DETERMINATION OF THE DENSITY OF CRYSTALLINE GERMANIUM

Maria Smith
(Partner: John Klein)

Sept. 25, 2008

Abstract

A pycnometer has been used to determine the density of two samples of germanium at 25°C. The values obtained, with their 95% confidence limits, are ρ(sample I) = 5.310 ± 0.003 g cm^{-3} and ρ(sample II) = 5.332 ± 0.003 g cm^{-3}. Both samples consisted of several pieces of crystalline material, with the pieces in sample I being larger and more irregular. It seems likely that sample I had hidden defects and voids that introduced a systematic error since the x-ray value of the density is 5.327 ± 0.002 g cm^{-3}. Although the density of sample II is slightly high, it does agree with the x-ray value just within the sum of the cited uncertainties.

TITLE PAGE AND ABSTRACT

The front page of the report should display a title, your name, the name of any experimental partners, the date on which the report is submitted, and a brief abstract. An abstract is typically 50 to 100 words long; the example above contains about 90 words. It should summarize the results of the experiment and state any significant conclusions. *Numerical results with confidence limits should be included.*

I. Introduction

The purpose of this experiment is to measure the density of crystals of germanium. Since the density ρ is defined by

$$\rho = W_S/V_S, \qquad (1)$$

it is desired to measure the volume V_S occupied by a known weight W_S of the metal sample.

The method involves the use of a pycnometer of known volume which is first weighed empty, then weighed containing the solid sample to be studied. The difference gives the weight of the solid, W_S. Finally the pycnometer (containing the solid sample) is filled with a liquid of known density and reweighed; the weight, and therefore the volume, of the liquid can be found by difference. Since the total volume of the pycnometer is known, one can then calculate the volume V_S which is occupied by the solid.

Theory needs to be added in this section.

II. Experimental Method

The experimental method was similar to that described in the textbook (Aardvark and Zebra, 3rd ed., Exp. 13). The design of the pycnometer used, which differs from that described in the textbook, is shown in Fig. 1.

INTRODUCTION

The introduction should state the purpose of the experiment and give a *very brief* outline of the necessary theory, which is often accomplished by citing pertinent equations with references wherever appropriate. Each equation should appear on a separate line and should be part of a complete sentence. Number all equations consecutively throughout the entire report, and refer to them by number. All symbols should be clearly identified the first time they appear. A very short description of the experimental method can also be included. Use the present tense throughout this section.

The introduction should cover the above topics as concisely as possible; the sample contains about 130 words. More complicated experiments will require longer introductions, but the normal length should be between 100 and 300 words. In the case of a journal article, the introduction is often much longer, since pertinent recent work in the field is cited and briefly summarized.

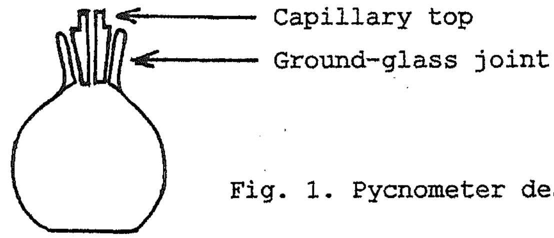

Fig. 1. Pycnometer design.

The procedure was modified as follows: After distilled water had been added to the pycnometer containing the sample, the pycnometer (with capillary top removed) was completely immersed in a flask of distilled water and boiled under low pressure for 15 minutes to remove air trapped by the solid or dissolved in the water [1]. After this boiling, the pycnometer was equilibrated for 30 min in a 25°C thermostat bath before the top was inserted.

