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THE EFFECT OF PRESSURE ON THE MÖSSBAUER
SPECTRUM OF Fe^{57} IN IRON METAL

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MÖSSBAUER SPECTRUM OF Fe⁵⁷ IN IRON METAL

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May 1963

The Effect of Pressure on the Mössbauer Spectrum of Fe⁵⁷ in Iron Metal

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Abstract

The effect of pressure on the Mössbauer spectrum of Fe⁵⁷ in iron metal has been studied to a pressure presumably higher than 140 Kbar. The sample was a disc of iron metal in .250-inch diameter Bridgman anvils, and pressure is used to denote the average load on the anvil face. At pressures up to 120 Kbar, a six line spectrum characteristic of α -iron was observed. At 140 Kbar, a seventh line appeared in the spectrum at -0.12 ± 0.06 mm/sec relative to stainless steel. This line was attributed to the appearance of the high pressure phase of iron. The variation of the magnetic splitting of the spectrum in α -iron agreed with that observed to 65 Kbar by nuclear magnetic resonance.¹²

Since the discovery of recoilless nuclear emission of photons in 1958 by Mössbauer,¹ the Mössbauer effect has been used for a great variety of experiments, many of which concern atomic environments in solids. The theory of the Mössbauer effect and the conditions for observation of recoilless radiation have been worked out quite thoroughly² and will not be discussed here. Experimentally, the effect has been observed in many isotopes; however, the most intense effect is found in Fe⁵⁷, due partly to the low (14.36 KeV) photon energy. The Fe⁵⁷ spectrum has been observed in metallic iron over the temperature range from 4° K to above 1300° K, where the γ -phase is stable,³ as well as for Fe⁵⁷ dissolved in many transition metals and alloys. The spectrum also has been studied in chemical environments ranging from simple compounds such as the oxides to such complex molecules as ferrocene and hemoglobin. Since only five nuclear parameters are needed to explain the spectra, a considerable quantity of information is available about the dependence of the spectrum on non-nuclear parameters such as the magnetic field at the nucleus due to the electrons. For example, Nagle et al.⁴ have shown that the temperature dependence of the magnetic field at the nucleus closely follows the temperature dependence of the saturation magnetization; and Walker, Wertheim and Jaccarino⁵ have been able to correlate the observed

chemical shift of the spectrum with the electron density at the nucleus.

This collection of information is available in new experiments to aid not only in explaining the changes in the spectra but also in inferring the changes in the solid which produce the spectral changes. Particularly in high pressure research, where so few experiments yield detailed information about behavior on an atomic scale, this background of information, together with the high sensitivity of the experiment to the solid environment, makes the Mössbauer effect a useful experimental tool. This paper reports on the development of a technique for conducting Mössbauer experiments on materials under very high pressures and the results obtained from a study of the effects produced by pressure on the Mössbauer spectrum of Fe^{57} in iron metal.

Two experiments observing Mossbauer spectra in solids at elevated pressures have been reported. Pound, Benedek and Drever⁶ made a precise study of the shift in the photon energy (the chemical shift) produced by hydrostatic pressures up to 3 Kbar. They attributed most of the shift $d \ln v/dP)_T = - (2.61 \pm 0.10) \times 10^{-18}$ per kg/cm^2 to the increase of the electron density at the nucleus. They did not report any details of the fine structure of the spectrum.

The authors with two coworkers studied the influence of pressure on the Mössbauer effect of Dy^{161} formed in situ in gadolinium metal by neutron irradiation of Gd^{160} in an earlier experiment.⁷ The theoretical prediction made by Hanks⁸ that application of pressure would permit observation of recoilless radiation not detectable at one atmosphere was demonstrated, and very dramatic changes of the fine structure were found.

The experiment was performed with a constant velocity drive spectrometer, which has the advantage of high precision in velocity measurement. However, a serious disadvantage was found with this technique due to the variation of intensity of radiation from the source due to the slow creep of the sample because of the very long periods of time required to determine the spectrum. This variation necessitated many repetitions of a spectrum in order to obtain reproducible results. Because of the experience with the above experiment, it was decided to develop a Mössbauer spectroscopic technique for high pressure application by adapting a velocity-sweep type of spectrometer.

