age were examined. Each gland was counted in a 5- by 5-cm NaI, well-crystal detector with multichannel pulse analysis for 30 minutes. The minimum amount of I^{131} giving a detectable photopeak with this equipment was 30 pc. No gamma radioactivity of any sort was detected in any of these glands. That is, in the energy range from 0.06 to 2.0 Mev, the net counting rate was not significantly above background and no peaks were present. The natural K^{40} in the glands was not observed because the amounts of tissue were too small.

During this April to September period, the milk supplies of several cities in the United States contained readily measurable amounts of I^{131} (*7*). However, several milk samples from the Los Angeles area were examined by us and found to contain no I^{131}, a result not unexpected inasmuch as there was no local rainfull during that period. Furthermore, the dairy stock in the area is fed stored hay and grains, rather than open pasturage, a procedure which permits time for decay of the relatively short-lived radioiodines (*8*).

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3. Cesium-137 concentrations in 155 Los Angeles residents ranged from 9 to 160 pc per gram of body potassium, with a mean of 43 pc.
4. The sensitivity was 2.7 × 10^3 count/min per microcurie of K^{40} in a band 200-kev wide in which the background was 24 count/min. For Cs^{137} the values were 2.2 × 10^4 count/min per μc, background 104 count/min in a 200-kev band, and for I^{131}, 5.8 × 10^5 count/min per μc in a 110-kev band with a background of 90 count/min.
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Mössbauer Spectrum of Iron-57 in Iron Metal at Very High Pressures

Abstract. *The effect of pressure on the Mössbauer spectrum of Fe^{57} in iron metal has been studied as the pressure was increased presumably to more than 140 kbar. 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.*

This paper reports on the development of a technique for conducting Mössbauer experiments (*1*) on materials under very high pressure and on the results obtained from a study of those changes produced by pressure in the Mössbauer spectrum of Fe^{57} in metallic iron. The effect of pressure on the Fe^{57} Mössbauer spectrum in iron metal is of interest for several reasons. All the nuclear parameters upon which the Fe^{57} spectrum depends can be determined from other experiments. Thus, the spectrum can be used as a probe into non-nuclear effects to which the spectrum is sensitive, such as the electronic charge density and spin polarization at the nucleus. The Mössbauer spectrum of Fe^{57} can be used both to detect and to characterize the reported high-pressure phase of iron metal (*2, 3*). Furthermore, the dependence of the spectrum on the magnetic field at the nucleus can be used to study the effect of compression on the ferromagnetism of α-iron. Few other high-pressure experiments can yield such detailed information about behavior on an atomic scale, which makes the Mössbauer effect a most useful experimental tool for high-pressure research.

The theory of the Mössbauer effect and conditions for observation of recoil-free radiation have been worked out quite thoroughly (*4*) and will not be discussed here. Two experiments observing Mössbauer spectra in solids at elevated pressures have been reported. In a precise study (*5*) of the shift in the photon energy (the chemical shift) produced by hydrostatic pressures up to 3 kbar, most of the shift was attributed to the increase of the electron density at the nucleus. Other effects of pressure on the fine structure of the spectrum were not reported. The influence of pressure on the Mössbauer effect of Dy^{161} (dysprosium) formed *in situ* in Gd (gadolinium) metal has also been studied. The theoretical prediction made by Hanks (*7*) that higher pressure would permit observation of recoil-free radiation not detectable at one atmosphere was demonstrated, and very dramatic changes of the fine structure were found.

Since the 14-kev radiation would be attenuated severely in passing through the walls of the pressure vessel, it was decided to contain the radiation source under pressure in order that the radiation pass through the wall of the pressure vessel only once. For simplicity, the source was a disk of iron metal 0.175 inch (0.445 cm) in diameter and 0.007 inch (0.017 cm) thick. In order to minimize the absorption of the recoil-free radiation within the disk, it was made of iron enriched to 99.9 percent in Fe^{56}.