The weight of the solid sample is given by

$$W_S = W_2 - W_1, \tag{2}$$

where W_1 is the weight of the empty pycnometer and W_2 is that of the pycnometer plus solid sample. The weight of water contained in the pycnometer, W_L, is

EXPERIMENTAL METHOD

The section on experimental method is usually *extremely brief* and merely cites the appropriate references that describe the details of the experimental procedure. If reference is made to the textbook and/or laboratory notes assigned for the course, an abbreviated title may be cited in lieu of a complete bibliographic entry. Any reference to other books or materials should be assigned footnote numbers and should be properly listed at the end of the experiment in the form illustrated by the references in this book. Description of experimental procedures should normally be given *only* for those features not described in or differing from the cited references. A simple sketch of apparatus is appropriate only when it differs from that described in the references. A summary statement or condensed derivation of the phenomenological equations used to analyze the raw data should be given. Except for text concerning such equations, this section should be written in the past tense and follow a chronological order. NOTE: A statement of the number of runs made and

$$W_L = W_3 - W_2, \qquad (3)$$

where W_3 is the weight of the pycnometer plus sample plus water. If the density of the liquid water is denoted by ρ_L, it follows from Eq. (3) that the volume of the solid sample is given by

$$V_S = V - V_L = (\rho_L V + W_2 - W_3)/\rho_L, \qquad (4)$$

where V is the total volume of the pycnometer. From Eqs. (1), (2), and (4), we obtain

$$\rho = W_S/V_S = \rho_L(W_2 - W_1)/(\rho_L V + W_2 - W_3). \qquad (5)$$

Since the values of V and ρ_L are known, it is only necessary to determine W_1, W_2, and W_3 in order to calculate the density of the solid.

Two duplicate runs, carried out using the same procedure, were made on each of two different germanium samples. Sample I consisted of larger and somewhat more irregular pieces than did Sample II.

the conditions under which they were carried out (concentration, temperature, etc.) should always be included at the end of this section.

RESULTS

The section on results should present the experimental results in full detail, making use of tables and figures where appropriate. No result should be excluded merely because it is unexpected or inconsistent with other data or theoretical models. The cause for discrepancies, if known, can be pointed out in the Discussion section. Primary measurements ("raw data") should be given, as well as derived quantities. It is essential that the units be completely specified.

It is undesirable to present detailed computations in the main body of the report; however, a typical sample calculation should be given in an appendix to illustrate how the data

+ Tables + Figures

III. Results

The average values of the measured weights W_1, W_2, and W_3 are listed below together with the stated value of the pycnometer volume V. The literature value for the density of water ρ_L is 0.99705 g cm^{-3} at 25°C (taken from the Handbook of Chemistry and Physics[2]).

$V = 12.445 \pm 0.003$ cm^3 (given by instructor)

$W_1 = 8.6309$ g

Sample I: $W_2 = 42.0301$ g, $W_3 = 48.1732$ g

Sample II: $W_2 = 45.8479$ g, $W_3 = 51.3036$ g

The density of germanium can now be calculated by substitution of the above data into Eq. (5). The resulting densities are 5.315 g cm^{-3} for sample I and 5.337 g cm^{-3} for sample II. The weights used in these calculations have not been corrected for the effect of air buoyancy. Rather than correct each weight, we have used a simple formula given by Bauer and Lewin[1] for correcting the final result. This formula for the corrected density ρ^* is

$$\rho^* = \rho + 0.0012[1 - (\rho/\rho_L)]. \qquad (6)$$

analysis was carried out. If specialized computer programs have been used in processing the data, they should be cited in the Results section. If a program is not well known and documented elsewhere, provide a complete listing of the program in an appendix.

Tables should be numbered and given self-explanatory captions. The quantities displayed in a table should be clearly labeled with the units specified. Integrate the tables into the text so that the information presented flows smoothly for the reader.

