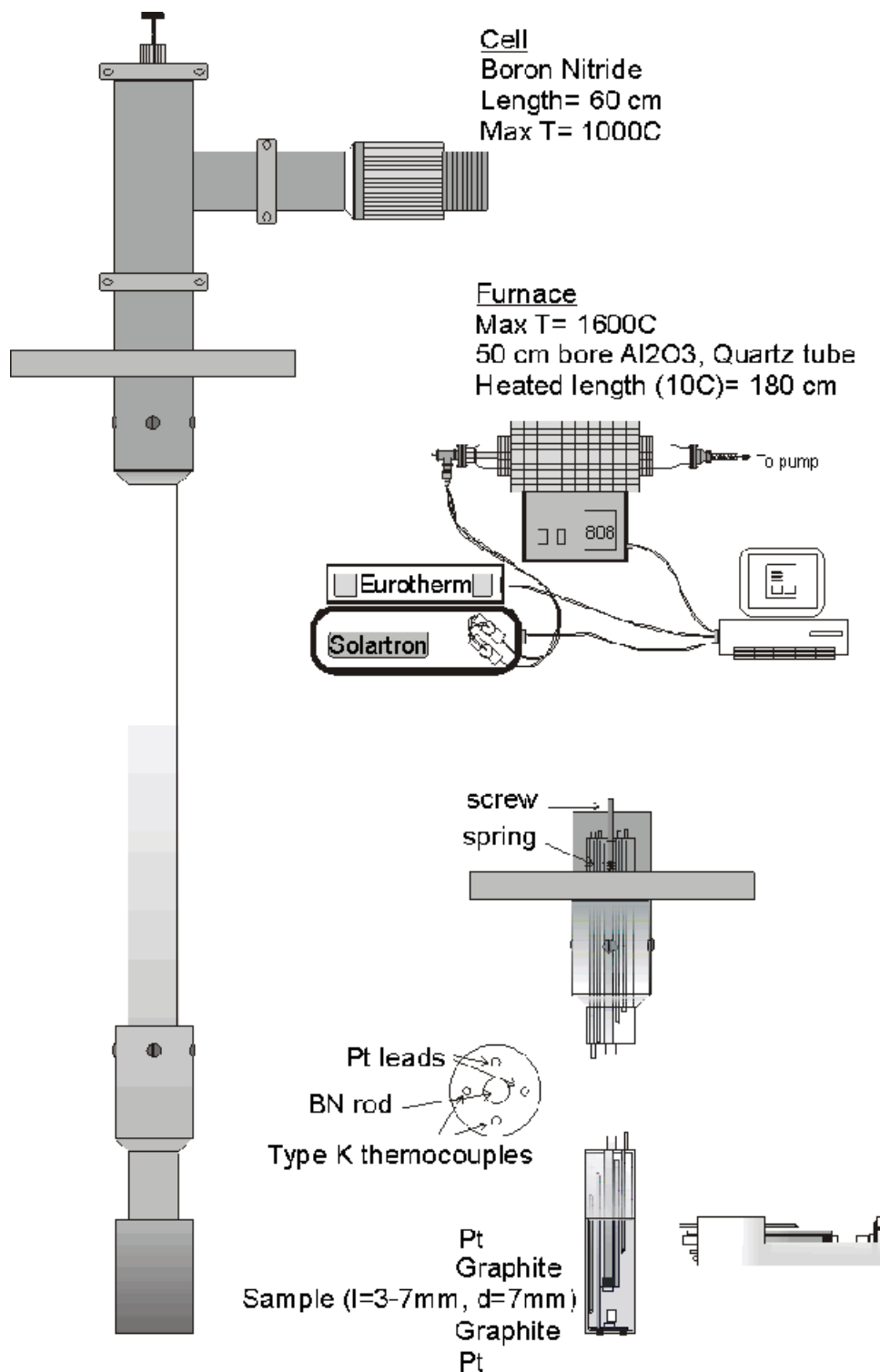


Figure 8. Diagrams showing the cell for the tube furnace.

2. High-Pressure High-Temperature Cell.

A neutron high-pressure, high-temperature (Hiphit) cell has been modified to allow two point impedance spectroscopy measurements to be made over a wide region of p-T space. The design and features of this cell and the sample setup are shown in Figure 9 - Figure 18.

The sample (of dimensions 2-3mm long and 4.0mm diameter) is held between two graphite electrodes, which are in contact with tungsten carbide pistons. The sample is contained inside a boron nitride ceramic block, which is inert and unreactive. One of the tungsten carbide pistons is longer than the other to allow a small ring of pyrophyllite to be placed on top of the quartz. Pyrophyllite is very soft and is compressed allowing pressure to be maintained on the sample, without destroying the quartz. A diagram of this arrangement is shown in Figure 9.

The copper foil around the outer sheath of the thermocouple, which is electrically isolated from the thermocouple wires, is used to take the impedance spectroscopy measurements. It is positioned in good electrical contact with the tungsten carbide piston using high-temperature copper paste (99.99%), which also improves the thermal conductivity. It is insulated from the outer body of the cell and also the heater circuit by means of the insulating alumina pieces.

The cell is heated by passing a large DC current through a graphite tube surrounding the sample. This tube is isolated from the sample by using more pyrophyllite, as shown in Figure 9. The outer ring of copper provides contact between the outside of the larger tungsten carbide pistons and the graphite. The resistance of the heater circuit is quite small ($\sim 0.05 \Omega$), allowing the 8V 350A power supply to pass large heating currents through the graphite, even at relatively low voltages. At the highest temperatures, the thin layer of graphite seems to have a relatively unstable resistance and careful setting of the PIDs is required to ensure that temperature control is maintained.

The cell is pressurised by using a manual hydraulic press to increase the pressure on the sample via a load cell attached to the top piston. A calibration curve for sample pressure against applied load is shown in Figure 10. As the cell is pressurised, it is important to ensure that the load cell is properly located and that its lead does not catch on the side of its mounting.

The sample is prepared in a specially designed die. The sample is ground to a fine powder and then placed in the die between two small hardened steel pistons, with polished surfaces. This assembly is placed on a steel base and pressurised to a pellet (max 0.5t load) using a manual hydraulic press. The solid stainless steel base is then replaced with another base so that the sample can be pushed out of the die by the press. A diagram of this arrangement is shown in Figure 11.

To prepare the cell for an experiment, the following steps must be observed:

- Lightly grease the outer faces of the castellated pieces (A), the outer faces of the bicone (B) and the stainless steel pins (C) before placing them into the cell [Figure 12]
- Place the large nut [Figure 13 (D)] required to mount the cell on the hydraulic ram over the cell and then tighten the two nuts at the bottom of the cell, leaving at least a turn free on the bottom thread for tightening during the prestressing of the alumina. This is achieved by tightening the larger nut until it is fully tight, releasing it by $1\frac{1}{4}$ - $1\frac{1}{2}$ turn and then tightening the smaller counterthreaded nut [see Figure 14, (E)].
- Prestress the alumina, tightening the bottom nuts (using the larger spanner), whilst pressurising the bicone to 15 t, using the small block (F) to apply pressure onto the castellated stainless steel pieces [see Figure 14]. Note the use of the safety shield, whilst the press is in operation.
- Form the sample assembly [Figure 9]:
 - Insert the sample (G) into the quartz (H)
 - Insert graphite electrodes top and bottom (I)
 - Place pyrophilite spacer (J) onto WC piston (K)
 - Place inner tungsten carbide pistons (K) (ensuring that there is a little copper paste in each piston to provide good electrical contact with the thermocouple) into the quartz.
 - Insert into pyrophilite cylinder (L).
 - Place alumina insulator pieces (M) onto the end of the tungsten carbide pistons (K).
 - Insert into graphite cylinder (N).
 - Place copper tubes (O) around alumina insulators (M).
 - Insert large tungsten carbide pistons (P) onto alumina insulators (M).
 - Insert alumina insulator pieces into holes in the large tungsten carbide pistons (Q). (These insulator pieces both provide insulation and also guide the thermocouples into the inner tungsten carbide pistons).
- Insert the sample assembly into the bicone using the shaped tray (R) [Figure 15 and Figure 16].