The 14-kev excited state of Fe^{57} was obtained from the decay of Co^{57} by electron-capture. The long half-life (270 days) of this decay controls the intensity of radiation which is essentially constant for a period 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 disk; and the sample was annealed at 1000°C for 2 hours in an atmosphere of hydrogen after electroplating. A sample (*8*) with 4 mc of Co^{57} plated on the 0.007-inch (0.017-cm) edge along one-half of the circumference was used for the measurements reported here. Assuming a uniform distribution of Co^{57} within 0.005 inch (0.013 cm) from the edge, the cobalt concentration should be less than 0.1 percent.

The iron disk was contained between a set of Bridgman anvils, with face diameter of 0.250 inch (0.63 cm), by a ferric oxide coated ring of pyrophyllite 0.031 inch (0.079 cm) thick and 0.010 inch (0.025 cm) high. The Fe_2O_3 with which the ring was coated was enriched to contain 99.9-percent Fe^{56} to minimize absorption of the 14-kev radiation. 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 percent at any pressure.

No information is available about the distribution of pressure in an iron disk in the Bridgman anvils, so pressures reported are average pressures on the anvil face. Experience in an assembly of this shape with silver chloride as a pressure medium indicates that about only 82 percent of the area of the anvil face is load bearing and the average pressure on the disk is about 20-percent 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 disk with a circular section from the circumference and filling most of the volume with a well-studied pressure medium.

Difficulties in obtaining reproducible spectra were experienced with the constant-velocity spectrometer used in the Dy^{161} experiment. Therefore a velocity-sweep type Mössbauer spectrometer technique was developed for this experiment by modifying the low-temperature spectrometer design of Shirley *et al.* (*10*). The absorber was made with a 0.001-inch (0.0025-cm) thick foil of 19-9 stainless steel, enriched to contain 65-percent Fe^{57}. The foil has a broad single absorption so that the components of the Fe^{57} spectrum in iron could be resolved. The absorber was connected to a loudspeaker through the moving element of a Sanborn 6LV1 velocity transducer in order to make the transducer detect all the motion of the absorber relative to the press on which the stationary element of the transducer was mounted. The output voltage of the transducer was directly proportional to the velocity of the absorber relative to the source.

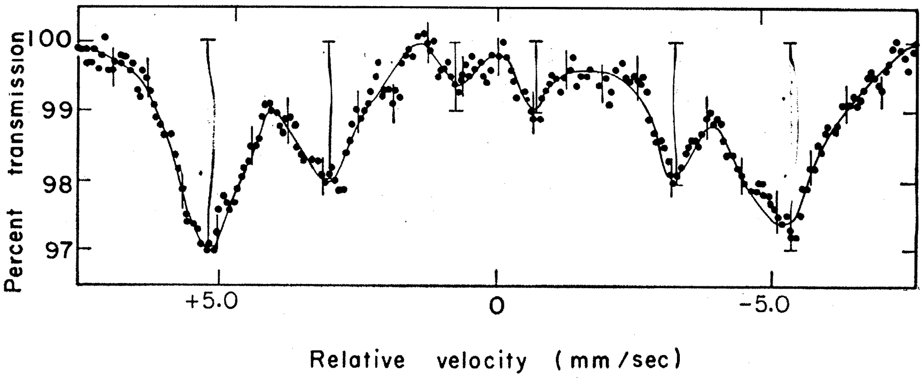
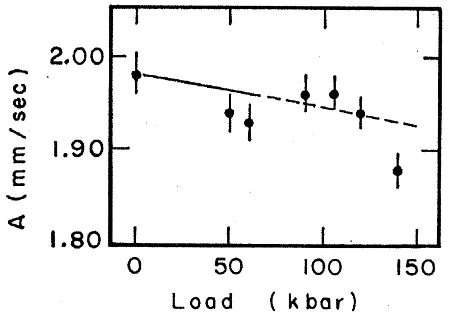


Fig. 1. Spectrum at 105 kbar, typical of the α-iron spectra. The statistical error is indicated on every tenth channel. The bars indicate the position and theoretical intensities of the absorption lines. The positions of the two inner lines are calculated.