Many reports will require a graphical presentation of data or calculated results. Each figure should be numbered and given a legend. Figures serve several purposes: to supplement or replace tables as a means of presenting results; to display relationships between two or more quantities; to find values needed in the calculation of results (see Chapter IIA). Some advantages of graphical display are that the relationship between two variables is shown more clearly by figures than by tables; deviations of individual results from expected trends are more readily apparent; "smoothing" of the data can be done better if it is necessary and appropriate; interpolation and extrapolation are easier. A disadvantage

When Eq. (6) is applied to our results, we obtain for ρ^* the following values:

$$\begin{aligned} \text{Sample I: } & 5.310 \text{ g cm}^{-3} \\ \text{Sample II: } & 5.332 \text{ g cm}^{-3} \\ \text{Average: } & 5.321 \text{ g cm}^{-3} \end{aligned} \qquad (7)$$

According to Eq. (5) the uncertainty in ρ will depend on the uncertainty in each of five variables; however, the value of ρ_L is known to five significant figures and its uncertainty may be neglected in comparison to those in the other variables. We may take as reasonable 95% confidence limits for the weighings $\Delta W_1 = \Delta W_2 = 0.001$ g and $\Delta W_3 = 0.002$ g. The higher value for ΔW_3 includes the possible failure to attain an exact filling of the pycnometer with water. For ΔV we take 0.003 cm^3, the value given by the instructor, although such a value seems rather high. On the basis of these uncertainty values, it is clear that the major contributions to the limit of error $\Delta\rho$ are the uncertainty in V and, to a lesser extent, the uncertainty in the difference $W_2 - W_3$. The contribution to $\Delta\rho$ due to the uncertainty in the difference $W_2 - W_1$ is much less (since $W_2 - W_1$ is about 5.5 times larger than $W_2 - W_3$ and the uncertainty in $W_2 - W_1$ is less than that in $W_2 - W_3$), and it can be neglected in obtaining a good approximation for the

of figures is that graphical displays cannot always show the full precision of the results. For a report, it is normally advisable to include both figures and tables. Some advice on the preparation of figures is given on pp. 34–36. Indicate clearly the scales and units used. Error bars should be included when the uncertainties are greater than the size of the symbols enclosing the points.

An *error analysis* dealing with the uncertainty in the final result due to random errors in the measurements will normally be part of the Results section. The type of error analysis undertaken will depend a great deal on the nature of the experiment; see Chapters IIB and XXI for more details. The analysis given in the sample report is typical of a straightforward propagation-of-errors treatment. If a long and complex propagation-of-errors value of $\Delta\rho$. With this simplification, the 95% confidence limit in ρ is given by

$$(\Delta\rho)^2/\rho^2 \approx [\rho_L^2(\Delta V)^2 + (\Delta W_2)^2 + (\Delta W_3)^2]/(\rho_L V + W_2 - W_3)^2 \quad (8)$$

The resulting error limits $\Delta\rho$ are $\pm$ 0.003 g cm^{-3} for both samples.

IV. Discussion + Conclusion

The values and limits of error obtained for the density of germanium at 25°C are

Sample I: 5.310 $\pm$ 0.003 g cm^{-3}

Sample II: 5.332 $\pm$ 0.003 g cm^{-3}

It is obvious that these two values deviate from each other by considerably more than the sum of the limits of error. Furthermore, this difference is even more disturbing when one considers the fact that any error in V will affect both densities in the same way and thus does not contribute to the uncertainty in the difference. Our results suggest

treatment is required, this should be given in an appendix and the resulting uncertainties should be stated, or shown as error bars on a plot, in the main body of the report. It is important to combine and simplify all expressions as much as possible in order to avoid obtaining unwieldy error equations. Since uncertainty values need not be calculated to better than about 15 percent accuracy, you should always try to find labor-saving approximations. Where the number of runs is so small that reliable limits of error cannot be deduced from statistical considerations, error limits must be assigned largely on the basis of experience and judgment. Try to make realistic assessments, avoiding excessive optimism (choosing error limits that are too small) or excessive pessimism (choosing limits that are unrealistically large). For a long and detailed report, a quantitative analysis of errors should always be derived and a numerical value of the uncertainty in the final result should be given. For a brief report, a qualitative discussion of the sources of error may suffice.

DISCUSSION

The Discussion is the most flexible section of the entire report, and you must depend heavily on your own judgment for the choice of topics for discussion. The final results of the experiment should be clearly presented. A comparison, often in a tabular or graphical that the material examined may be somewhat inhomogeneous, yielding two samples of slightly different bulk densities. We suggest the possibility that voids inaccessible to the liquid are present in sample I. On this assumption, the greater confidence should be placed in the higher value.