Fe^{57} in iron metal was chosen for several reasons. The high pressure phase diagram of iron metal is not clearly understood with respect to the nature of the reported high pressure phase. If this phase could be obtained, the Mössbauer spectrum could help in the characterization of the new phase. Furthermore, the variation with pressure of the coupling between electrons in ferromagnets is poorly understood. The observed variation of the magnetic field at the nucleus with temperature already mentioned suggested that this experiment could give useful information about the effect of compressing the lattice on the magnetic behavior of this important ferromagnet.

Because it was anticipated that the recoilless radiation would be attenuated severely in passing out from the pressure vessel, it was decided to contain the radiation source under pressure rather than the absorber. By doing this, the radiation had to pass through the wall of the pressure vessel only once. The source was a disc of iron metal

.175 inch in diameter and .007 inch thick. To minimize self-absorption of the recoilless radiation, iron enriched to 99.9% in Fe^{56} was used to make the disc.

The 14-KeV excited state of Fe^{57} was obtained from the 270-day half-life electron capture decay of Co^{57} . The long half-life of this decay controls the intensity of radiation which is essentially constant for periods of a few hours. To obtain the Fe^{57} in an iron rather than a cobalt environment, the Co^{57} was electroplated onto the iron disc, and the sample was annealed at 1000° C for two hours in a hydrogen atmosphere after electroplating. The sample preparation was done by the Nuclear Science and Engineering Corporation. About 6 millicuries of Co^{57} were plated near the edge of the circular face of the first sample prepared in this way. When this sample was contained in the anvils, it was impossible to detect the 14-KeV radiation even without an applied load because of the attenuation of the radiation by the iron metal. A second sample was prepared with 4 millicuries of Co^{57} plated on the .007 inch edge along one-half of the circumference, and this was used for the measurements reported here. Assuming a uniform distribution of Co^{57} within .005 inch from the edge, the cobalt concentration should be less than 0.1%.

The iron disc was contained between a set of .250 inch face diameter Bridgman anvils by a ferric oxide coated ring of pyrophyllite .031 inch thick and .010 inch high. The Fe_2O_3 with which the ring was coated was enriched to 99.9% Fe^{56} to minimize absorption of the 14-KeV radiation. The details of the ring and anvils have been described elsewhere.⁹

Pressure was applied with a 200-ton capacity hydraulic press through a calibrated, strain-gauge instrumented load cell. This permitted continuous monitoring of the load on the anvil faces, which was constant within at most 2% at any pressure.

No information is available about the distribution of pressure in an iron disc in this geometry, so pressures reported are average pressures on the anvil face. Experience in this geometry with silver chloride as a pressure medium indicates that about only 82% of the area of the anvil face is load bearing, and the average pressure on the disc is about 20% higher than the average pressure on the anvil face. Neither iron nor silver chloride is a hydrostatic pressure medium, and pressure is not uniformly distributed across the load bearing area. In silver chloride, the actual pressure was found to be highest at the interface with the pyrophyllite ring. Because 14-KeV radiation is attenuated severely by iron metal, most of the radiation detected comes from the region of the iron-ring interface, so the actual pressure seen by the Fe^{57} may be considerably higher than the average pressure on the anvil face. In future experiments, the uncertainty in the pressure can be reduced by replacing the solid metal disc with a circular section from the circumference and filling most of the volume with a well-studied pressure medium.

The spectrometer was a modification of the low temperature spectrometer of Shirley et al.¹⁰ The absorber was made with a .001 inch thick foil of 19-9 stainless steel, enriched to 65% with Fe^{57} . The foil had a broad, single absorption which permitted the six components of the

Fe^{57} spectrum in iron to be resolved. The foil was attached to a moving yoke which permitted insertion of a scintillation detector between the foil and the loudspeaker which provided the motion. The yoke was attached to the loudspeaker through the moving element of a Sanborn 6LVI velocity transducer which forced the transducer to sense all the motion of the absorber relative to the press, to which the stationary element of the transducer was rigidly mounted. Thus, the output voltage of the transducer was directly proportional to the velocity of the absorber relative to the source.