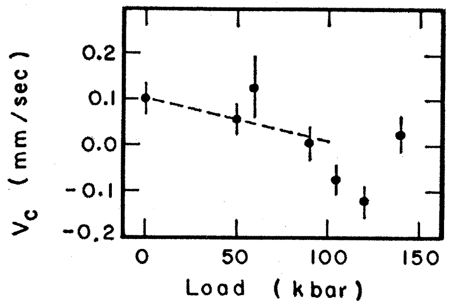


Fig. 2 (left). The variation of the magnetic splitting parameter with pressure. The straight line is calculated from the nuclear-magnetic-resonance results of Litster and Benedek (*12*). Fig. 3 (right). The variation of the chemical shift with pressure, compared with the results of Pound, Benedek, and Drever (*5*) (dashed line) obtained at lower pressures.

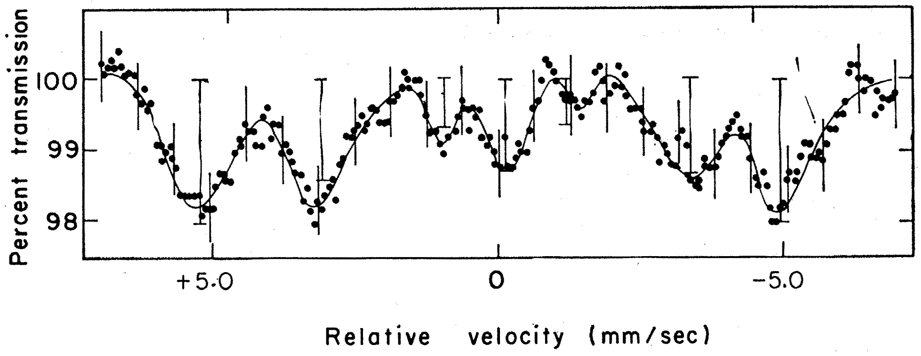


Fig. 4. Spectrum at 140 kbar showing seven lines. The statistical error is indicated on every tenth channel. The bars indicate the position and theoretical intensities of the absorption lines. The positions of lines 3 and 5 are calculated on the assumption of a six-line spectrum.

A thallium-activated sodium iodide crystal scintillation detector was mounted between the absorber and the loudspeaker to detect the radiation transmitted through the absorber. When a 14-kev γ-ray was detected, a square pulse proportional to the output of the velocity transducer was generated and fed into a 400-channel pulse-height analyzer. In this manner, a transmitted spectrum of photon intensity against photon energy was generated. Because a sinusoidal drive was used, this spectrum had to be normalized for variation in time elapsing in different velocity ranges. A typical normalized spectrum is shown in Fig. 1.

The spectrometer was calibrated with the 1-atmosphere spectrum from the data of Dash *et al.* (*11*). Then, pressure was applied to the sample. Detailed spectra were taken at loads of 50, 60, 90, 105, 120, and 140 kbar; the average pressure on the iron disk was probably 20-percent higher. At least two spectra were taken at each pressure; the average statistical error per channel was at most 0.5 percent of 100-percent transmission. The observed intensity of absorption decreased from 13 percent for the most intense peak of the uncovered sample at 1 atm to about 2 percent for the same peak in the 140-kbar spectrum. The decrease is due to attenuation of the recoil-free radiation by the ring relative to other radiation observed at the detector, 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 percent could be detected.

At pressures up to 120 kbar, these spectra consisted of six lines appropriate to Fe^{57} in ferromagnetic α-iron. In some of these spectra, weak inner lines were poorly resolved. The 105-kbar spectrum, shown in Fig. 1, is typical of these spectra. These spectra

Table 1. Magnetic and electric quadrupole splitting parameters defined by Eqs. 1 and 2, and the chemical shift for the Mössbauer spectrum of Fe^{57} in α-iron to 120 kbar.