The "literature" value given in the Handbook of Chemistry and Physics is 5.323 g cm^{-3} at room temperature [3], but no information about the precision or source of this value is given. A more accurate value can be calculated from the atomic mass (72.61 g mol^{-1} for Ge) and the volume of the unit cell as determined by x-ray crystallography. Germanium has a cubic crystal structure with 8 atoms per unit cell, and the cubic unit cell length is 5.65754 ± 0.00002 Angstrom at 25°C[4]. Thus the x-ray density is

$$\rho(\text{x-ray}) = (8 \times 72.61)/(6.022137\times10^{23})(5.65754\times10^{-8})^3$$
$$= 5.3266 \text{ g cm}^{-3} \text{ at } 25°\text{C} \qquad (9)$$

The uncertainty in this value is dominated by the uncertainty ±0.03 in the atomic mass of Ge[3]. The resulting uncertainty in the x-ray density is ±0.0022 g cm^{-3}.

Although these two literature values are in excellent agreement, the x-ray value is to be preferred since the temperature is specified and an uncertainty value is known. The value of ρ for sample I is lower than the x-ray value,

form, between these results and theoretical expectations or experimental values from the literature is usually appropriate. A comment should be made on any discrepancies with the accepted or expected values. In this sample discussion, comment is also made on "internal discrepancies," possible systematic errors and ways to reduce them, and the relative importance of various sources of random error. A brief suggestion should be made, if possible, for an improvement in the experimental method. Other possible topics include suitability of the method used compared with other methods, other applications of the method, which could be explained by the presence of voids as suggested above. In contrast to this, the ρ value for sample II is slightly larger than the x-ray value (by an amount equal to the sum of the uncertainties in these two values). We have no good explanation for this difference, but it cannot be due to the presence of voids in the sample. One possible source of error is the volume V of the pycnometer. The stated value of 12.445 cm^3 has a rather large error limit of ±0.003 cm^3. Indeed, Appendix B shows that the largest contribution to the overall error in ρ comes from the uncertainty in V. No information is available about how V was determined or why it has such a large uncertainty. Our experimental precision indicates that a more reliable value for V could be obtained by measuring the weight of the pycnometer filled with water alone. Such a recalibration of the pycnometer volume would reduce the uncertainty in ρ due to random errors and might also shift the ρ values by a significant amount, which could perhaps improve the agreement between our ρ(sample II) and the x-ray value. However, a change in the value of V would not improve the agreement between the two samples.

Further work should be done to test the quality of the method (by studying a reference sample of well-known density) and the reproducibility of measurements on germanium samples prepared in different ways.

mention of any special circumstances or difficulties that might have influenced the results, discussion of any approximations that were made or could have been made, suggestions for changes or improvements in the calculations, mention of the theoretical significance of the result. At the end of several of the experiments in this book, there are questions that provide topics for discussion; however, you should usually go beyond these topics and include whatever other discussion you feel to be pertinent.

References

1. N. Bauer and S. Z. Lewin, "Determination of Density", in A. Weissberger and B. W. Rossiter (eds.), *Techniques of Chemistry*, Vol. I, Part IV, Chap. 2, esp. pp. 101-105, Wiley-Interscience, New York (1972).
2. D. R. Lide (ed.), *CRC Handbook of Chemistry and Physics*, 81th ed., p. 6-5, CRC Press, Boca Raton, FL (2000-2001).
3. *Ibid.*, p. 4-61.
4. A. Smakula and J. Kalnajs, Phys. Rev. **99**, 1737(1955); W. B. Pearson, *A Handbook of Lattice Spacings and Structures of Metals and Alloys*, Vol. 2, p. 971, Pergamon Press, Oxford (1967).