- Add top and bottom piston assemblies, ensuring that each copper rod is tightly screwed into the tungsten carbide piston base [Figure 17].
- Screw the nuts around the piston assemblies, using, if necessary, the smaller spanner [Figure 16 (S)].
- On the 'top' of the cell, add insulator and then screw the copper heater rod (T), passing through the groove in the piston assembly piece, at 90° to the copper rod, which is attached to the piston cup [Figure 16].
- Insert insulator piece [Figure 16 (U)] that holds the copper in position and isolates the thermocouple inserted through the central hole at the top of the cell.
- Insert the thermocouple fully into the centre hole, ensuring that the inserted length is sufficient for contact between the inner tungsten carbide piston and the thermocouple and then attach the insulator [Figure 16 (V)] onto the end of the piston with PVC insulating tape.
- Use PVC tape to attach some alumina insulator pieces between the thermocouple on the top of the cell and the point where the load cell applies pressure onto the cell.
- Mount the cell onto the hydraulic ram assembly [Figure 18] and screw the cell onto the flange. Note the locating groove and ensure that the cell is properly located before the large nut is tightened.
- Connect the copper rod onto the bottom of the cell [Figure 16].
- Connect the copper rod on the top of the cell onto the copper wire attached to the heater circuit connection, using a 2 mm alum key.
- Insert the thermocouple down into the bottom of the cell, ensuring that sufficient length is inserted to allow contact between the thermocouple and the inner piston.
- Attach the copper wires to the sheathing of the thermocouples using the copper plates attached to the thermocouples. To reduce the circuit resistance attach the copper wires as close as possible to the point where the thermocouple leaves the cell.
- Ensure that there are no short circuits and all wiring is insulated with PVC tape, if necessary.
- Screw in the water pipe attachments and plug in the water pipes.
- Invert the cell and insert into the vacuum tank.
- Evacuate and connect the heater cables, the jaeger and the water pipes.

- Check connections again
- Start the experiment!

After an experiment, the following steps need to be taken to release the sample assembly:

- Remove cell from hydraulic ram assembly
- The top and bottom thermocouples are removed.
- The top and bottom copper heater rods are unscrewed from the connections to the cell.
- Insulator pieces around these attachments are removed.
- The piece holding the top piston may be unscrewed by hand from the Hiphit cell and the piston may be removed.
- The heater rod attached to the top piston is unscrewed and removed.
- The hiphit cell is placed in a press and the bottom piston pressed to ~ 1 t load.
- The piece holding the top piston should now be free to be unscrewed and removed.
- If possible, by hand, remove the tungsten carbide pistons from the bicone.
- The prestress on the bicone will prevent the cell from being unscrewed to release the castellated stainless steel pieces and the bicone being removed. The following steps need to be observed:
 - Use the small block and the press to exert pressure on the castellated pieces, whilst attempting to unscrew the large nut. A load of at least 15t will be required to release the thread and a maximum load of 25t may be applied if necessary.
 - Once the thread is released unscrew the nuts by several turns to ensure that the thread does not tighten again when the load is released.
 - Gently release the load.
 - Remove the castellated pieces and the bicone. (If these are stuck, they may need to be removed carefully, using a press. Consult a technician if help is required with this procedure.)
- The bicone may need to be physically broken by gentle tapping to remove the tungsten carbide pistons and the sample.

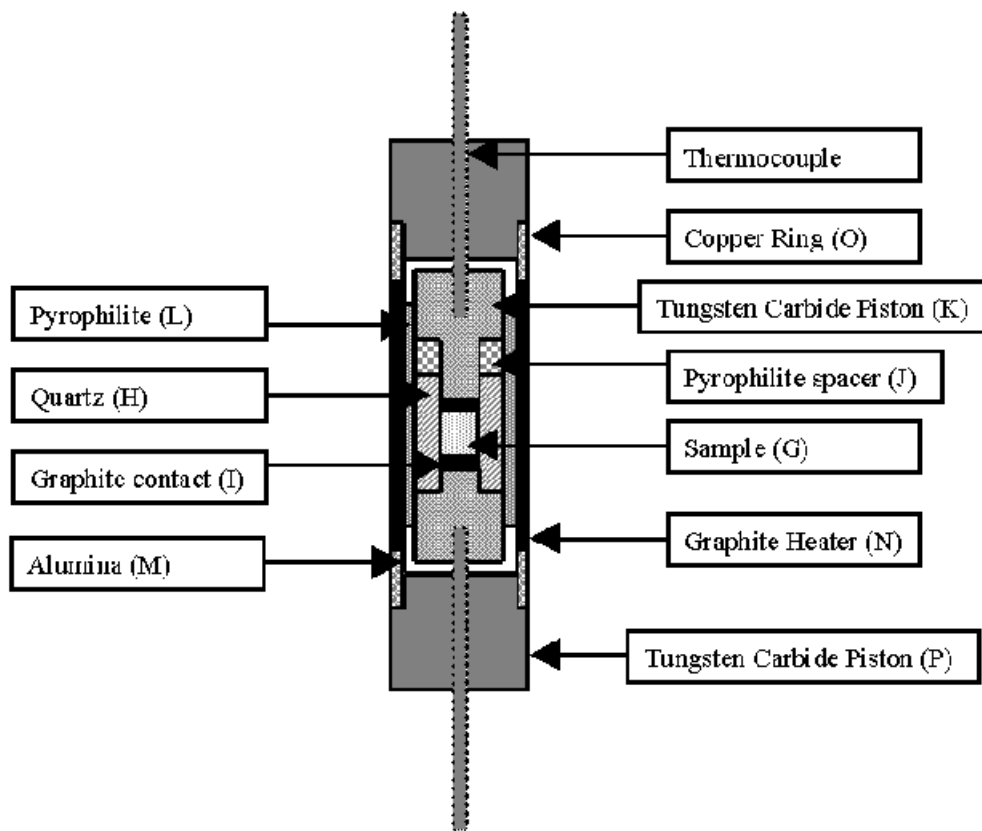


Figure 9. The sample assembly for the Hiphit cell.

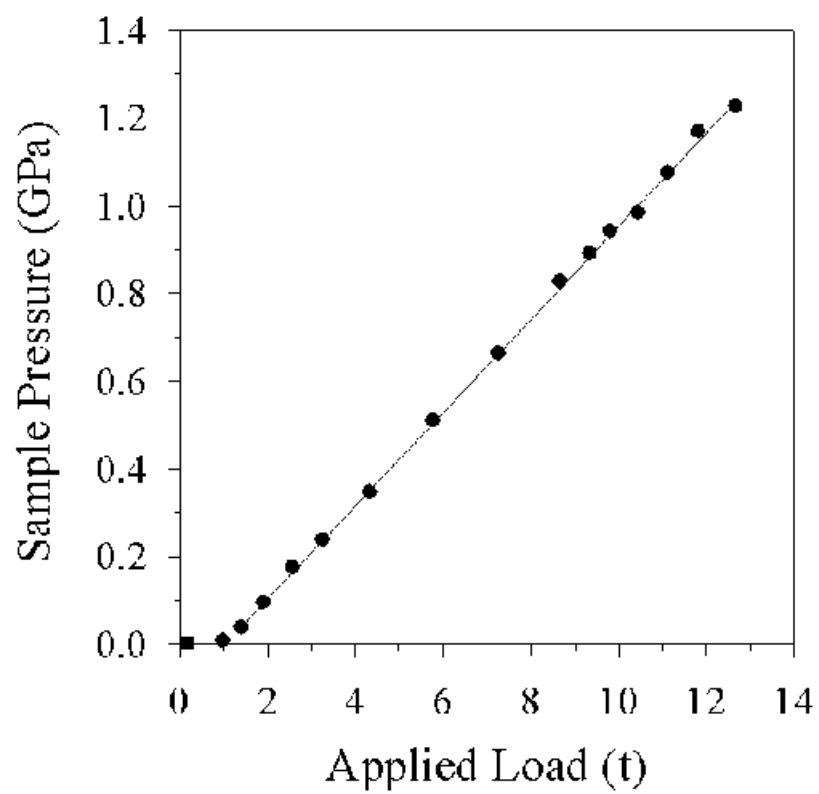


Figure 10. The calibration curve showing the relationship between applied load and sample pressure.

