W. D. Metz assisted in data acquisition and reduction. Joseph Berti designed much of the apparatus, constructed in our shop by A. Comeau and J. Cimino, while Miss Kathy Lappert provided many excellent drawings. Dependable operations of the accelerator were provided by P. Jewett, W. Knudsen, S. Clow, R. Rondoe, M. Garfield, and F. Hegedus. Finally, our thanks go to Professor Howard Schultz, director of the Electron Accelerator Laboratory, for his continuing interest in the electron scattering project.

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# Magnetic Moment of Sm145 and Attenuation Following the Decay of Oriented Sm145*

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Samarium-145 nuclei were oriented at low temperatures in neodymium ethylsulfate (NES) and cerium magnesium nitrate (CMN) lattices. From the temperature-dependent angular distribution of the 61-keV $\gamma$ ray in Pm145, a magnetic moment $\mu = 0.92 \pm 0.06 \mu_N$ was deduced for the ground state of Sm145. The ratio of attenuation coefficients in the 61-keV state of Pm145 in the two lattices was found to be $G_2(\text{CMN})/G_2(\text{NES}) = +0.44(10)$. Evidence is presented which indicates that temperature gradients in CMN can lead to erroneously low values for magnetic moments.

## INTRODUCTION

RADIOACTIVE isotopes of most of the rare-earth elements have been oriented in lattices of neodymium ethylsulfate $\text{Nd}(\text{C}_2\text{H}_5\text{SO}_4)_3 \cdot 9\text{H}_2\text{O}$ or of cerium magnesium nitrate $\text{Ce}_2\text{Mg}_3(\text{NO}_3)_{12} \cdot 24\text{H}_2\text{O}$.[1] In a systematic survey of oriented rare-earth nuclei in these lattices started in 1960, we obtained results for Sm145 that we could not explain. The magnetic moments derived from studies in the CMN and NES lattices were apparently quite different. While some of the earlier work in the field of nuclear orientation showed discrepancies of this kind, most of these could be attributed to poor experimental technique and would dispapear when the work was done more carefully. Our Sm145 results were checked for all the known sources of error, however, and refinement of technique only confirmed them. With the recent discovery that the temperature scales in use for both NES and CMN were seriously in error, the Sm145 data were corrected to the new scales,[2,3] and the magnetic moments derived from the two salts are now in excellent agreement. The determination of this magnetic moment is described here.

Having fitted the nuclear orientation data to derive moments, one also obtains, for each lattice, $A_2$, the coefficient of $P_2(\cos\theta)$ in the $\gamma$-ray angular distribution function. This $A_2$ may have the full value $B_2 U_2 F_2$ implied by angular momentum theory alone, or it may be attenuated by a factor $G_2$ which describes reorientation in an intermediate state. For this case (i.e., the 61-keV, $t_{1/2} = 2.6$-nsec state[4] of Pm145), we have found substantial attenuation in the CMN lattice, as described below.

## EXPERIMENTAL

The Sm145 activity was prepared by neutron irradiation of enriched Sm144, followed by ion-exchange separation from other rare-earth contaminants, notably (daughter) Pm145, immediately before use. The Pm145/Sm145 $\gamma$-ray intensity ratio was easily kept below 1%. Because the Pm145 $\gamma$ rays were shown in separate experiments to be essentially isotropic, no correction was necessary.

The heavier rare earths grow substitutionally into the CMN lattice poorly, the difficulty increasing with $Z$.

[1] D. A. Shirley, Ann. Rev. Nucl. Sci. **16**, 89 (1966).

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[3] J. Blok, D. A. Shirley, and N. J. Stone, Phys. Rev. **143**, 78 (1966).

[4] A. R. Brosi, B. H. Ketelle, H. C. Thomas, and R. J. Kerr, Phys. Rev. **113**, 239 (1959).
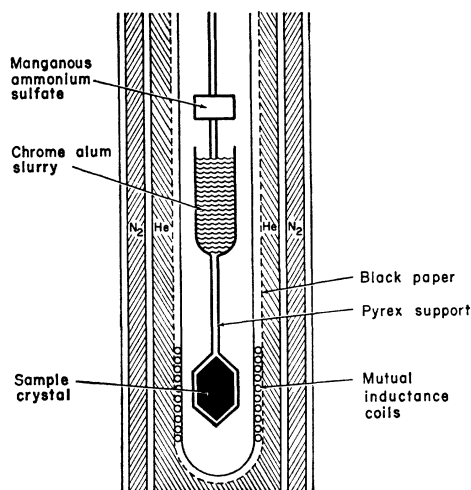
FIG. 1. The cryostat.

