# EXPERIMENT XII

**LOW TEMPERATURE HEAT CAPACITY** (S&G 2nd ed. Experiment 47)

## 1. Purpose

The heat capacity of aluminium (or copper, check with instructor) will be determined at three different temperatures in the range ≈ 80 K (liquid nitrogen) to room temperature (≈ 300 K). The results will be interpreted in terms of the Debye theory of specific heats.

## 2. Pre-lab preparation

The full description of this experiment has disappeared from the most recent edition of S&G. Copies of older editions of S&G should be in the reserve section of the Library, or a copy of the corresponding chapter may be obtained from the instructor or a photocopy downloaded from the course web site.

Before starting this experiment, make sure that you understand the principle of operation of an oil diffusion pump and how the vacuum line shown in Fig. 3 is to be handled.

*There is a chapter in S&G devoted to vacuum lines and vacuum techniques; you are strongly encouraged to read it before doing this experiment.*

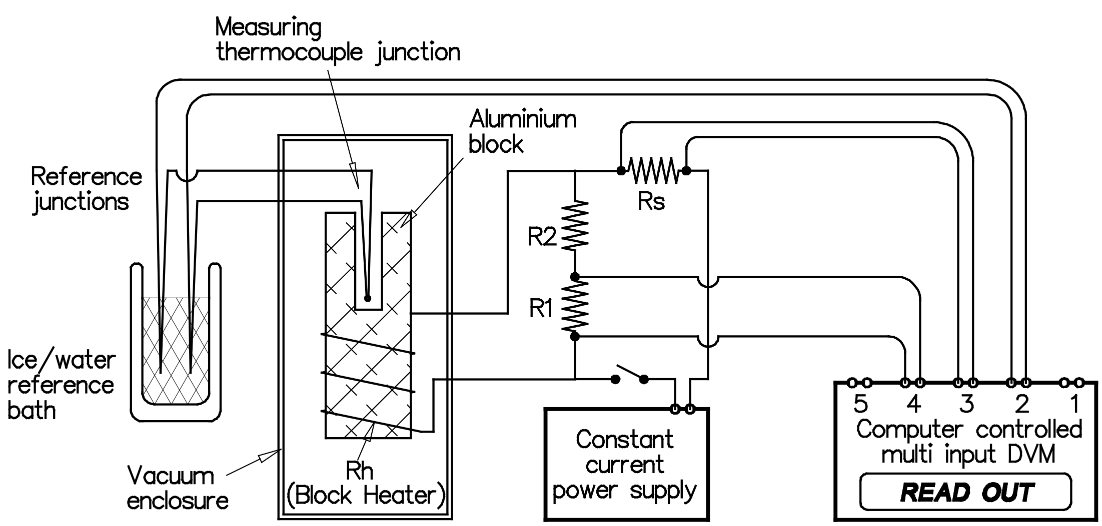


Figure 1. Schematic diagram of the Low Temperature Heat Capacity Experiment set-up.

## 3. Safety

Wear eye protection at all times, and use thick protecting gloves when handling dry ice or pouring liquid nitrogen. When preparing the dry-ice/acetone bath, at the beginning add only small pieces of dry-ice to control the foaming and prevent spillage of acetone.

## 4. Set-up

The potentiometer set-up described in the text book is not used in your experimental arrangement. It has been replaced by a precision DVM (digital voltmeter) or, if available, a computer controlled scanning DVM.[1] The instructor will brief you on how to collect the data with these instruments. Fig. 1 shows a sketch of the set-up (somewhat different from the diagram shown in S&G) and Fig. 2 shows the heating circuit diagram.

Measure the heat capacity of the assigned metal at room temperature, at dry-ice temperature and at liquid nitrogen temperature. The temperature is monitored with a type K thermocouple (chromel-alumel), for which the EMF–temperature calibration data can be found in Table 1.

Before starting, verify

1) that the reference junction is kept at 0°C in a dewar of ice-water

2) that the constant current power supply is ON (*NJE* Model QR36-4),

3) that the double-pole double-throw switch DPDT-1 is in the middle position, and

4) that the block heater (resistance $R_h$ – see Fig. 2) is actually connected (the lower large double-pole double-throw switch – DPDT2 – should be pointing towards the vacuum line).

5) that the cold trap (see Fig. 3) is filled with liquid nitrogen at all times; it may need to be topped up regularly.

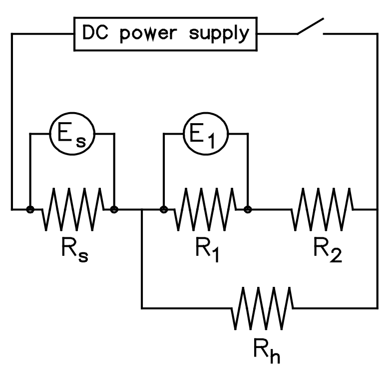


Figure 2. Diagram of the heating circuit used in the Low Temperature Heat Capacity Experiment.