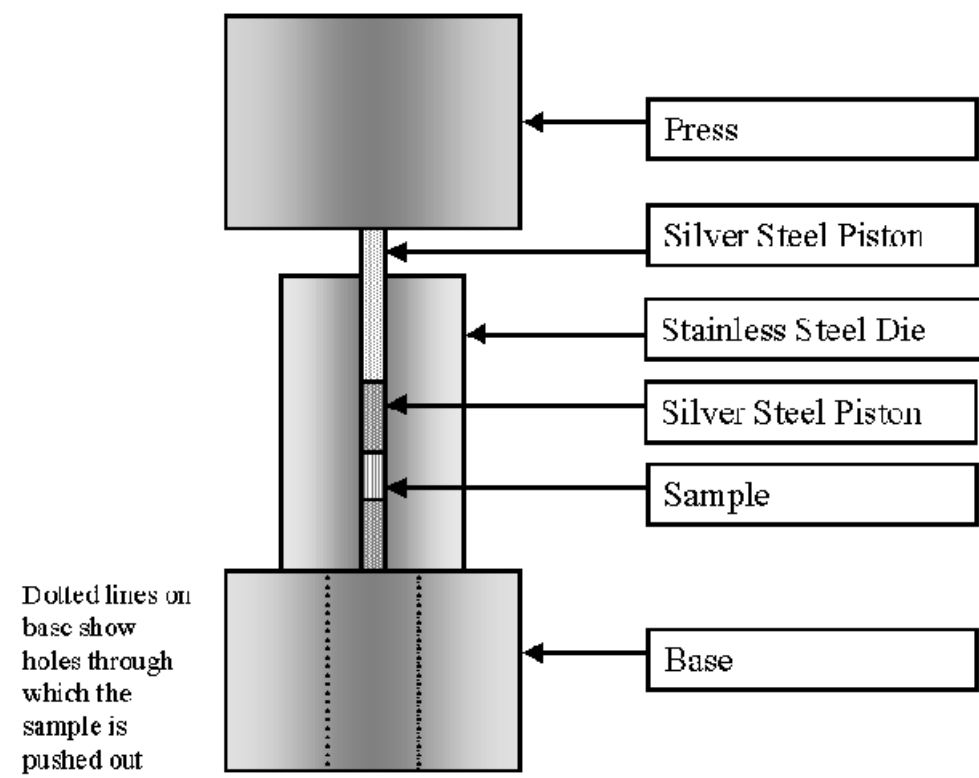


Figure 11. The specially designed die, showing how to prepare a pellet for use in the high pressure cell.

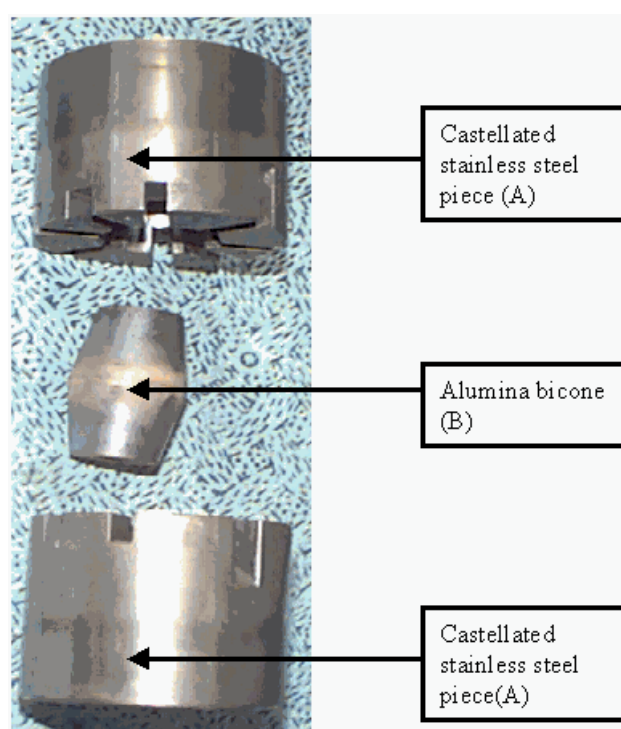


Figure 12. The bicone and the castellated stainless steel pieces.

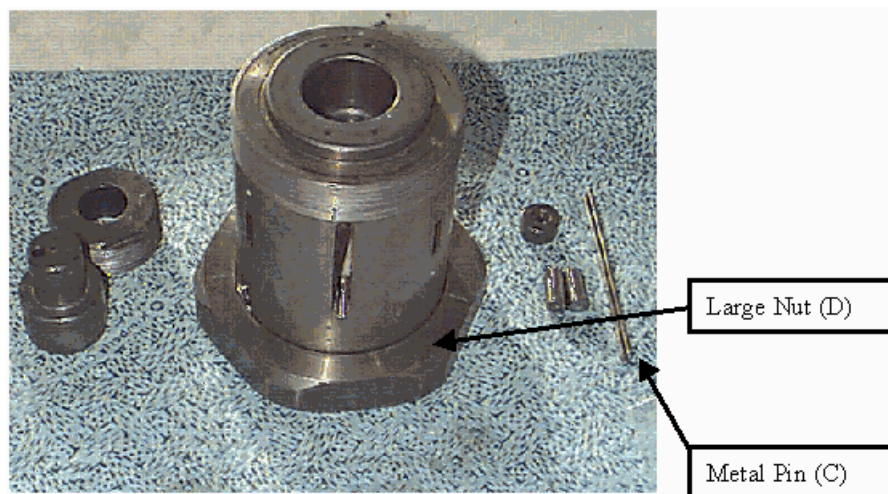
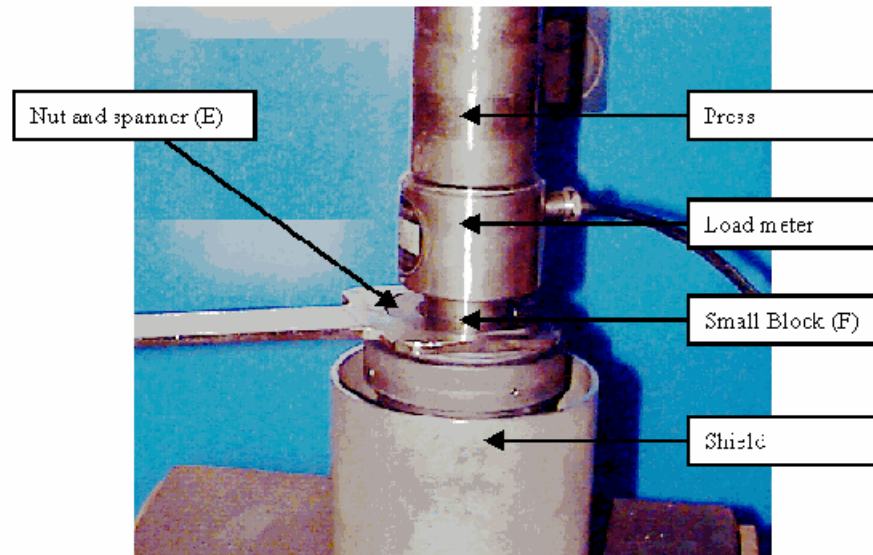


Figure 13. The castellated steel pieces after they have been inserted into the cell.