The 14-KeV radiation from the sample passed normal to the absorber, and the transmitted intensity was detected with a $\text{NaI}(\text{Tl})$ crystal scintillator 1 mm thick mounted on the face of a Dumont 5292 phototube. The crystal face made an angle of about 45° to the incident radiation. The output of the phototube was amplified with a Model VI Linear Amplifier using a double delay clipping mode. By careful choice of components, the noise level of the detector and amplifier was reduced to a level corresponding to less than 5-KeV. The output of the amplifier was analyzed for 14-KeV radiation. When a 14-KeV photon was detected, a square pulse proportional to the velocity at which the absorber was moving was generated and was fed into a 400-channel pulse height analyzer. In this manner a spectrum of transmitted photon intensity against photon energy spectrum was generated. Because a sinusoidal drive was used, this spectrum had to be normalized for variation in time spent in different velocity ranges. A typical normalized spectrum is shown in Figure 1.

The spectrometer was calibrated with the one atmosphere spectrum using data of Dash et al.¹¹ Pressure was applied to the sample, and detailed spectra were taken at loads of 50, 60, 90, 105, 120, and 140 Kbar. The average pressure on the iron disc is 20% higher. At least two spectra were taken at each pressure; the average statistical error per channel was at most 0.5% of 100% transmission. The observed intensity of absorption decreased from 13% for the most intense peak of the uncovered sample at 1 atm to about 2% for the same peak in the 140-Kbar spectrum. The decrease is due to attenuation of the recoilless radiation by the ring relative to the amount of scattered radiation and no information could be obtained about the variation of the recoil free fraction with pressure. The loss of intensity at 160 Kbar was so great that no absorption greater than 0.5% could be detected.

At pressures up to 120 Kbar, the spectra consisted of six lines appropriate to Fe^{57} in ferromagnetic α -iron. In some of the spectra, the weak inner lines were poorly resolved. The 105-Kbar spectrum, shown in Figure 1, is typical of these spectra. The spectra were analyzed in terms of three parameters: A, measuring the magnetic splitting as defined in Equation (1); B, measuring the electric quadrupole splitting as defined by Equation (2); and v_0 , the velocity of the center of gravity of the spectrum, or chemical shift, with respect to the stainless steel absorption. μ_0 is the magnetic moment of

$$A = -\mu_0 H \quad (1)$$

$$B = \frac{1}{4} e^2 qQ \quad (2)$$

the ground state of Fe^{57} , Q the electric quadrupole moment of the 14-KeV state of Fe^{57} , and H and q are the magnetic field and the electric field gradient at the iron nucleus, respectively. These parameters can be determined from the position of the four outer lines of the spectrum according to Equations (3)-(5),

$$A = (v_6 - v_1)/5.430 \quad (3)$$

$$B = (v_1 + v_6 - v_2 - v_5)/4 \quad (4)$$

$$v_c = (v_1 + v_2 + v_5 + v_6)/4 \quad (5)$$

where v_i is the velocity of the i th line, numbered from the lowest energy line. The values obtained from these parameters are given in Table I. A and v_c are plotted as functions of pressure in Figures 2 and 3, respectively.

At 140-Kbar (Figure 4), a seventh line appeared in the spectrum near zero velocity (at -0.127 ± 0.064 mm/sec) in addition to six lines which are characteristic of α -iron. The constants calculated for the six line spectrum are $A = 1.87 \pm 0.02$ mm/sec, $B = 0.1 \pm 0.1$ mm/sec, and $v_c = +0.025 \pm 0.035$ mm/sec. These values seem consistent with values for α -iron at lower pressures, although the variation with pressure is not so smooth as at lower pressures. In particular, the chemical shift is positive with respect to 105 Kbar and 120 Kbar, and A has dropped abruptly. More precise data is needed on this point. The positions of the two inner lines of the six line spectrum calculated with these parameters agrees quite well with the observed absorptions. The additional line can not be explained in terms of radiation of higher

Table I.