[1] Note: the computer attached to the DVM runs on an old operating system DOS/Windows 3.1 which allows for file name a maximum of only 8 characters (no spaces) + three character extension. Example: "NICEFILE.366".

As indicated in the write-up, first the temperature is monitored for ≈ 5 min before heating is applied to get a baseline corresponding to the variation of the temperature before anything is done to the sample, then the sample is heated until a temperature rise of approximately 3 - 5°C is observed (however do not heat for longer than ≈ 10-12 min, even if the temperature does not seem to have changed by the expected amount). After having stopped the heating, keep monitoring the temperature for at least another 10 minutes. Setting the DVM to collect data every 10 s is quite sufficient.

The necessary apparatus parameters are given below:

- Resistance of heater ≡ $R_h$ = 195.22 ± .02 Ω
- Resistance of leads ≡ $R_L$
- $R_h + R_L$ = 208.38 ± .02 Ω
- $R_S$ = 10.008 ± 0.002 Ω
- Mass of Al cylinder 97.364 ± 0.001 g
- The values of $R_1$ and $R_2$ are to be recorded from the actual set-up used for the experiment. *Do not forget to record this information!*

## 5. Vacuum line operation

A diagram of the vacuum system used here is shown in Fig. 3. When you come in, the system should be in a vacuum state, *ie*, pressure gauge should read ≈ −90 kPa, and the vacuum gauge should read < $10^{-4}$ mm Hg.

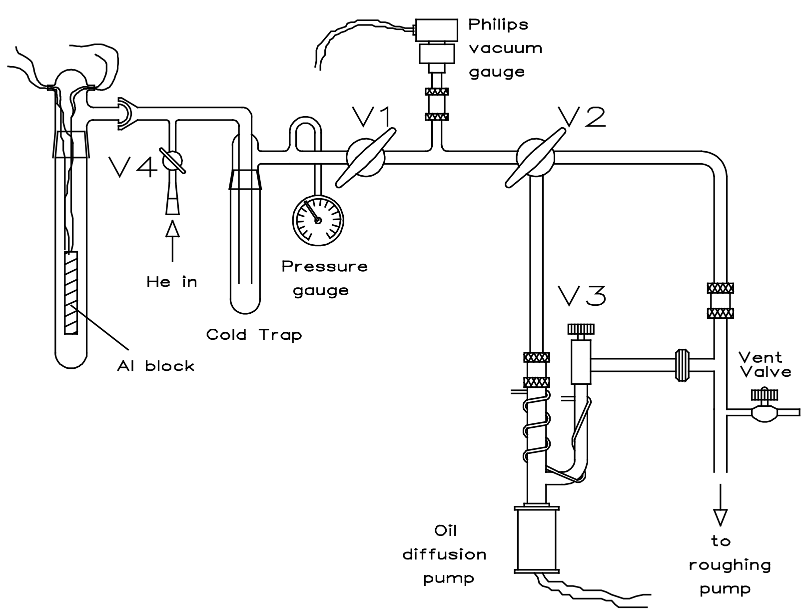


Figure 3. Diagram of vacuum line used in Low Temperature Heat Capacity Experiment

### 6. Changing temperature.

For the temperature of the aluminium block to change in a reasonable length of time, a convenient heat transfer agent (He gas in the present instance) must be introduced into the vacuum space around the aluminium block. To accomplish this, the following manipulations should be performed:

- Close V1 (This valve may be stiff; do NOT force when turning the valve, just turn slowly; if necessary warm up the barrel with a heat gun to soften the grease).
- Admit slowly He gas by opening gently V4 until the pressure gauge reads $\approx -10$ kPa (relative to ambient pressure). Do not exceed 2 psi relative pressure or one of the stopcock key might pop out.
- Let the temperature of the metal block equilibrate with that of the surrounding bath (within 5 °C); this may take some 20 minutes.

*Before starting a new run, the line must be evacuated.*

- Open slowly V1; the gurgling noise of the mechanical pump will be heard.
- Before starting a new run, wait for ≈ 5 min for the pressure to drop below $10^{-4}$ mm Hg as indicated by the Philips vacuum gauge.

Do *not* switch anything off at the end of the experiment; vacuum lines operate best when left pumping all the time, but *do* shut *off* the tank valve of the helium cylinder.

### 7. Data analysis and discussion

The write-up suggests to obtain graphically ΔT at the mid-point of the heating period. Better results (explain why) are obtained by measuring ΔT on the heating curve at the time at which the shaded areas in Fig. 4 are equal.