**Figure 14. Prestressing the alumina bicone.
Note the use of the safety shield in this operation.**

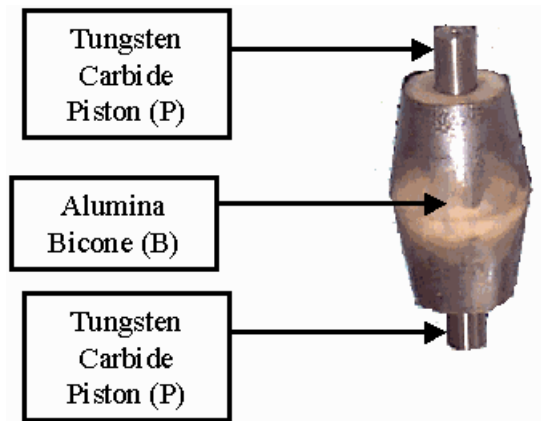


Figure 15. The sample assembly inserted into the alumina bicone.

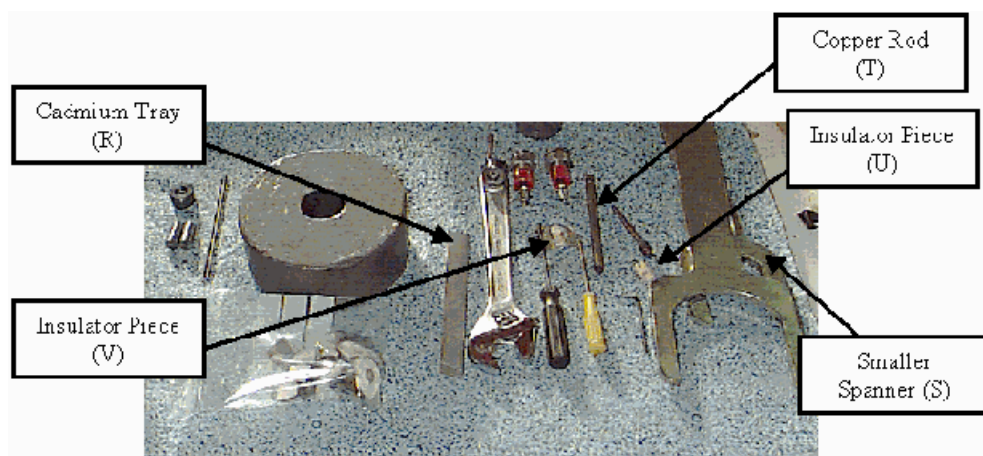


Figure 16: Connecting pieces and tools for the Hiphit Cell. Note the large circular lead block, which is used for standing the cell upright, with the top upwards, when the copper rod for the heater circuit protrudes above the piston piece.

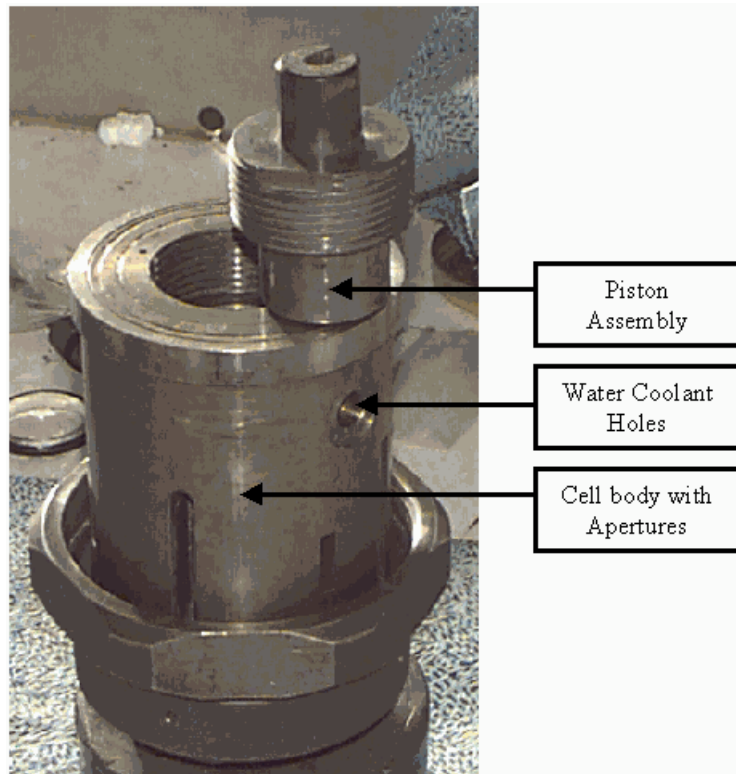


Figure 17. An example of a piston assembly, showing the groove at the top of the cell, through which the heater rod and the thermocouple leave the cell, when it is attached onto the hydraulic assembly.

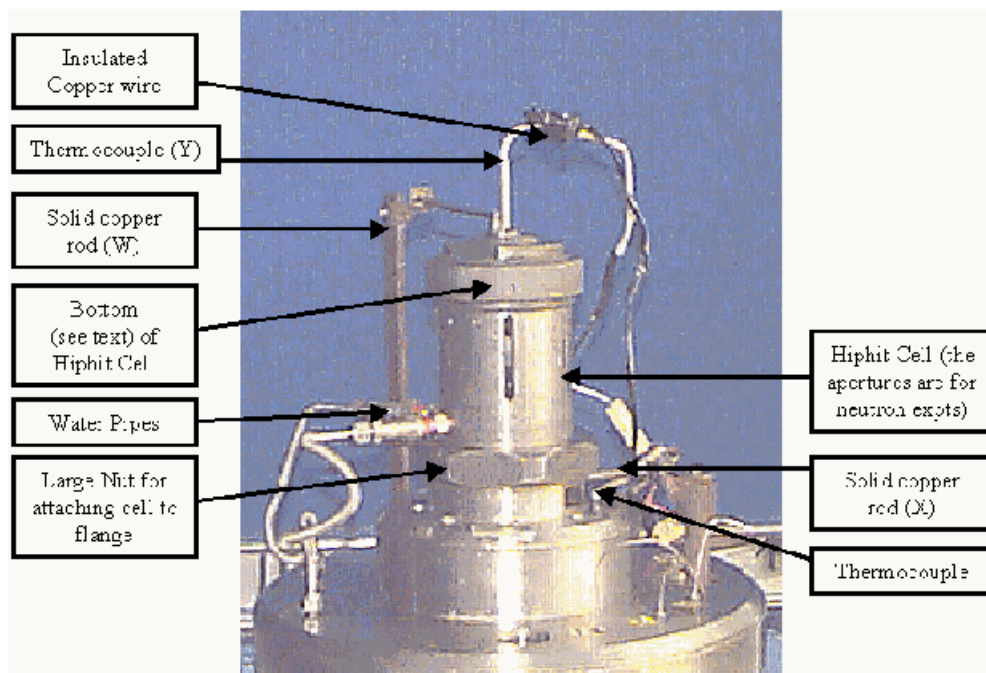


Figure 18. The Hiphit cell mounted onto the hydraulic ram assembly,

with the water pipes, heater cables and thermocouples connected to the cell.

2. Low temperature measurements using a Closed Cycle Refrigerator

2 or 4 point measurements may be taken in one of the ISIS Closed Cycle Refrigerators (CCRs). Fine copper wires are soldered onto the contacts on the sample and these wires are soldered to a piece of circuit board (see Figure 19). The circuit board is connected to microcoaxial cables, which pass through a Jaeger vacuum port and are connected to the Solartron. The circuit board is mounted onto a thermally conducting copper stand, which is then bolted onto the bottom of the CCR.

A standard Eurotherm cold controller is used to power the heater circuit, which enables temperature control of the CCR. A standard rhodium-iron sensor is used for temperature measurement. There is a considerable proportion of CPU time and so data analysis or data fitting are normally best done on other machines (PCs or VAX/VMS computers).