Pressure	A	B	v_c
Kbar	mm/sec	mm/sec	mm/sec
	± 0.02	± 0.1	
.001 (1 atm)	1.97	0.0	+ 0.045 \pm 0.032
50	1.93	0.0	+ 0.057 \pm 0.035
60	1.92	0.0	+ 0.120 \pm 0.060
90	1.95	0.0	- 0.007 \pm 0.032
105	1.95	0.0	- 0.069 \pm 0.032
120	1.93	0.2	- 0.113 \pm 0.032

multipolarity from an α -iron lattice site; and the seventh line is most easily explained in terms of a second iron atom environment such as the high pressure phase of iron.

In Figure 2, the observed variation of the magnetic splitting is compared with the more precise results obtained in a nuclear magnetic resonance experiment to 65 Kbar by Litster and Benedek.¹² The agreement is quite good. The decrease in the magnitude of the magnetic splitting, which arises from a magnetic field anti-parallel to an externally applied field, can be explained by a combination of two effects.

According to the calculations of Freeman and Watson,¹³ the major contribution to the magnetic field at the nucleus arises, through a Fermi contact interaction, from exchange polarization of the s electrons by the unpaired 3d electrons, which are responsible for the atomic magnetic moment. The resulting field has the direction of the net spin density at the nucleus. Exchange acts to decrease the electrostatic repulsion of electrons of parallel spin, drawing them closer together than electrons with anti-parallel spin. For 1s and 2s electrons, whose average radii are less than the 3d electrons, the distribution of the electrons with spin parallel to the 3d electrons is expanded, lowering the parallel spin density at the nucleus compared to the anti-parallel spin density. This creates a net magnetic field at the nucleus directed anti-parallel to the 3d spins. The behavior of electrons whose average radius is greater than the 3d electrons is similar except the parallel spin density is compressed relative to the anti-parallel spin density, resulting in a field parallel to the 3d spin. As Marshall first suggested,¹⁴ at least the contribution to the total magnetic field $H(0)$ which is due

to exchange polarization by the 3d electrons should be roughly proportional to the atomic magnetic moment, which can be measured in terms of the saturation magnetization σ_0 . At constant volume, this field might be expected to vary directly with the saturation magnetization, which is supported by the measurement of the temperature variation of the field at the nucleus by Hanna et al.⁴ mentioned earlier. This variation can be expressed mathematically as:

$$\left. \frac{d \ln H(0)}{d \ln \sigma_0} \right)_V \approx 1$$

As the lattice is compressed, the distribution of the "outer" electrons is compressed while the distribution of the "inner" electrons remains unchanged to first order. (By implication, this defines "outer" electrons.) The compression of the "outer" electrons affects the field in two ways. To the extent that the spin distribution is unaffected by compression of the electrons, the parallel field H_0 at the nucleus due to these electrons increases in proportion to the compression of the lattice. (The s electron density also is increased by transfer of some 3d electrons to the s band, by which mechanism σ_0 is reduced.) As the "outer" electrons are compressed relative to the 3d electrons, however, increased mixing of the 3d and 4s electrons reduces the effectiveness with which exchange can polarize the spin density of the "outer" electrons, which are primarily 4s electrons. This decreases the parallel spin density at the nucleus, reducing the parallel magnetic field at the nucleus. The net dependence of $H(0)$ on volume is the sum of these effects.

The observed variation of the magnetic field at the nucleus with pressure can be attributed to a combination of the volume and saturation magnetization dependence. This is expressed in Equation (6). By definition,

$$\frac{d H(O)}{d P} = \left. \frac{\partial H(O)}{\partial \sigma_o} \right)_V \frac{d \sigma_o}{d P} + \left. \frac{\partial H_o}{\partial V} \right)_{\sigma_o} \frac{d V}{d P} \quad (6)$$

H_o is the fraction of $H(O)$ which varies with volume; thus, Equation (6) is equivalent to the more general Equation (6a).

