may play a role in the radiation-induced recovery.

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X-RAY MEASUREMENT OF THE DISTRIBUTION OF ELECTRONS IN IRON AND COPPER

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Batterman[1] has recently reported some x-ray measurements on powdered Fe and Cu in which he concludes that the number of 3*d* electrons in Fe is 6.2 ± 0.4 measured relative to copper (assumed to have ten 3*d* electrons) and is 6.9 ± 1.0 relative to a NaCl standard. Simultaneously with reporting this he has made available to us samples of Fe and Cu powder which he used in making these determinations and we are grateful for his consideration.

We have repeated his measurements and made several of our own and while we find no major differences in the raw data, our analysis of the data fixes the number of 3*d* electrons in Fe at approximately 2.5 ± 1.5. The differences in the analyses undoubtedly arise from factors such as absorption coefficients, Hönl corrections, Debye-Waller factors, extinction corrections, and packing effects which cannot all be evaluated until Batterman reports these in detail. However, some of these differences can be discussed.

Firstly, Batterman has used tabulated Hönl corrections whereas we have measured these and find them to be -1.9 for Fe and -1.84 for Cu (Fe $K\alpha$). These have been measured by comparing the intensity ratio of the Fe 211 and Cu 311 peaks to an Al 311 peak at both Mo $K\alpha$ and Fe $K\alpha$. In this comparison the Debye-Waller factors, preferred orientation, and any uncertainty in the scattering factors approximately cancel out and the only major difference in the ratios at the two wavelengths is the Hönl correction at Fe $K\alpha$ (the correction is small at Mo $K\alpha$). Secondly, we have found large packing density differences in the integrated intensities of Fe between compressed and loose-packed samples (~25%) and believe this to be the case for NaCl since its linear absorption coefficient is not much smaller than that of Fe. Unfortunately the onset of preferred orientation in compressed NaCl prevents a direct measurement, but an indication that such an effect is present can be obtained by comparing the total integrated intensities of all the peaks in a compressed and loose-packed sample. If only preferred orientation sets in on compression, then intensity lost from some peaks will be gained by others. We have done this and find a 45% increase in the compressed sample. While some peaks are unchanged on compression, this is no doubt due to preferred orientation and packing effects cancelling each other. The precise reason for this effect is not understood as we have also observed this effect with more penetrating radiation (Mo $K\alpha$) on samples of V, Fe, and α brass. We believe that this effect may be related to the packing density since it is independent of angle. Hence the use of loose-packed NaCl to standardize compressed Fe and Cu samples will be in error. Thirdly, we find at least 12% extinction in Batterman's Cu sample as determined from the 111/222 ratio (this is free of preferred orientation). Of course this depends on the Debye temperature used (we used $\theta = 320°$),[2] and on precisely how one draws the background since the 222 peak is broad. In order to circumvent these uncertainties, we have standardized his Cu 111 peak against the 311 peak of compressed 325-mesh Al filings which were free of extinction on compression and exhibited little preferred orientation. We found his Cu 111 peak to be (12 ± 4)% low which we believe supports our contention of extinction.

If we combine the 12% extinction correction with our measured Hönl correction, then Batterman's measured Fe 110 scattering factor is reduced from 18.9 (relative to copper) to 17.1 or, in terms of 3*d* electrons, from 6.2 to 2.8. (As a result of the changed 3*d* electron configuration a correction of ~0.5 3*d* electron has been made due to the argon core contraction.[3])

As an independent check, we have measured the Fe 110 peak relative to the above-mentioned Al standard and obtained $f = 17.0 \pm 0.9$ with Fe $K\alpha$ and 17.0 ± 0.9 with Mo $K\alpha$. These measurements correspond to 2.5 ± 1.5 3*d* electrons.

While the numbers we quote result from a straightforward analysis of the data, we believe that powder measurements can be taken seriously only when all possible sources of error are understood, and to date we do not believe this to be the case.

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[3] D. R. Hartree (private communication).

INVERSION OF PARAMAGNETIC RESONANCE LINES IN IRRADIATED CALCITE*

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Spin-lattice relaxation times as long as three minutes have been observed for radiation-induced paramagnetic resonance lines in calcite at 1.6°K. At 77°K the corresponding time is 5 milliseconds.

Resonance was observed with a conventional superheterodyne bridge spectrometer operating at 9 kMc/sec.[1] Samples were cut from reasonably clear natural calcite crystals and ground to fit rectangular X-band waveguide. Their lengths were adjusted to be resonant at the desired frequency; no iris was used.

Irradiations were performed with 3-Mev electrons. Measurements on one crystal indicated that a dose of 60 microcoulombs/cm^2, administered in two separated 30-second periods, produced $\sim 10^{17}$ paramagnetic centers/cm^3 of the type which contributed to the strongest line. This spin density was measured by comparison with a crystal of copper-doped zinc Tutton's salt of known copper concentration. The above dosage rate caused the crystal to heat appreciably, and lower rates were found to be more efficient in producing centers. The centers fade in a few hours at room temperature and in a few days at liquid nitrogen temperature.

Several of the crystals, both before and after irradiation, exhibited a number of relatively weak lines which were identified as the spectrum of Mn^{++}, present as an impurity in the calcite. The positions and intensities of these lines agreed exactly with the work of Hurd et al.,[2] and served as a convenient calibration for the radiation-induced lines.

At least seven additional lines appeared as a result of irradiation. These are listed in Table I for a typical run on a crystal cut so that its optic axis is parallel to the static magnetic field ($\theta = 0°$). All Mn^{++} lines would be classified as "weak" or "very weak" on the scale of relative intensities used in the table. The strongest line (3271 gauss) exhibits a g value of 2.00, constant with changing radio-frequency and nearly constant with changing θ. The other lines change considerably with both frequency and θ.

Inversion of the various lines has been accomplished by adiabatic fast passage, using both field and frequency sweeps. Spin-lattice relaxation times (T_1) were measured by observing the rate of decay of the inverted absorption signal back toward its equilibrium value. Measured T_1 values at 1.6°K for five of the irradiation-induced lines are listed in Table I. Inversion of the other lines in the table was not attempted. An effort to invert a few of the larger Mn^{++} lines was unsuccessful, indicated $T_1 < 1$ second for these lines.

The relaxation times shown in Table I are probably somewhat smaller than the true values, for two reasons. First, the strength of the lines is so large that it is extremely difficult to avoid saturating the lines with the microwave power ($< 10^{-12}$ watt) used for observation. Further reduction of this power was impossible because of system noise, originating principally in the detector. Second, the inverted spin system reduced the loaded Q of the cavity appreciably, causing regeneration and an artificially rapid decay. This effect was minimized for T_1 measurements by using low-Q cavity modes and by

Table I. Resonance lines caused by irradiation in CaCO$_3$. Radio-frequency 9155 Mc/sec, $\theta = 0°$.[a]

Relative intensity	Magnetic field (gauss)	T_1 at 1.6°K (min)
Strong	4620	3
Medium	3950	
Strong	3406	1
Weak	3288	
Very strong	3271	1
Weak	3245	2
Strong	2410	3

[a] The first, second, and last lines in the table were outside the range of the proton resonance magnetometer. Magnetic fields for these lines were estimated from the magnet current readings.