There are numerous different output files generated by this program, which all have different extensions. The .dat files record the immittance measurements. Two journal files (journal.txt and journal1.txt) record the temperature for each immittance measurement. The .log files record the time, date, measured temperature, and power output levels from the Eurotherm temperature controllers. The .ord files record the commands sent to the Eurotherm temperature controllers. A graphic of the relationship between these files and the data measurements is shown in Figure 20.

File	Location	Description
a*****.dat	c:\gpiib-pcw\prog\data\admit	Admittance data
s*****.dat	c:\gpiib-pcw\prog\data\imped	Impedance data
s*****.log	c:\gpiib-pcw\prog\log	Temperature log file
s*****.ord	c:\gpiib-pcw\prog\log	Eurotherm communication file
journal.txt	c:\gpiib-pcw\prog	Journal file of impedance measurements
journal1.txt	c:\gpiibj-pcw\prog	Journal file of admittance measurements

Table 3. The location of data files created by Solcon.

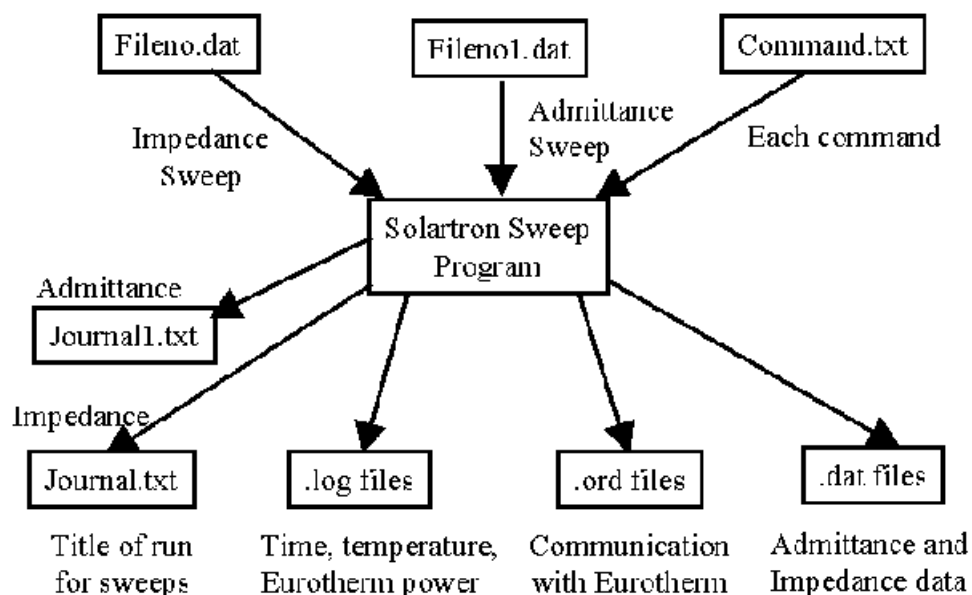


Figure 20. The relationship between the various files used by Solcon.

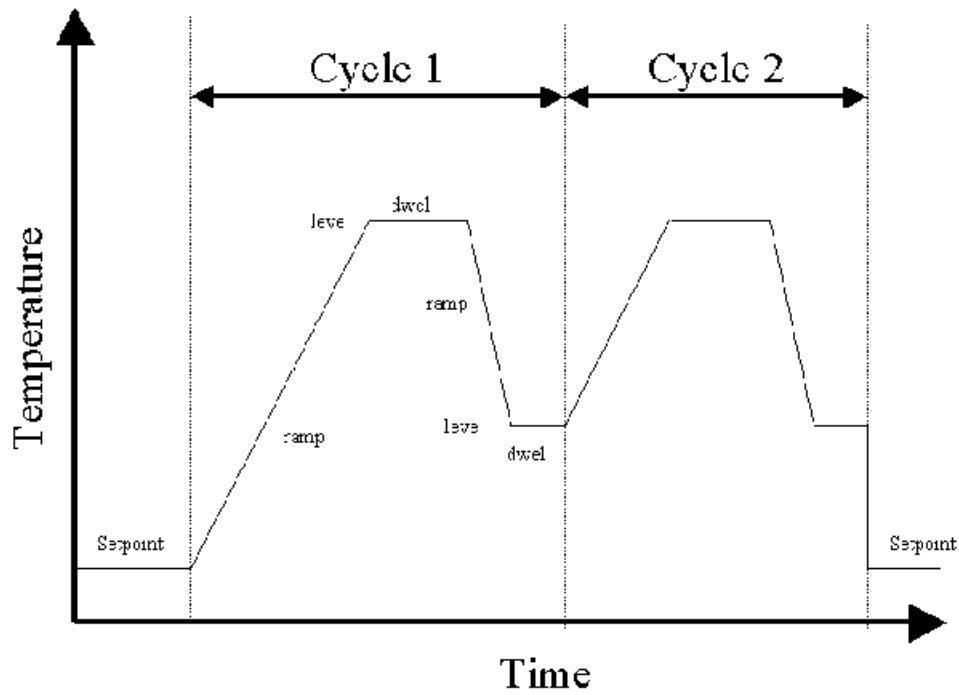


Figure 21. An idealised temperature measurement as the Eurotherm ramps up in temperature, indicating the parameters which are set before the program is run.

Set derivative on Eurotherm 2 to x

deri3::x

Set derivative on Eurotherm 3 to x

dwell1::x

Set length of time Eurotherm 1 remains at level to x minutes

dwell2::x

Set length of time Eurotherm 2 remains at level to x minutes

dwell3::x

Set length of time Eurotherm 3 remains at level to x minutes

hold1

Pause program on Eurotherm 1

hold2

Pause program on Eurotherm 2

hold3

Pause program on Eurotherm 3

imped::x

Start impedance "sweep" for x.xx minutes

inte1::x

Set integral on Eurotherm 1 to x minutes

inte2::x

Set integral on Eurotherm 2 to x minutes

inte3::x

Set integral on Eurotherm 3 to x minutes

level1::x

Set level on Eurotherm 1 to x °C

leve2::x

Set level on Eurotherm 2 to x °C

leve3::x

Set level on Eurotherm 3 to x °C

log:::x

Change interval between temperature recordings to x minutes

loop1::x

Set number of times Eurotherm 1 performs the program

loop2::x

Set number of times Eurotherm 2 performs the program

loop3::x

Set number of times Eurotherm 3 performs the program

maxp1::x

Set max_power on Eurotherm 1 to x %

maxp2::x

Set max_power on Eurotherm 2 to x %

maxp3::x

Set max_power on Eurotherm 3 to x %

prop1::x

Set proportional band on Eurotherm 1 to x

prop2::x

Set proportional band on Eurotherm 2 to x

prop3::x

Set proportional band on Eurotherm 3 to x

ramp1::x

Set ramp on Eurotherm 1 to x K/hour

ramp2::x

Set ramp on Eurotherm 2 to x K/hour

ramp3::x

Set ramp on Eurotherm 3 to x K/hour

rese1

Reset program on Eurotherm 1

rese2

Reset program on Eurotherm 2

rese3

Reset program on Eurotherm 3

stop1

Tell Solcon that this is the end of the program for Eurotherm 1

stop2

Tell Solcon that this is the end of the program for Eurotherm 2

stop3

Tell Solcon that this is the end of the program for Eurotherm 3

temp1::x

Set temp1 to x °C

temp2::x

Set temp2 to x °C

temp3::x

Set temp3 to x °C

title::

1. Eurotherm commands

The command name for each Eurotherm command is of a specific format: it consists of a four digit command immediately followed by a number, which indicates the address of the Eurotherm.

