

### Paramagnetic Resonance Absorption in Aqueous Solutions of Manganese Sulfate\*

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 October 15, 1948

PARAMAGNETIC resonance absorption has been observed in aqueous solutions of manganese sulfate<sup>1</sup> contained in thin-walled glass tubes placed along the axis of a cylindrical cavity oscillating in the  $TE_{001}$  mode at 9375 mc/sec. Because the electric field is zero along the axis of this cavity,<sup>2</sup> electric losses in the sample are not large enough to prevent observation of the magnetic losses. As in previous experiments,<sup>3</sup> the experimental arrangement consisted of a square-wave modulated, low power oscillator, part of the power from which passed through the resonant cavity and was absorbed by a detecting crystal. The voltage output of this crystal was bucked against that of another crystal whose output voltage was proportional to the power output of the oscillator. The output of the bucking-out circuit was, as before, amplified, rectified, and then compared with a stable d.c. voltage, the difference between the two being indicated as a galvanometer deflection. It can be shown that these deflections are proportional to  $\chi''$ , the imaginary part of the high frequency susceptibility of the sample.

Resonance absorption has been observed in manganese sulfate solutions for various concentrations, a typical curve being that shown in Fig. 1. As concentration increases, the half-width of the absorption curve also increases. The value of the magnetic field for maximum absorption decreases as the concentration of manganese ions in the water solution increases. The magnetic field was calibrated against the proton moment and the magnetic induction for maximum absorption as shown in Fig. 1

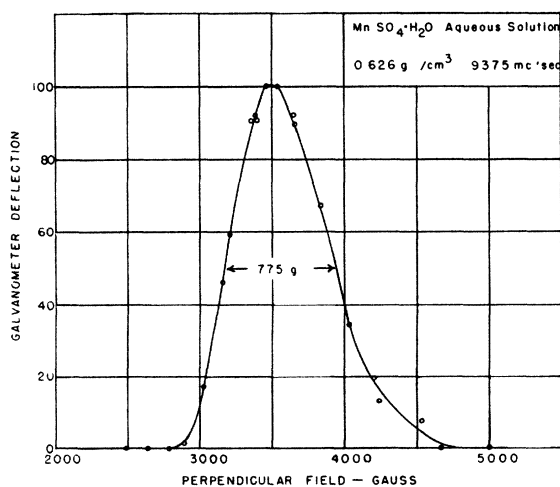


FIG. 1. Relative values of  $\chi''$  for an aqueous solution of manganese sulfate at 9375 mc/sec. and 25°C.

is believed to be accurate to  $\pm 15$  gauss. Measurements are being continued with apparatus of greater sensitivity.

We wish to acknowledge the continued assistance and encouragement of Dr. A. J. Allen.

\* Publication number 6L-48. Assisted by the Joint Program of the Office of Naval Research and the Atomic Energy Commission.

<sup>1</sup> D. Halliday and J. Wheatley, *Bull. Am. Phys. Soc.* **23**, 13 (1948).

<sup>2</sup> Collie, Hasted, and Ritson, *Proc. Phys. Soc.* **60**, 71 (1948).

<sup>3</sup> Cummerow, Halliday, and Moore, *Phys. Rev.* **72**, 1233 (1947).

### A New Naturally Occurring Isotope of Dysprosium

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 October 14, 1948

THE isotopic constitution of dysprosium was first studied by Aston<sup>1</sup> who found isotopes at masses 161, 162, 163, and 164. Later, Dempster,<sup>2</sup> working with a spark source, discovered the rare isotopes at masses 158 and 160. This paper reports work leading to the discovery of an additional rare isotope at mass 156.

Three separate chemical preparations of dysprosium oxide were studied with a mass spectrometer using techniques previously described.<sup>3</sup> All three samples showed ion currents at mass 156, which could be explained only by postulating a previously undetected naturally occurring isotope in dysprosium of mass 156 present to about 0.05 percent of the total.

The first sample investigated was prepared by Dr. J. K. Marsh of Oxford. The optical spectrographic analysis, supplied with the sample, indicated the presence of praseodymium, gadolinium, terbium, and holmium in detectable quantities. When this material was placed on the filament of a surface ionization type of mass spectrometer source, it gave currents corresponding to the metallic and metallic oxide ions of the known isotopes of dysprosium and a number of weaker currents corresponding to impurities of cerium, lanthanum, europium, samarium, and ytterbium which were present as impurities in addition to those optically detected. However, in spite of the known presence of all these impurities and consideration of other possible impurities, the peak observed at mass 156 in the metallic ion group could not be explained. It could conceivably have been due to  $Gd^{156}$ ,  $Ce^{140}O^{16}$ ,  $La^{139}O^{17}$ , or  $Ba^{138}O^{18}$ . Gadolinium was ruled out since the neighboring isotope at mass 157 was many times too weak to account for the 156 observed. Lanthanum was ruled out since the neighboring isotope at mass 155 was likewise too weak to account for the 156 observed. Ba was ruled out since it does not emit as the oxide. Cerium, however, could not be easily ruled out since it emits only as the oxide and since its other strong peak is masked by  $Dy^{158}$ . In order to study this possibility, it was necessary to utilize the fact that the ion emission from a surface ionization source varies from element to element with time and temperature. When the ion currents were observed continually as the temperature of the emitting surface was raised, the im-

purity current corresponding to mass 156 decreased rapidly from 0.3 percent to 0.05 percent of the total dysprosium content and thereafter remained constant. This indicated that the residual peak of 0.05 percent was not  $Ce^{140}O^{16}$ , and that it was probably due to a previously undetected isotope of dysprosium of mass 156. It does not, however, rule out the faint possibility that the cerium fractionates to a fixed ratio with the dysprosium and thereafter remains constant. Study of the 156 position in the oxide group of ions with this sample was not possible due to the lower intensity of this group and to the presence of gadolinium and ytterbium impurities.

In order to check further the possibility that the cerium impurity might be the cause of the current at mass 156, a second sample of dysprosium oxide was obtained from Dr. E. R. Tompkins of Oak Ridge. This sample had been prepared in an ion exchange column. With this method of purification, impurities are expected to be only the adjacent elements. The optical analysis supplied with the material showed impurities of terbium and gadolinium as "weak" and erbium as "very weak." The mass spectrometer analysis showed in addition ion current corresponding to holmium, lanthanum, and samarium. With this sample, the 156 peak appeared initially at 0.05 percent and remained constant throughout the run. However, as with the previous sample, it was not possible to obtain the additional verification of the isotope by observing the  $Dy^{160}O^{16}$  position because of the gadolinium impurity.

A final sample of much higher purity prepared by the ion exchange process was obtained from Dr. D. H. Harris of Oak Ridge. No rare earth ions other than the known dysprosiums and the new peak at mass 156 were detectable to greater than one part in seventy thousand. Again with this sample the ion current appeared initially at 0.05 percent and did not change detectably with time or temperature. It was possible with this sample to observe the  $Dy^{160}O^{16}$  position free of impurities. The abundance was again about 0.05 percent of the total dysprosium isotopes.

We therefore conclude that dysprosium has a previously unknown isotope of mass 156 which is present to about 0.05 percent of the total. A future publication will give more accurate data on the abundance of this rare isotope and new values for the abundances of the remaining isotopes.

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<sup>2</sup> A. J. Dempster, Phys. Rev. **53**, 727 (1938).

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### The Gamma-Ray Spectrum from $F^{19}+H^1$ Using Photographic Emulsions Containing $D_2O$

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October 13, 1948

ONE method of measuring gamma-ray energies is to observe the range in photographic emulsions of the protons from the photo-disintegration of deuterium. The

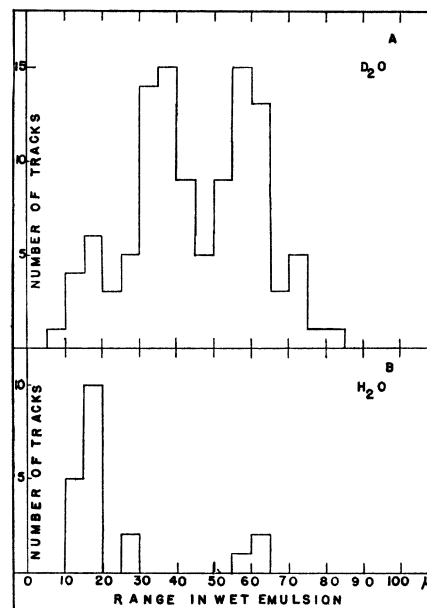


FIG. 1. (A) Emulsion A. Histogram of 109 tracks found on 26 mm<sup>2</sup> of nuclear emulsion exposed to  $F^{19}(p, \gamma)O^{16}$  gamma-rays while wet with  $D_2O$ ,  $E_p = 2.6$  Mev. (B) Emulsion B. Histogram of 20 tracks found on 16 mm<sup>2</sup> of nuclear emulsion exposed together with emulsion A while wet with  $H_2O$  (control emulsion).

major problem of the method is to load the emulsions with sufficient deuterium. Gibson, Green, and Livesey<sup>1</sup> have realized 6 percent  $D_2O$  by weight by using emulsions loaded with  $Ca(NO_3)_2$  and  $D_2O$  as water of crystallization. In the present investigation, by soaking Ilford (C2200 $\mu$ , stripped) nuclear emulsions in  $D_2O$ , from 30- to 80-percent  $D_2O$  by weight can be introduced into the emulsions.

Emulsions containing approximately 50-percent  $D_2O$  were exposed while wet to  $F^{19}(p, \gamma)O^{16}$  gamma-rays obtained by bombarding a thick  $CaF_2$  target with 2.6-Mev protons from the Wisconsin electrostatic generator. The maximum exposure that could be tolerated before background gamma-ray fog interfered with the track measurements was 0.2- $\mu$  ampere hours.

Two emulsions were exposed together; emulsion A was soaked in  $D_2O$  and emulsion B (control emulsion) in  $H_2O$  for 5 minutes. They were then placed perpendicular to the proton beam, at a distance of 6 and 8 mm, respectively, from the target, and with 1.8 g per cm<sup>2</sup> of intervening material.

As a provisional range energy relation in wet emulsions, the "range in wet emulsions ( $R_w$ )" was converted to "equivalent range in dry emulsions ( $R_d$ )" by taking  $R_d = 0.93R_w$ . This is based on a rough calibration with  $d-d$  neutrons at  $E_n = 4.25$  Mev. The range energy relation given by Lattes, Fowler, and Cuer<sup>2</sup> was then used for  $R_d$ .

**Results.** On emulsion A and B 26 and 16 mm<sup>2</sup>, respectively, were viewed in the microscope, and 109 and 20 tracks, respectively, were measured (Fig. 1A and 1B).

All tracks found with angle of dip  $\alpha < 50^\circ$  were measured. Corrections (factor  $\sec \alpha$ ) being applied to those tracks