High-Pressure Calibration
A Critical Review

D. L. Decker, W. A. Bassett, L. Merrill, H. T. Hall, and J. D. Barnett

High Pressure Data Center
Brigham Young University
Provo, Utah 84601

A critical review of experimental technique for measuring high pressures has been made. The broad coverage includes discussions relating to (a) the establishment of a primary pressure scale using the free-piston gage, (b) the selection and precise measurement of identifiable phase changes as fixed pressure points, and (c) the use of interpolation and extrapolation techniques such as resistance gages, equations of state, and optical changes. The emphasis is on static pressure measurements above 10 kbar, but shock measurements are also considered for completeness. The pressure values to be associated with the fixed points have been analyzed in detail. Temperature measurement in the high pressure environment is also reviewed. The accuracy with which pressures can be measured has been carefully considered; the maximum accuracies now obtainable are considered to be of the order of 0.02 percent at 8 kbar, 0.25 percent at 25 kbar, 2 percent at 50 kbar, and 4 percent at 100 kbar.

Key words: Calibration of pressure scales; critically evaluated data; high pressure; high pressure phase changes; pressure measurement.

Contents

	Page
1. Introduction	774
1.1. Pressure	774
a. Definitions of Pressure	775
b. Hydrostaticity	776
c. Equilibrium Pressure	777
1.2. Analogy with Temperature Scale	779
2. The Primary Pressure Scale	780
2.1. The Free-Piston Gage	781
a. Theoretical, Followed by Gage Intercomparison	783
b. Controlled-Clearance Gage	784
c. Similarity Method	786
d. Summary	788
2.2. The Mercury Manometer	789
2.3. Piston-Cylinder Gage	791
2.4. Galvanic Cell as a Primary Pressure Indicator	791
3. Fixed Points of Pressure Scale	791
3.1. Criteria for Selection	792
a. Detectability	792
b. Kinetics	792
c. Sample Purity	793
d. Transformation Characteristics	793
3.2. Error Analysis	793
3.3. Fixed Points Below 30 Kbar	794
a. Mercury	794
b. Bismuth I–II	795
3.4. Fixed Points Between 30 and 80 Kbar	797
a. Thallium	797
b. Cesium	798
c. Barium I–II	799
d. Bismuth III–V	800
3.5. Fixed Points Above 80 Kbar	802
a. Tin	802
b. Iron	802
c. Barium II–III	803
d. Lead	803
4. Interpolation and Extrapolation Methods and their Calibration	804
4.1. Theoretical Equations of State	804
a. Proposed Isothermal Equations	804
b. The Equation of State Including Temperature	807
c. Equations of State for Specific Materials	808
d. Alkali Metals	808
e. Ionic Compounds and in Particular NaCl	808
4.2. Experimental Techniques for P–V–T Measurements	810
a. Dilatometry	811
b. High Pressure-Temperature X-ray Diffraction	811
c. Neutron Diffraction	814
d. Sonic Velocity Measurements	814
4.3. Resistance Gage	816
a. Requirements of Acceptable Resistance Gage	816
b. The Manganin Gage	817
c. Stability and Reversibility	818
d. Variation of Resistance and Pressure Coefficient with Temperature	819
e. Linearity of the Resistance Change	819
f. Gold–2.1 Percent Chrome Gage	821
g. Conclusion	821
4.4. Other Pressure Gages	821
a. Optical Absorption as a Pressure Gage	821
b. Refractive Index as a Pressure Gage	822

Contents — Continued

Page

c. Phase Boundaries as Pressure Gages.....823
5. Dynamic Pressure Methods.....824
5.1. The Hugoniot Equation of State.....824
5.2. Phase Transitions Via Shock Measurements.....825
a. Bismuth I–II Transition.....826
b. Transition in Iron.....827
6. Pressure Scale at Elevated Temperatures.....827
7. Summary.....830
7.1. Interpolation Methods.....830
7.2. Dynamic Pressure Studies.....831
8. References.....831

List of Tables

Page

Table 1. Estimated Uncertainties of the Assigned Values of the Defining Fixed Points in Terms of Thermodynamic Temperatures.....780
Table 2. Elastic Parameters for Metals in Similarity Intercomparison.....787
Table 3. Mercury (Liquid-α) at 0 °C.....795
Table 4. Bismuth I–II Transition at 25 °C.....796
Table 5. Thallium II–III Transition.....798
Table 6. Cesium Transitions.....799
Table 7. Barium I–II Transition at 25 °C.....801
Table 8. Bismuth III–V Transition at 25 °C.....802
Table 9. Tin I–II Transition at 25 °C.....803
Table 10. Iron α–ϵ Transition.....803
Table 11. Barium II–III Transition at 25 °C.....803
Table 12. Lead I–II Transition at 25 °C.....804
Table 13. Reported Pressure-Volume Relationships for NaCl.....809
Table 14. Calculated Pressures vs Compression for NaCl at 25 °C.....810
Table 15. Reported Pressure-Volume Relationships for MgO.....810
Table 16. Relationship of Elastic Moduli to Acoustic Wave Velocities in Cubic Crystals.....815
Table 17. Fixed Point vs Manganin Gage Intercomparison Values.....820
Table 18. Bi Transition by Shock Measurements...826
Table 19. Temporary Working Pressure Scale.....831

1. Introduction

1.1. Pressure

The use of pressure as a parameter in the study of materials was pioneered principally by Professor P. W. Bridgman, who for forty years investigated most of the elements and many other materials using diverse techniques (Bridgman, 1964). By the early 1950's high-pressure phenomena began to attract widespread interest. The following list of the number of papers published in the field of high-pressure research indicates its very rapid growth in the last few years.

Year	Number of papers
1950	96
1955	243
1960	542
1964	746
1966	1025
1968	1271
1969	1367
1970	1700 (estimated)

The successful synthesis of diamond in 1954 (Hall, 1961) made possible by the development of new apparatus and techniques clearly showed the potential of high-pressure applications for industrial purposes and stimulated the growing interest in the field. For those in basic research the high pressures available using the new techniques opened up a whole new vista in investigations of properties of materials. Studies at high pressures have been successfully undertaken involving such things as chemical synthesis, melting curves, solid-solid phase boundaries, x-ray and neutron diffraction, optical phenomena, magnetic properties, NMR, EPR, Mossbauer, and ultrasonic experiments, among others. Due to the geometry and/or the complexity of the apparatus necessary to contain samples and generate the high pressures, many standard measuring techniques must be drastically modified, and associated experimental accuracies are generally reduced.

As any technological field develops, the need for precise and accurate characterization of the parameters of interest is paramount. In the high-pressure field two calibration problems are apparent: (a) the establishment of a workable and accurate high-pressure scale defined in procedural detail, and (b) the accurate determination of temperatures in a high-pressure