Initially, values such as the proportional band, the integral, the derivative are specified. Solcon is able to control the temperature by two different methods. The first simply sets the temperature on the

Eurotherm, waits for equilibrium to occur and then measures the immittance. The second method maintains a constant temperature ramp and records the immittance as the temperature is increased. The first control method is straightforward, simply using commands to set the temperature and the maximum power, to wait and then to take measurements on the Solartron. Program setting on the Eurotherm is slightly more complex and a schematic of this is shown in Figure 21. The various parameters for the temperature levels, heating rate and the dwell time are entered and then Solcon is started by using the "begi" command.

Note that each Eurotherm needs to be informed that the program has finished (by including the command stop into the control program, which is read by Solcon). In writing the command file, the same commands (e.g. ramp or leve commands) must not be bunched together. The command file should contain the ramp commands in sequence. There must always be one "leve" command, one "dwel" command and one "ramp" command before the next "ramp", "leve" or "dwel" command is entered. In section 3.2.3, some example command files demonstrate how to use these programmable Eurotherms.

2. Miscellaneous commands

It is important to begin each command file with the command "title". This sets the title that will be recorded in the journal.txt file for each immittance run. The title of the file should indicate the chemical formula of the sample and any relevant preparation conditions. It is not necessary to record the temperature of the run or the current load, because they are recorded in the log files.

The command "wait:" allows for a pause in the command file, whilst the computer continues to log and record the measured temperature and the power output. The number associated with the "wait : " command specifies the time in minutes for which Solcon performs this operation.

The command "log :: " allows for a change in the rate that the temperature is logged. By default the temperature is measured every 0.5 minutes. The number associated with this command is the new interval in minutes for temperature logging.

3. Example command files

Listed in the following sections are three example command files that use most of the features available.

Example 1: A listing for a command file the Hiphit cell

```
title::AgI 14.06t
```

```
prop1::16
```

```
intel::100
```

```
der1::20
```

```
maxp1::20
```

```
templ::25
```

```
wait:::6
```

```
imped::1.2
```

```
admit::1.2
```

```
maxp1::25
```

```
templ::40
```

```
wait:::6
```

```
imped::1.2
```

```
admit::1.2
```

NB The symbols (:) included in the commands represent the number of "spaces" included between the command and the data string accompanying the command.

Explanation of the steps involved in this program:

- The title recorded in the journal.txt file is **ONT FACE="Courier New" SIZE=1>50**

```
dwel3::120
```

```
ramp3::3
```

```
loop3::2
```

```
temp3::20
```

```
begin
```

```
imped::1.2
```

```
admit::1.2
```

```
imped::1.2
```

```
admit::1.2
```

[The imped and admit commands continue many more times, so that the impedance and admittance are continually measured]

NB The symbols (:) included in the commands represent the number of "spaces" included between the command and the data string accompanying the command.

Explanation of the steps involved in this program:

- The title recorded in the journal.txt file is "PbKF2 in tube furnace".
- The set point 1 in the program is set to 300 °C.
- The time spent at set point 1 is set to 60 minutes.
- The rate of ramping to set point 1 is set to 2 °C per minute.
- The set point 2 in the program is set to 50 °C.
- The time spent at set point 2 is set to 120 minutes.
- The rate of ramping to set point 2 is set to 3 °C per minute.
- The program will run two times.
- The current setpoint is set to 20 °C.
- The program starts.
- An impedance measurement is made while temperature is logged over the next 1.2 minutes.
- An admittance measurement is made while temperature is logged over the next 1.2 minutes is started.
- An impedance measurement is made while temperature is logged over the next 1.2 minutes.
- An admittance measurement is made while temperature is logged over the next 1.2 minutes is started. ... etc.

This program causes the tube furnace to heat at 2 °C per minute to 300 °C, where it remains for 120 minutes. Then the furnace cools at 3 °C per minute to 50 °C, where it remains for 60 minutes. This cycle is repeated twice. As the sample is heated and cooled, the impedance and admittance are continually measured.

Example 3: Heating AgI in the pressure cell

```
title::AgI 3.0t Load
level::230
```

```

dwell::30

ramp1::1

level::25

dwell::45

ramp1::2

loop1::3

stop1

templ::20

begin

imped::1.2

admit::1.2

imped::1.2

admit::1.2

[The imped and admit commands continue many more times, so that the impedance and admittance are
continually measured]

NB The symbols (:) included in the commands represent the number of "spaces" included between the
command and the data string accompanying the command.

```

Explanation of the steps involved in this program:

- The title recorded in the journal.txt file is "AgI 3.0t Load".
- The set point 1 in the program is set to 230 °C.
- The time spent at set point 1 is set to 30 minutes.
- The rate of ramping to set point 1 is set to 1 °C per minute.
- The set point 2 in the program is set to 25 °C.
- The time spent at set point 2 is set to 45 minutes.
- The rate of ramping to set point 2 is set to 2 °C per minute.
- The program is set to run three times.
- This states that the program has finished is the end of the program.
- The current setpoint is set to 20 °C
- The program starts.
- An impedance measurement is made while temperature is logged over the next 1.2 minutes.

- An admittance measurement is made while temperature is logged over the next 1.2 minutes is started. ... etc.

This program causes the hiphit cell to heat at 1 °C per minute to 230 °C, where it remains for 30 minutes. Then the furnace cools at 2 °C per minute (or its maximum cooling rate if that is slower) to 25 °C, where it remains for 45 minutes. This cycle is repeated three times. As the sample is heated and cooled, the impedance and admittance are continually measured.

The screenshot shows a window titled "Solartron Controller". Inside the window, there are several input fields and buttons arranged in two columns. On the left side, there are four buttons labeled "Filename", "Sweep Setup", "Start Measurements", and "Exit". On the right side, there are five input fields labeled "Command", "Value", "Message Sent", "Errors", and "Sweep Number". At the bottom of the window, there are three input fields labeled "Temp 1 (bottom)", "Temp 2 (top)", and "Load (t)".

Figure 22. The Solcon Window, showing the screen as it appears on the PC.

2. Solcon Operation

Figure 22 shows the window obtained by double clicking on the "Solartron controller" icon on the PC. Click on the "Filename" button and you will be asked to specify the name of the command file. If you give the name "file.txt", Solcon "_Toc417183219"> Output Files

Solcon outputs various different files to different directories. Each of these files is listed below along with a brief explanation of its contents.

3.4.1 The .log file

An example of a .log file is listed on the next page. It has the following columns, each separated by a comma.

- The day of the year
- The month of the year
- The year
- The time in hours
- The time in minutes
- The time in seconds
- "p1", the name for the power fed out by the first Eurotherm
- The value of p1 in terms of the percentage power output
- "p2", the name for the power fed out by the second Eurotherm
- The value of p2 in terms of the percentage power output
- "p3", the name for the power fed out by the third Eurotherm
- The value of p3 in terms of the percentage power output
- "t1", the name for the temperature on the first Eurotherm
- The value of t1 in C
- "t2", the name for the temperature on the second Eurotherm
- The value of t2 in C
- "t3", the name for the temperature on the third Eurotherm
- The value of t3 in C

```
29,01,1998,17,03,40,p1,028.70,p2,100.00,p3,050.00,t1,0098.90,t2,0071.10,t3,0000.80
29,01,1998,17,04,10,p1,028.70,p2,100.00,p3,050.00,t1,0099.00,t2,0070.00,t3,0000.80
29,01,1998,17,04,40,p1,029.60,p2,100.00,p3,050.00,t1,0099.00,t2,0069.60,t3,0000.81
29,01,1998,17,05,10,p1,029.60,p2,100.00,p3,050.00,t1,0099.00,t2,0069.30,t3,0000.80
29,01,1998,17,05,40,p1,029.70,p2,100.00,p3,050.00,t1,0099.00,t2,0069.20,t3,0000.81
```

3.4.2 The .ord file

An example of a .ord file is listed below. It has the following columns, each separated by a comma.