Appendix A: Data sheet

Appendix B: Sample calculations

REFERENCES

In citing a reference in the text of the report, use either a numerical superscript or a number in brackets to refer the reader to the appropriate entry in the reference list (e.g., . . . Smith[3] or . . . Smith [3]). The former style is used in this book, but the latter style is now more frequently used in scientific journals. An appropriate style for referring to a book is illustrated by entry 4. If the publisher's name is not well known, it should be given in full; if the city of publication is not well known, the state or country should also be given (see entry 2). The citation style for referring to a book containing chapters by several different authors is illustrated by entry 1. A recommended citation style for journal articles is shown in entry 4. Standard abbreviations for the titles of many important journals are given in Appendix E. For word-processed or handwritten reports, it is acceptable practice to underline the journal volume number (instead of using boldface type) and to use quotation marks to indicate book titles instead of italics.

APPENDICES

Important material that is so detailed that inclusion in the main body of the report would break the continuity of the text should be assembled in appendices. Examples are a long mathematical derivation, extensive tables of primary data (e.g., temperature–time values in calorimetry or composition–time values in chemical kinetics), extensive printout of computer data files, or a detailed listing of any nonstandard computer program.

Appendix A: Xerox copy of data for Exp. 13

24

Maria Smith — 18 Sept. 2008

Partner: John Klein

Exp. 13: Density of Ge

Pycnometer #7 has $V = 12.445 \pm 0.003\ cm^3$

Bath temperature $= 24.96°C$

	W_1 (empty)	W_2 (solid)	W_3 (solid + water)
Sample I			
Run 1	8.6313 g	42.0307 g	48.1749 g
Run 2	8.6308	42.~~0259~~ 0295	48.1715
Ave	—	42.0301 g	48.1732 g
Sample II			
Run 3	8.6316	45.8468	51.3055
Run 4	8.6299	45.8490	51.3017
Ave	—	45.8479 g	51.3036 g

Ave W_1 = 8.6309 g

estimate weighing uncertainty ± 0.001 g

temp variations of bath $\sim \pm 0.05°C$

All experimental data must be recorded directly in a laboratory notebook along with any identifying numbers on special apparatus and all necessary calibration data; see pp. 7–9 for further details. For reports made in an undergraduate laboratory course, a carbon copy or a photocopy of all pertinent notebook pages should be included as an appendix to the report. The first page of this appendix should have a clear heading with the student's name, the name of any partner, and the dates on which the experimental work was performed.

Appendix B: Sample calculation for Exp. 13

The computation of ρ for germanium sample I is shown here in detail. Using Eq. (5) with weights in gram, V in cm^3, and ρ_L in $g\ cm^{-3}$, we obtain

$$\rho(I) = \frac{(0.99705)(42.0301 - 8.6309)}{(0.99707)(12.445) + 42.0301 - 48.1732}$$

$$= \frac{(0.99705)(33.3992)}{12.4085 - 6.1431} = \frac{33.3007}{6.2654}$$

$$= 5.3150\ g\ cm^{-3}$$

The correction for air buoyancy is given by Eq. (6), and we find

$$\rho^* = 5.3150 + 0.0012\left(1 - \frac{5.3151}{0.99707}\right)$$
$$= 5.3098\ gm\ cm^{-3}$$

for the corrected density ρ^* of sample I. The 95% confidence limit for the density of this sample is obtained from Eq. (8):

$$(\Delta\rho)^2 = \frac{(5.315)^2}{(6.265)^2}\left[(0.997)^2(.003)^2 + (.001)^2 + (.002)^2\right]$$

$$= 0.720\ [8.95 + 1 + 4] \times 10^{-6} = 10.0 \times 10^{-6}$$

$$\therefore \Delta\rho = 0.0032\ g\ cm^{-3}$$

Error Analysis

A sample calculation should also be presented as an appendix to an undergraduate laboratory report. This appendix should show how one obtains the final results starting from the raw data. In general, the numbers used in the computations should have more significant figures than are justified by the precision of the final result, in order to avoid mathematical errors due to roundoff. Units should be included with each step of the calculation. Also specify the source of raw data used (e.g., run 5 on page 14 of notebook).