$$\frac{d H_o}{d V} \equiv \frac{d H(O)}{d V}$$

$$\frac{d \ln H(O)}{d P} = \left. \frac{\partial \ln H(O)}{\partial \ln \sigma_o} \right)_V \frac{d \ln \sigma_o}{d P} + \left. \frac{\partial \ln H(O)}{\partial \ln V} \right)_{\sigma_o} \frac{d \ln V}{d P} \quad (6a)$$

If (6a) is evaluated using the data of Litster and Benedek for $\frac{d \ln H(O)}{d P}$,¹² Kouvel's data for $\frac{d \ln \sigma_o}{d P}$,¹⁵ and Bridgman's value of the

compressibility of iron,¹⁶ $\left. \frac{\partial \ln H(O)}{\partial \ln V} \right)_{\sigma_o}$ is Litster and Benedek's $\frac{\partial \ln A}{\partial \ln V}$,

which equals - 0.19. The increase of the magnetic field at the nucleus $H(O)$ with decreasing volume at constant σ_o then arises from predominance of a decrease in exchange coupling of the "outer" electrons to the 3d electrons over the effect of compression of the "outer" electron density, at constant atomic magnetic moment. This decreases the parallel magnetic field at the nucleus making $H(O)$, which is anti-parallel to the 3d spins, increase.

Furthermore, from this simple model, the contribution of the "outer" electrons to the magnetic field at the nucleus is indicated to be about 20% of the magnitude of the total field.

α -iron is a cubic lattice system for which electric field gradients are prohibited by symmetry for the undistorted lattice. Observation of non-zero values of B at the higher pressures achieved can be explained in two ways. A pressure gradient, such as the observed in silver chloride in this geometry, might distort the lattice sufficiently for the observed splitting. The distortion should increase gradually with pressure and might be obscured at lower pressures by the broad absorption line of the stainless steel foil. However, the thickness of the sample from which the 14-KeV radiation can be detected is small, and a very large gradient would be required. A phase transition to a lattice system of lower symmetry, such as the hcp lattice suggested by Jamieson for the high pressure phase of iron,¹⁷ also could produce quadrupole splitting.

The variation of the chemical shift with pressure is shown in Figure 3 together with an indication of the variation obtained by Pound et al.⁶ at lower pressures. Except for the 140 Kbar point, there is a gradual shift of the spectrum to lower energy which increases with pressure. However, as the spectrometer does not measure the absolute velocity directly, the precision of this measurement is not high. A minor variation in the voltage of the square pulse corresponding to zero velocity shifts the entire spectrum. This contribution can be checked by observing the location of the ends of the velocity spectrum on the pulse height analyzer; however, the precision in this correction will be of the order of a channel width, 0.063 mm/sec. This might account for the apparent change in direction of the shift observed at 140 Kbar.

The dependence of the chemical shift on the 4s electron density at the nucleus has been discussed by Walker, Wertheim and Jaccarino.⁵

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According to their Figure 1, for a $3d^{8-x} 4s^x$ electron configuration, the change of the chemical shift from 0.10 mm/sec to - 0.11 mm/sec relative to stainless steel, observed from 1 atm to 120 Kbar, corresponds to an increase of about 8 - 10 % in the $4s$ electron density at the nucleus. This corresponds closely to the decrease in the volume of the iron lattice at 130 Kbar observed in the dynamic high pressure investigation of Brancroft et al.¹⁸

The seventh line observed in the spectrum at 140 Kbar is not at the position of the normally forbidden transition of the α -iron spectrum, and it can be explained in terms of a second environment for Fe^{57} . The high pressure work by a number of workers^{17,18,19} would be such a second environment. Jamieson and Lawson have suggested that this phase is hexagonal close packed from x-ray measurements.¹⁷ In static high pressure experiments,^{17,19} a sharp transition is not observed which could explain the coexistence of a six line (bcc) spectrum and an apparent single line (hcp) spectrum. The coexistence has been observed at 170 Kbar in x-ray studies by Jamieson.²⁰

The single Mössbauer line indicates that Fe^{57} may not be aligned in the hexagonal phase. This might indicate a paramagnetic medium; however, a single line spectrum also has been observed for Fe^{57} in a number of magnetic alloys and as a dilute impurity in antiferromagnetic chromium. This effect might be attributed to a rapid relaxation of the nuclear substates. Recently, Clogston and Jaccarino have discussed the observation of unsplit Fe^{57} Mössbauer spectra in some magnetic transition metal alloys in terms of delocalization of the magnetic moments in these materials.²¹

In summary, a technique for making reliable Mössbauer spectral measurements on materials at very high pressures has been developed and applied to the spectrum of Fe^{57} in iron metal up to pressures above 140 Kbar. The spectrum of Fe^{57} in the high pressure phase of iron has been detected and factors contributing to the variation of the magnetic field at the Fe^{57} nucleus in α -iron have been discussed. The decrease in the magnetic field at the nucleus in α -iron has been attributed to the predominance of the changes due to the decrease of the atomic magnetic moment over the increase arising from changes in the exchange polarization of the 4s electron.

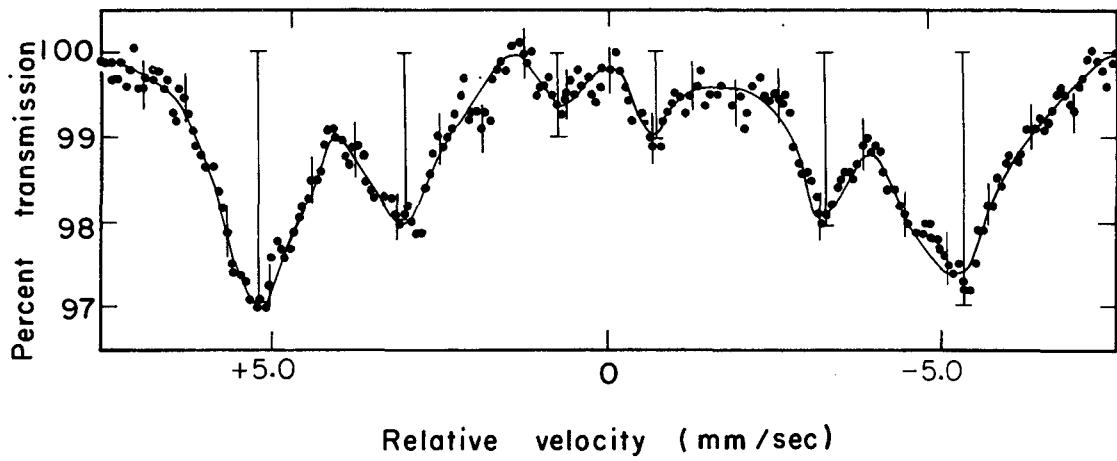
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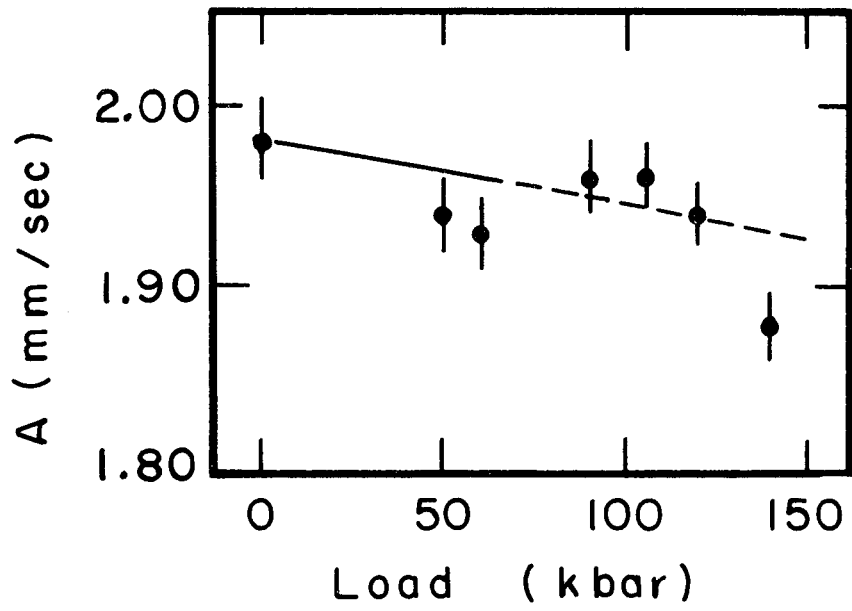
Figure Captions