- The day of the year
- The month of the year
- The year
- The time in hours
- The time in minutes
- The time in seconds
- The command word
- The command value
- The communicated word sent to the Eurotherm [NB "No Comm on comm2" simply refers to the fact that there is no communication to the Eurotherm.

```
21,12,1997,22,18,39,admit,1.2,No Comm on comm2,  
21,12,1997,22,20,21,temp1, 1.565,_0011_SL 1.565__,  
21,12,1997,22,20,23,wait,4,No Comm on comm2,  
21,12,1997,22,24,23,imped,1.2,No Comm on comm2,
```

3.4.3 The .dat file

An example of a .dat file is listed below. These files have the following columns, each separated by a comma.

- The frequency of the measurement
- The real component of the measurement
- The imaginary component of the measurement

```
+1.0000000E-01,+2.9719E+04,-1.7561E+03  
+4.7639000E-01,+2.8919E+04,-1.4161E+03  
+2.2695000E+00,+2.7774E+04,-1.0895E+03  
+1.0811700E+01,+2.6886E+04,-8.1447E+02
```

```
+5.1506100E+01,+2.5906E+04,-2.2975E+03
+2.4537107E+02,+2.5813E+04,-4.7303E+02
+1.1689287E+03,+2.5495E+04,-4.1830E+02
+5.5686855E+03,+2.5227E+04,-6.4170E+02
+2.6528785E+04,+2.4743E+04,-1.8501E+03
```

3.4.4 The journal.txt file

An example of the journal.txt file is listed below. It has the following columns, each separated by a comma.

- The unique number assigned to the sweep, called the "sweep number"
- The title followed by "="
- The date in format dd-mm-yyyy followed by "="
- The time in format hh:mm:ss followed by "="
- "Temp of top (2)" followed by "=". This indicates the reading of the Eurotherm with address 0.2. This is standardly the top Eurotherm for the vacuum furnace and the Hiphit cell and hence is known as "temp of top".
- The temperature of the thermocouple in the format xxxx.xx followed by "=", where "x" refers to a digit from 0 to 9.
- "Temp of bottom (1)" followed by "=". This indicates the reading of the Eurotherm with address 0.1. This is standardly the bottom Eurotherm for the vacuum furnace and the Hiphit cell and hence is known as "temp of bottom".
- The temperature of the thermocouple in the format xxxx.xx followed by "="
- "T3" followed by "=". This indicates the reading of the Eurotherm with address 0.3
- The temperature of the thermocouple in the format xxxx.xx

An example file is listed below:

```
0010226   CuI   0.66t   =01-30-1998   =06:25:08=Temp   of   Top(2)=0347.80=Temp   of   bottom(1)=0420.50=T3=0001.27
0010227   CuI   0.66t   =01-30-1998   =06:32:36=Temp   of   Top(2)=0352.40=Temp   of   bottom(1)=0433.40=T3=0001.27
0010228   CuI   0.66t   =01-30-1998   =06:40:08=Temp   of   Top(2)=0350.70=Temp   of   bottom(1)=0423.70=T3=0001.28
0010229   CuI   0.66t   =01-30-1998   =06:47:40=Temp   of   Top(2)=0355.70=Temp   of   bottom(1)=0429.70=T3=0001.28
0010230   CuI   0.66t   =01-30-1998   =06:55:12=Temp   of   Top(2)=0359.00=Temp   of   bottom(1)=0442.30=T3=0001.28
```


0010231	CuI	0.66t	=01-30-1998	=07:02:43=Temp	of	Top(2)=0359.40=Temp	of	bottom(1)=0437.20=T3=0001.28
0010232	CuI	0.66t	=01-30-1998	=07:10:16=Temp	of	Top(2)=0361.60=Temp	of	bottom(1)=0439.90=T3=0001.28
0010233	CuI	0.66t	=01-30-1998	=07:17:47=Temp	of	Top(2)=0368.90=Temp	of	bottom(1)=0444.90=T3=0001.29
0010234	CuI	0.66t	=01-30-1998	=07:25:16=Temp	of	Top(2)=0372.70=Temp	of	bottom(1)=0456.30=T3=0001.28
0010235	CuI	0.66t	=01-30-1998	=07:32:43=Temp	of	Top(2)=0368.20=Temp	of	bottom(1)=0441.60=T3=0001.29

3.

4. Data File Transfer

Immfit has been written in fortran, so the data and log files need to be transferred onto a unix or vax machine with an available fortran compiler. To transfer the files, the following procedure should be observed:

- *Ftp them using win_ftp in ASCII mode.*
- *Double click on the "win_ftp" icon.*
- *Connect to the machine with IP Address isisa.nd.rl.ac.uk, using your username and password.*
- *Select the local and the destination directory and also ASCII mode for file transfer. (The local destination directory is the one on your PC containing the conductivity data e.g. "c:\tube furnace\)", whereas the remote directory will be one with the form e.g. "scratch\$disk:[username.conductivity]", where "username" is replaced by the name of your user account at ISIS).*
- *Select the files to be transferred*
- *Click send.*

Running Immfit

First connect to an alpha computer on the VMS/VAX cluster at ISIS. Then the current directory must be changed to the directory into which you put the files. Use the command "set def" to change the default directory to the directory where the data files have been stored, as shown below:

```
set def scratch$disk:[username.conductivity]
```

All files listed here for data analysis are located in the directory user\$disk:[njgg.datafit.alpha]. They can be run on any alpha machine (most commonly "horus.nd.rl.ac.uk") by typing:

```
r user$disk:[njgg.datafit.alpha]"program name"
```

at the command prompt, where 'program name' in this instance refers to the program Immfit.

1. Immfit Options

When Immfit is run, the program will ask you to respond to a series of questions, defining the scan, fitting routine and the data input and output and then it will produce a fit to each data set, recording this in several output files. The questions asked are as follows:

- *"Enter output device ('xwindow' or 'file')". If you are working from a PC or VAX/Alpha computer at the Rutherford, then specify 'xwindow' to view the data output on screen or 'file' to save the output in Hewlett Packard Graphic Language. If you are using the program away from the Rutherford, then you will not be able to view the fit on screen, so type 'file'.*
- *"Confirm each fit individually ('y') or automatic fit ('n')". This gives you the option of specifying whether the fit is acceptable and if it is not acceptable, then the computer will attempt to determine a better fit.*
- *"Enter 'imped' for impedance or 'admit' for admittance". This allows you to specify the type of data to be analysed.*
- *"Enter name for output files". Specify the body (not including an extension for the output files) e.g. "Cool" gives output files "cool.out", "cool.par", "cool.pow" and "cool.tem".*
- *"Enter first and last run numbers". Enter the integral numbers of the run numbers to be analysed, making sure that all the data and log files required exist in the subdirectory from which the program is run.*
- *"Enter no of first and last data for display". Enter the number of the first and last data points from the file to be displayed on the graph (10 and 50 are good suggested values if the data file has 60 lines, over frequencies 0.1 Hz to 10 MHz).*
- *"Enter no of first and last points to scan initially". Enter the line numbers of the data file, over which Immfit performs its initial scan to determine the minimum (15 and 45 are good suggested values).*
- *"Enter no of points to fit to each line". Enter the line numbers of the data file, which are to be used for the fitting of each line (7 is a good suggested value).*
- *"Determine null measurement? ('y' or 'n')". Data can be normalised to try to reduce the effect of the measuring circuit, using a "short circuit" and an "open circuit" measurement of the impedance. The normalised impedance, Z_u is*

$$Z_u = \frac{1}{\left(\frac{1}{Z_m - Z_{sc}} \right) - \frac{1}{Z_{oc}}},$$

where Z_m is the measured impedance, Z_{sc} is the short circuit impedance and Z_{oc} is the open circuit impedance.

- If you respond "y" to this question then a further question is asked:
 - "Enter number of open circuit sweep and of short circuit sweep respectively". Give the two integral values of the relevant run numbers.