Pressure (kbar)	A (mm/sec ± 0.02)	B (mm/sec ± 0.1)	v_c (mm/sec)
0.001 (1 atm)	1.97	0.0	+0.09 ± 0.03
50	1.93	.0	+0.06 ± .04
60	1.92	.0	+0.12 ± .06
90	1.95	.0	−0.01 ± .03
105	1.95	.0	−0.07 ± .03
120	1.93	.2	−0.11 ± .03

were analyzed in terms of three parameters: A, measuring the magnetic splitting as defined in Eq. 1; B, measuring the electric quadrupole splitting as defined by Eq. 2; and v_c, the velocity of the center of gravity of the spectrum with respect to the stainless-steel absorption. Here, μ_o is the magnetic moment of the ground state of Fe^{57},

$$A = -\mu_o H \qquad (1)$$

$$B = \tfrac{1}{4}\, e^2 qQ \qquad (2)$$

Q is 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 Eqs. 3–5. The velocity of the ith line numbered from the lowest energy line is v_i. The values

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

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

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

obtained from these parameters are given in Table 1. Figures 2 and 3, respectively, show A and v_c plotted as functions of pressure.

At 140 kbar (Fig. 4), a seventh line appeared in the spectrum near zero velocity (at -0.127 ± 0.064 mm sec^{-1}) 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^{-1}, $B = 0.1 \pm 0.1$ mm sec^{-1}, and $v_c = +0.025 \pm 0.035$ mm sec^{-1}. 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 are needed on this point. The positions of the two inner lines of the six-line spectrum calculated with these parameters agree quite well with the observed absorptions. The additional line can not be explained in terms of radiation of higher multipolarity from an α-iron lattice site. The seventh line is most easily explained in terms of a second iron-atom environment such as the high-pressure phase of iron.

The observed variation of the magnetic splitting is compared in Fig. 2 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 origin of this field has been discussed (*13*), and the pressure variation has been treated in terms of changes in volume and saturation magnetization (*14*). Because of the volume dependence, the pressure dependence of the magnetic field at the nucleus cannot be used alone to measure the dependence on pressure of the ferromagnetic coupling.

α-Iron is a cubic-lattice system for which electric-field gradients at the nucleus are prohibited by symmetry for the undistorted lattice. Observation of nonzero values of B at the higher pressures achieved can be explained in two ways. A pressure gradient, such as that observed in silver chloride in the Bridgman anvils, might distort the lattice sufficiently for the observed splitting. The distortion should increase gradually with pressure and might be obscured at low 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 (*15*), also could produce quadrupole splitting.

The variation of the chemical shift with pressure is shown in Fig. 3 together with an indication of the variation obtained (*5*) 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 4*s* electron density at the nucleus has been discussed by Walker, Wertheim, and Jaccarino (*16*). According to their Fig. 1, 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 to 10 percent in the 4*s* electron density at the nucleus for a $3d^{8-x}\,4s^{x}$ electron configuration. This corresponds closely to the decrease in the volume of the iron lattice at 130 kbar observed in the dynamic high-pressure investigation of Bancroft *et al.* (*2*).

The seventh line observed in the spectrum at 140 kbar is not at a position of a 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 phase, which has been observed in both static and dynamic high-pressure work (*2, 3, 15*), would be such a second environment. Jamieson and Lawson have suggested that this phase is hexagonal close packed from x-ray measurements (*15*). In static high-pressure experiments (*2, 15*), 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 of these phases has been observed at 170 kbar in x-ray studies by Jamieson (*17*).

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 (*18*).

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Ion-Exchange Removal of Sodium Chloride from Water with Calcium Hydroxide as Recoverable Regenerant

Abstract. *A mixture of cation- and anion-exchange resins, in proportions corresponding to their particular exchange properties and charged with calcium hydroxide, removes seawater salts from solution exchanging calcium hydroxide for the sodium chloride. The regenerant is recoverable. A portion of it may be used again without handling.*

Ion-exchange resins have been seriously considered for desalting seawater (*1*). The prevalent opinion is that large-scale use of such resins depends on finding a suitable regenerating agent.

Such a regenerant should be safe and easy to handle; its deleterious effects on the resin should be nearly negligible; its affinity for the resin should allow the desalting to proceed at a reasonable rate; it should be readily available; and its cost should be low. It must be taken up by the resin from solutions of sodium in preference to that ion, and yet be held lightly enough to permit exchange for sodium from solutions in which it is present at concentrations lower than those of equilibrium, in terms of relative affinities. It must also be recoverable at low cost.

Of the recoverable regenerants investigated, thermolabile salts, as represented by ammonium carbonates, were unsuitable owing to the high cost of reclaiming them (*2*). For economical recovery, low solubility in water would allow the regenerant of the ion-exchange system to precipitate and to be used over and over again for column regeneration. The solubility of calcium hydroxide at prevalent temperatures is low enough for a reasonably direct recovery of the regenerant during the desalting run [92 percent of regenerant at $Ca(OH)_2$ solubility of 1.65×10^{-1}], as well as for recovery of the excess regenerant used during the regeneration cycle. The precipitate is removed by simple mechanical means.

Lime slurries have been used for regeneration of anion-exchange resins (*3*), of cation-exchange resins (*4*), and for the simultaneous regeneration of both the cation- and anion-exchange resins in mixed systems (*5*). The direct taking up of solids by ion-exchange resins is described by Deuel and Krishnamoorthy (*6*).

The percentage of removal of an electrolyte from its solution by exchange for another pair of ions can be calculated with reasonable accuracy; the exchange is stoichiometric (*7*). Thus, when the electrolyte content of seawater is exchanged for Ca^{++} and OH^- ions, the resulting medium contains 2.215-percent $Ca(OH)_2$.

We have used calcium hydroxide as a recoverable regenerant for the removal by ion-exchange of sodium chloride from its solutions at concentrations slightly higher than those encountered in seawater.

In a simplified fashion we have demonstrated the basic principle of removal of sodium chloride from its solutions by replacement with calcium hydroxide, and the feasibility of regenerating a mixed bed in the sodium and chloride form by a slurry of calcium hydroxide in a 3.5-percent solution of sodium chloride. Also, we have shown that the deposited regenerant may be used directly to regenerate the ion-exchange system.

We have examined only the exchange of sodium chloride for calcium hydroxide. In the demineralization of seawater, other cations present (calcium, magnesium, and so forth) would be taken up by the resin before or together with the sodium ion, and hence would affect the process only insofar as available active resin sites are concerned. Because of the higher affinity of the resin for these ions, the useful volume of the column, if seawater were used, would be somewhat bigger.

Water suitable for drinking, irrigation, or industrial uses may be attained by one of several techniques. For example, spraying through the air will precipitate some calcium carbonate; ion exchange of remaining $Ca(OH)_2$ for sodium chloride will yield utility water containing 0.25-percent NaCl; ion exchange for the Cl ion will give water with a 0.25-percent $CaCl_2$ content; or, ion exchange of the calcium ion for another cation will precipitate an insoluble hydroxide.

For demineralization of water, we used a 150-cm Lucite column, with a 2.5-cm inside diameter, both ends of which were fitted with stainless-steel housings having dacron filters, needle valves as outlets, and capped vents without valves. The useful volume was 740 ml.

The ion-exchange resins in the column were a mixture of 471 ml of 12-percent divinylbenzene cross-linked polystyrene sulfonic cation-exchange resin with 269 ml of trialkylalkanolammonium polystyrene anion-exchange resin.

The cation-exchange resin was converted to the calcium form and the anion-exchange resin to the hydroxyl form, by downward passage of water of lime. The column was then rinsed with distilled water and left full.

A 3.62-percent solution of technical sodium chloride was fed into the column under gravity at a flow rate of 10

Table 1. Salt removal from 3.62-percent NaCl solution on column of mixed ion-exchange resin in Ca^{++} and OH^- form.

Effluent (ml)	NaCl Concentration (%)	 Total removal (meq)
100 to 500	0.00	312.05
600	0.00	374.46
700	0.05	435.99
800	0.6	488.05
900	1.1	530.64
1000	1.3	570.64
1100	1.6	605.53
1500	2.45	699.8
2000	2.85	779.3
2250	3.1	805.2