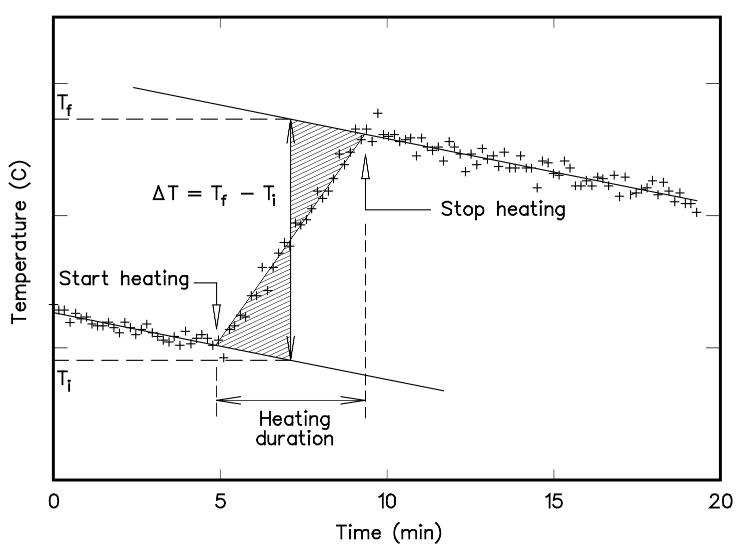


Figure 4. Method used to obtain ΔT from heating curve; ΔT is measured at such time where the areas of the two triangular shaded are equal.

There is an EXCEL file, `LOWTANAL.XLS`, designed to help in the determination of $v_i$ and $v_f$, the emf values of the thermocouple from which $T_i$ and $T_f$ (hence $\Delta T$) can be obtained using Table 1. The file is available from some of the PCHEM computers.* During analysis, pay attention to the error in determining the temperature change $\Delta T$; for this matter, the EXCEL template provides the slope and the intercept (and corresponding errors) of the pre- and post-heating lines. Use this information to estimate the error on $\Delta T$. For theoretical calculations, tabulated values along with the corresponding plot of the Debye function can be found in the EXCEL file `DEBYEINT.XLS`*. Present your heat capacity results plotted on this Debye curve.

*Note. If your data were collected with the computer, be aware that even when no voltage is present the DVM still records some small random value (floating voltage) which has no meaning and should be read as 0.*

In your discussion, explain why the temperature is monitored for some time, before and after the heating period; comment on the slopes of the pre and post-heating curves; can you rationalize the trends observed? How is this related to the quality of the vacuum in the experimental set-up? The manual recommends that for each run the temperature change $\Delta T$ be ≈ 3 to 5 C; why not a much smaller temperature change, or much bigger? What would be the advantages or drawbacks in each case?. Why is it important to fill the line with helium gas when changing the temperature of the sample, and why is it important to pump out this helium gas before starting a new run?

In the result section of your report, include a table of your results in the following format and provide this same information on a diskette or as an email to the instructor (Excel template may be found in the course web site).

| | Run | | | | | |
|---|---|---|---|---|---|---|
| | Room Temp. | error | Dry Ice/ acetone | error | Liquid Nitrogen | error |
| Data Filename | … | - | … | - | … | - |
| $E_S$/V | … | … | … | … | … | … |
| $E_1$/V | … | … | … | … | … | … |
| Heating duration/s | … | … | … | … | … | … |
| Emf initial/mV | … | … | … | … | … | … |
| Emf final/mV | … | … | … | … | … | … |
| $T_i$/K | … | … | … | … | … | … |
| $T_f$/K | … | … | … | … | … | … |
| $\Delta T$/C | … | … | … | … | … | … |
| <T>/K | … | … | … | … | … | … |

*Can be downloaded from the course web site

## 8. Supplemental material

Excerpt of calibration table for type K thermocouple (Table 1). A table and a plot of the Debye integral are available in the laboratory or from the course web site.

Table 1. Chromel-alumel thermocouple (type K): selected values of mV versus temperature (reference junction at 0 ºC)[a]

| t/ºC | 0 | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 |
|---|---|---|---|---|---|---|---|---|---|---|
| **-200** | -5.891 | -5.907 | -5.922 | -5.936 | -5.951 | -5.965 | -5.980 | -5.994 | -6.007 | -6.021 |
| **-190** | -5.730 | -5.747 | -5.763 | -5.780 | -5.796 | -5.813 | -5.829 | -5.845 | -5.860 | -5.876 |
| **-180** | -5.550 | -5.569 | -5.587 | -5.606 | -5.624 | -5.642 | -5.660 | -5.678 | -5.695 | -5.712 |
| **-170** | -5.354 | -5.374 | -5.394 | -5.414 | -5.434 | -5.454 | -5.474 | -5.493 | -5.512 | -5.531 |
| **-160** | -5.141 | -5.163 | -5.185 | -5.207 | -5.228 | -5.250 | -5.271 | -5.292 | -5.313 | -5.333 |
| | | | | | | | | | | |
| **-80** | -2.920 | -2.953 | -2.985 | -3.018 | -3.050 | -3.082 | -3.115 | -3.147 | -3.179 | -3.211 |
| **-70** | -2.586 | -2.620 | -2.654 | -2.687 | -2.721 | -2.754 | -2.788 | -2.821 | -2.854 | -2.887 |
| **-60** | -2.243 | -2.277 | -2.312 | -2.347 | -2.381 | -2.416 | -2.450 | -2.484 | -2.518 | -2.552 |
| **-50** | -1.889 | -1.925 | -1.961 | -1.996 | -2.032 | -2.067 | -2.102 | -2.137 | -2.173 | -2.208 |
| **-40** | -1.527 | -1.564 | -1.600 | -1.637 | -1.673 | -1.709 | -1.745 | -1.782 | -1.818 | -1.854 |
| | | | | | | | | | | |
| **0** | 0.000 | 0.039 | 0.079 | 0.119 | 0.158 | 0.198 | 0.238 | 0.277 | 0.317 | 0.357 |
| **10** | 0.397 | 0.437 | 0.477 | 0.517 | 0.557 | 0.597 | 0.637 | 0.677 | 0.718 | 0.758 |
| **20** | 0.798 | 0.838 | 0.879 | 0.919 | 0.960 | 1.000 | 1.041 | 1.081 | 1.122 | 1.162 |
| **30** | 1.203 | 1.244 | 1.285 | 1.325 | 1.366 | 1.407 | 1.448 | 1.489 | 1.529 | 1.570 |
| **40** | 1.611 | 1.652 | 1.693 | 1.734 | 1.776 | 1.817 | 1.858 | 1.899 | 1.940 | 1.981 |

a) The table lists mV value for every Celsius; perform a linear interpolation to obtain fractional Celsius.

## 9. Linear interpolation.

The problem is to find a temperature $t_x$ corresponding to a certain measurement $mv_x$ (mV), the exact value of which is not listed in the table.

1) Find in Table 1 the two consecutive closest voltage values $mv_1$ and $mv_2$ which are just below and just above $mv_x$:, and read the corresponding temperatures $t_1$ and $t_2$. We have:

$mv_1 < mv_x < mv_2$ and therefore $t_1 < t_x < t_2$

2) The linear interpolation assumes that between the small interval $t_1$ and $t_2$ the voltage varies linearly, *i.e.*

$$\frac{\Delta t}{\Delta mv} = \text{constant} = \frac{(t_x - t_1)}{(mv_x - mv_1)} = \frac{(t_x - t_2)}{(mv_x - mv_2)} = \frac{(t_2 - t_1)}{(mv_2 - mv_1)}$$

One can find $t_x$ by re-arranging the above equations:

$$t_x = t_1 + \frac{(mv_x - mv_1)}{(mv_2 - mv_1)}(t_2 - t_1) \quad \text{or} \quad t_x = t_2 + \frac{(mv_x - mv_2)}{(mv_2 - mv_1)}(t_2 - t_1)$$

Chem 366W report check list

A report will not be accepted without all the items of this list checked. If a checked item is found missing in the report, the report will be automatically down-graded.

Student Name:_______________________________________________________

**Report: Low Temperature Heat Capacity**

**Title page.**

Correct title of the experiment ........................................................................ ☐
Student Name & student ID ........................................................................ ☐
Partner name (*if applicable*)_____________________________________________
Date of performance of experiment ........................................................................ ☐
**Abstract**........................................................................................ ☐
**Introduction and theory** ........................................................................ ☐
**Experimental**
Changes from text description mentioned (*if applicable*)...................................... ☐
Sample ID, ser no, stock solution …etc recorded (*if applicable*) .......................... ☐
**Results**
Results as Tables ........................................................................................ ☐
Graphs
Size, at least ½ page ........................................................................................ ☐
Axis labelled ........................................................................................ ☐
Axis labels have units ........................................................................................ ☐
Axis scales are sensible ........................................................................................ ☐
Only significant figures ........................................................................................ ☐
Uncertainties quoted ........................................................................................ ☐
Raw data provided (*electronic form, if applicable*) ............................................. ☐
**Calculations**
Sample calculation provided ........................................................................ ☐
Error analysis ........................................................................................ ☐
Sample error calculation provided ........................................................................ ☐
**Discussion**
Comments on results ........................................................................................ ☐
Questions in text book and in manual answered .................................................. ☐
Comparison with literature value(s) ........................................................................ ☐
**Conclusion**........................................................................................ ☐

Blank Page