1. Immfit Operation

Each time the program fits the data, a plot is displayed on the screen. If this plot is a good estimate of the immittance, then press any key, other than Y and r. If this plot is not a good fit, then enter Y and r to reject the plot. The program then tries to fit these data over a different range of points. Keep entering Y and r until there is a good fit to the data. The program fits each run number, storing the results in the memory. When all the runs have been analysed, it writes a series of output files, which can be read into any spreadsheet or graphics package, such as "Sigmaplot".

2. Immfit Output files

1. The .out file

This file contains the basic data related to the immittance of the sample in five columns. The first is the run number, the second is the Temperature of Eurotherm (address 0.1), the third is the temperature of Eurotherm (address 0.2), the fourth is the calculated immittance value and the fifth is the number of the general minimum (if the data set had one).

An example of the file is listed below:

```
10244 421.6000 351.1000 1.868192 0.0000000E+00
10245 416.3000 348.1000 1.832837 0.0000000E+00
10246 408.6000 343.0000 1.795129 0.0000000E+00
10247 410.1000 343.7000 2.008008 0.0000000E+00
10248 409.5000 342.2000 2.098195 0.0000000E+00
10249 406.3000 343.2000 2.018496 0.0000000E+00
10250 400.8000 338.7000 2.134143 0.0000000E+00
10251 400.7000 337.4000 2.078669 0.0000000E+00
```

```

10252 396.1000 335.2000 2.082982 0.0000000E+00
10253 394.2000 335.9000 2.094032 0.0000000E+00
10254 390.4000 333.7000 2.081607 0.0000000E+00

```

2. The .par file

This file contains the parameters obtained from the linear fitting of the data in five columns. The first is the run number, the following columns are a,b,c,d respectively, where the two fitted lines have the equations:

$$Z_{\text{imaginary}} = a \times Z_{\text{real}} + b$$

$$\text{and } Z_{\text{imaginary}} = c \times Z_{\text{real}} + d$$

An example of the file is listed below:

```

10244 1.974536 -1.214651 1.594424 -1.011186
10245 4.065326 -2.439621 1.091273 -0.8169715
10246 35.22023 -19.75714 2.334585 -1.437758
10247 3.015028 -1.746179 1.441210 -0.9624082
10248 2.636169 -1.508225 1.678332 -1.051720
10249 2.127173 -1.245788 1.670854 -1.019719
10250 2.942449 -1.635880 1.695812 -1.051741
10251 2.981171 -1.684323 1.414261 -0.9305186
10252 3.016654 -1.687842 1.481656 -0.9509183

```

3. The .pow file

On occasions the .tem (see next section) file may indicate that the temperature failed to control properly. At this stage it is important to determine whether this was caused by the Eurotherm's inability to control the sample environment with the set proportional band, integral and derivative times (PIDs), or whether the problem was that the Eurotherm had insufficient power to reach the sample temperature. The .pow file enables you to determine the power output from each Eurotherm as a function of run number. This file contains four columns, giving the power output of each Eurotherm. The first column is the run number, with the following columns being the percentage power output of the Eurotherm with address 0.1, 0.2 and 0.3 respectively.

An example of the file is listed below:

```

10244 72.20000 0.0000000E+00 50.00000
10245 64.00000 0.0000000E+00 50.00000

```

```

10246 49.00000 0.0000000E+00 50.00000
10247 54.90000 0.0000000E+00 50.00000
10248 63.10000 0.0000000E+00 50.00000
10249 51.50000 0.0000000E+00 50.00000
10250 51.00000 0.0000000E+00 50.00000
10251 61.90000 0.0000000E+00 50.00000
10252 60.30000 0.0000000E+00 50.00000
10253 56.10000 0.0000000E+00 50.00000
10254 58.00000 0.0000000E+00 50.00000

```

4. The .tem file

This file contains the temperature measured by each Eurotherm as a function of time. The first column is the run number, with the following columns being the time in hours, minutes and seconds, respectively, and then the temperature of the Eurotherm with address 0.1, 0.2 and 0.3 respectively.

An example of the file is listed below:

```

10244 8 39 32 0.4216000E+03 0.3511000E+03 0.1260000E+01
10245 8 47 0 0.4163000E+03 0.3481000E+03 0.1260000E+01
10246 8 54 29 0.4086000E+03 0.3430000E+03 0.1210000E+01
10247 9 2 5 0.4101000E+03 0.3437000E+03 0.1200000E+01
10248 9 9 40 0.4095000E+03 0.3422000E+03 0.1210000E+01
10249 9 17 9 0.4063000E+03 0.3432000E+03 0.1200000E+01
10250 9 24 49 0.4008000E+03 0.3387000E+03 0.1210000E+01

```

3. Importing output files into SigmaPlot

Once the output files have been produced, they can be viewed simply by any graphics package. Use "win_ftp" or some other version of ftp to transfer the output files from the VAX/VMS cluster to the PC. To load the data into SigmaPlot, the "importing" procedure should be followed as described in section 4.1 "Data Manipulation".

It is possible to use transforms (by pressing "F10", when in the SigmaPlot data worksheet view) to transform the data into conductivity values or to find the average temperature etc. An example of a transform for this purpose is listed and described below. Thus this provides a quick method to view the data and determine the behaviour of the sample.

Example of Transform File (cond.xfm)

col(4)=0.29/((3.141592657*0.2*0.2)*col(2))

$$\text{col}(5)=1000/(\text{col}(3)+273)$$

$$\text{col}(6)=\log(\text{col}(4))$$

Column 2 contains the measured sample resistance and column 3 contains the sample temperature in °C and the pellet's dimensions are 2.9mm (length) and 4.0mm (diameter). This transform produces columns 4,5,6. Column 4 contains the conductivity of the sample, in units of $(\Omega\text{cm})^{-1}$. Column 5 gives $(1000/T)$, where the temperature, T, is measured in Kelvin. Column 6 gives the log of the conductivity.

2. Equivcrt program

Equivcrt is an immittance data analysis system for IBM-PC computers and compatibles written in Turbo Pascal version 3.0. The program will fit immittance data using an equivalent circuit and a series of non linear least squares fitting routines. A "data cruncher" function greatly simplifies the process of choosing an equivalent circuit to fit to the data. A copy of this program is available for use at the Rutherford.

However, before the data files from Solcon can be used in *Equivcrt*, they need to be converted from their comma delineated format into space delineated columns. The fortran program *convert.exe* (located in the directory `user$disk:[njgg.datafit.alpha]`) performs this operation, changing comma delineated admittance or impedance (`a/sxxxxxxx.dat`) files into space delineated `qxxxxxxx.dat` files. When this program is run, it asks for the run numbers to be converted and the type of data to be entered (admittance or impedance), in the same way as *Immfit.exe* does. The `qxxxxxxx.dat` files can be loaded directly into *equivcrt*, with the options in *equivcrt* set such that it does not look for a header in the loaded file. For further details, consult the *equivcrt* manual.

5. Archiving, backup and file retrieving systems

1. Outline of archive operation

Files from the computers running the experiment frequently have to be deleted to ensure that there is space for files from the next experiment. They are backed up onto two different areas (Figure 23): they are both stored centrally on a fileserver at the Rutherford Laboratory and also "zipped" up and placed onto a zip disk. The Iomega zip disk drive provides a permanent archive for data and the fileserver provides instant access for old data files. The fileserver can be accessed from any PC on the Rutherford site, and also for access via a modem from any PC, but security prevents these files from being available via LANs at university. Files, which are required for use

elsewhere, should be copied either onto floppy disks or onto Iomega zip disks.

2. Directory and file server ved.

1. Copying files onto Windows NT server and local hard drive

Files are copied using Windows NT explorer from the PC in the Polaris Cabin, or any other PC connected to the POL001 or POL002 conductivity measuring computers. A table of example directory names is shown below in Table 5: