

FIG. 4. Pressure calibration curve. The horizontal lines indicate the maximum spread of several calibration runs of Ce, Bi, Yb, and Ba. Only one pressure calibration point for thallium appears. The dashed portion of the curve indicates an extrapolation to zero pressure.

The calibration curve obtained appears in Fig. 4. Evidently considerable deformation of the pyrophyllite washers surrounding the cell takes place before the cell itself is significantly compressed, thus making the pressure curve uncertain near the origin.

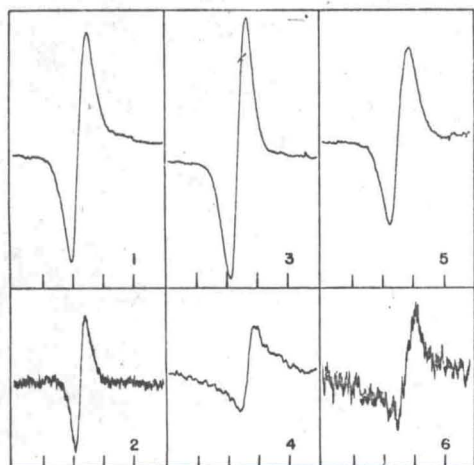


FIG. 5. Sample resonances in ruby under pressure: (1) $m_s = \frac{1}{2} \rightarrow m_s = -\frac{1}{2}$ transition at 10 kilobars, (2) $m_s = \frac{3}{2} \rightarrow m_s = \frac{1}{2}$ transition at 10 kilobars, (3) $m_s = \frac{1}{2} \rightarrow m_s = -\frac{1}{2}$ transition at 30 kilobars, (4) $m_s = \frac{3}{2} \rightarrow m_s = \frac{1}{2}$ transition at 30 kilobars, (5) $m_s = \frac{1}{2} \rightarrow m_s = -\frac{1}{2}$ transition at 50 kilobars, (6) $m_s = \frac{3}{2} \rightarrow m_s = \frac{1}{2}$ transition at 50 kilobars. In upper graphs, each division represents 96 G. In lower graphs, each division represents 169 G.

Sample Resonances in Ruby Under Pressure

Figure 5 shows resonance patterns of ruby taken at 10, 30, and 50 kilobars. The two patterns shown at each pressure are those due to the transition in which the spin quantum number changes from $\frac{1}{2}$ to $-\frac{1}{2}$ and from $\frac{3}{2}$ to $\frac{1}{2}$ with the gross magnetic field oriented parallel to the c axis of the single ruby crystal.⁹ The pressure transmitting medium was Viscasil in each case. The ruby sample was a roughly circular disk 0.007 in. thick by approximately 0.050 in. in diameter.

The apparatus described herein provides a new tool for the study of very high pressure effects in materials which exhibit paramagnetic resonance. This tool is very sensitive to minute changes in the crystalline field of a sample and therefore reveals the need for a truly hydrostatic pressure medium at the elevated pressures under consideration. Some lack of consistency in the zero field splitting of ruby (calculated from the spin Hamiltonian: $\mathcal{H} = \frac{1}{2} \delta S_z^2 + \beta H g_{\parallel} S_z$)⁹ from run to run which correlates with increasing linewidths at pressures above 15 kilobars suggests that Viscasil does not produce a strictly hydrostatic pressure in this region. Litster and Benedek¹⁰ conclude that silver chloride becomes a hydrostatic pressure medium above 15 kilobars, but our experience has been that the thin ruby sample is broken by the silver chloride before the hydrostatic condition is reached.

The high pressure limit has not been established for this apparatus. Resonances have been observed in ruby well above the barium transition. The limiting element appears to be the strength of the lower alumina anvil cavity.

ACKNOWLEDGMENTS

We wish to acknowledge the assistance of the following: Dr. H. Tracy Hall, Dr. Daniel L. Decker, and Dr. Howard Vanfleet with each of whom many valuable discussions were held; Dr. James S. Hyde of Varian Associates who was instrumental in developing the superheterodyne microwave bridge; Electroceramics Company who developed an especially pure alumina for our work; Mr. Herbert Kirchoff who fabricated many of the parts of the apparatus.

⁹ J. E. Geusic, Phys. Rev. **102**, 1252 (1956).

¹⁰ J. D. Litster and G. B. Benedek, J. Appl. Phys. **34**, 688 (1963).