- Figure 1. 105-Kbar spectrum, typical of the α -iron spectra. The statistical error is indicated on every tenth channel. The bars indicate the position and theoretical intensities of the absorption lines. The positions of the two inner lines are calculated.
- Figure 2. The variation of the magnetic splitting parameter with pressure. The straight line is calculated from the nuclear magnetic resonance results of Litster and Benedek.¹²
- Figure 3. The variation of the chemical shift with pressure, compared with the results of Pound, Benedek and Drever⁶ (dashed line) obtained at lower pressures.
- Figure 4. 140-Kbar spectrum showing seven lines. The statistical error is indicated on every tenth channel. The bars indicate the position and theoretical intensities of the absorption lines. The position of lines 3 and 5 are calculated assuming a six line spectrum.



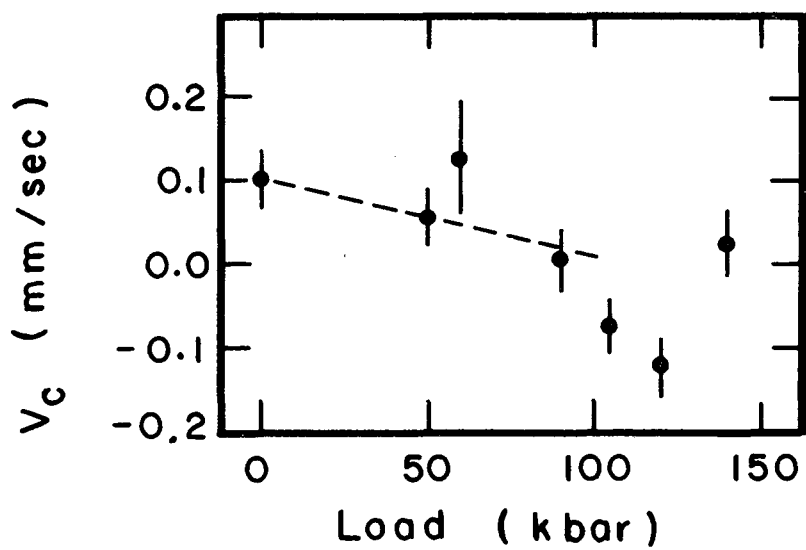
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Fig. 1.



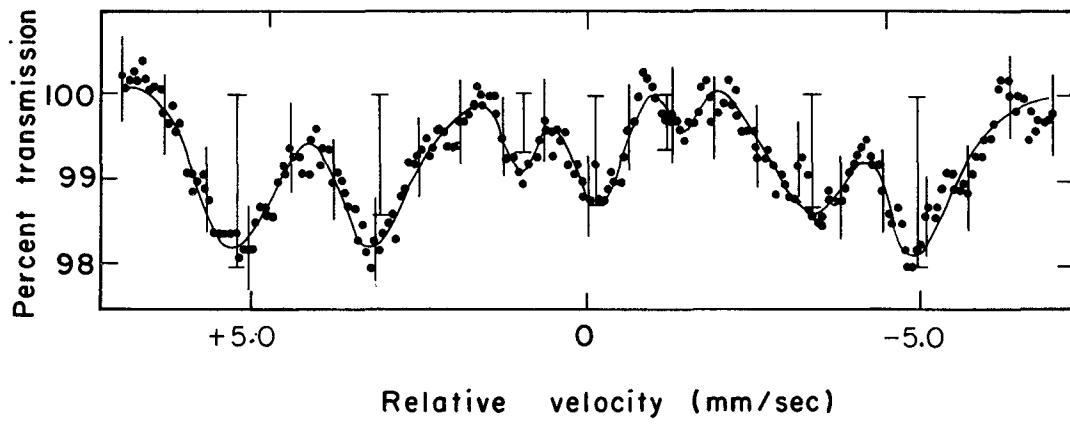
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Fig. 2.



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Fig. 3.



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Fig. 4.

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