Although samarium is known to go into the double nitrate lattice,[5] the complete analysis of a nuclear orientation experiment requires that nearly all of the activity "in the crystal" should, in fact, be substitutionally in the trivalent lattice sites. Most of the CMN crystals that were used in this work were quite clear, though none was completely clear. There was no tendency for $\gamma$-ray anisotropy to increase with crystal clarity (as would be the case if significant fractions of the activity were present as inclusions). A discrimination factor of only 10:1 against Sm going into CMN was determined in a separate experiment. Thus, it seems unlikely that the low value of $A_2$ reported below
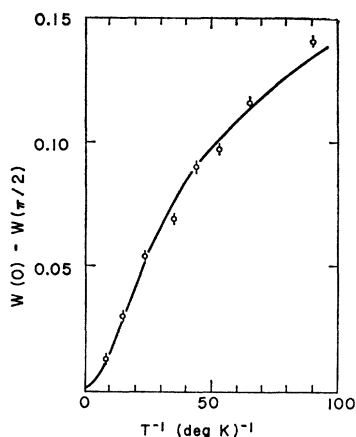


FIG. 2. Plot of $W(0) - W(\tfrac{1}{2}\pi)$ for the 61-keV $\gamma$ ray of Pm$^{145}$ following the decay of Sm$^{145}$ oriented in an NES crystal. The theoretical curve is shown.

[5] A. H. Cooke and H. J. Duffus, Proc. Roy. Soc. (London) A229, 407 (1955).

can be attributed to Sm$^{145}$ that is in the sample but not oriented in lattice sites.

The apparatus has been briefly described elsewhere.[6] It seems worthwhile, however, to discuss here in some detail the factors relating to a very important parameter in nuclear orientation experiments: the heat leak.

In most cryogenic experiments involving adiabatic demagnetization, it is possible, by isolating the specimen and employing a paramagnetic salt with a large heat capacity, to reduce the heat leak into the specimen to negligible proportions. Heat leaks as low as 1 erg/min have been reported, although 100 erg/min is a more common figure.

For single-crystal nuclear orientation work many of the devices used to reduce the heat leak in other experiments are automatically excluded, and except in special cases one is forced to think in terms of a total "warm-up
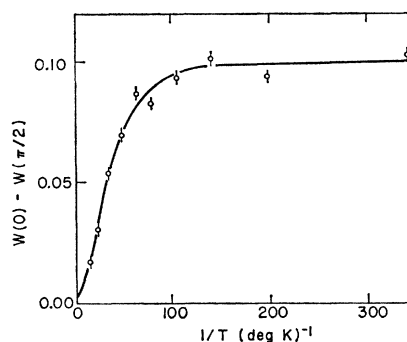


FIG. 3. Plot of $W(0) - W(\tfrac{1}{2}\pi)$ for the 61-keV $\gamma$ ray of Pm$^{145}$ following the decay of Sm$^{145}$ oriented in a CNM crystal. The theoretical curve is shown.

time" (i.e., the time required, after demagnetization, for the specimen to reach the temperature of the helium bath). If one wishes to determine radiation distributions with any reasonable accuracy, it is essential that the warm-up time be much longer than the counting interval. Inasmuch as meaningful angular distributions may be obtained only when the entire specimen is at a nearly uniform temperature(i.e., soon after demagnetization), this impies that warm-up times of many minutes, or preferably several hours, are highly desirable.

In order to produce warm-up times of 1–2 h in a CMN crystal, or $\sim$8 h in an NES crystal, in a simple apparatus with which it is possible to go from room temperature to demagnetization temperatures in less than 2 h, we have developed the design shown in Fig. 1. At the top (not shown) is a brass cap, silver-soldered to a tube (with light traps to prevent direct radiation from above) which acts as a vacuum line and support. A

[6] C. E. Johnson, J. F. Schooley, and D. A. Shirley, Phys. Rev. 120, 2108 (1960).

short tungsten rod is silver-soldered to the inside of the cap, and a 2-mm glass rod is sealed to its lower end. This glass rod is about 30 cm long and supports a compressed salt pill of $Mn(NH_4)_2(SO_4)_2 \cdot 6H_2O$, which has a high magnetic heat capacity and cools, in the stray field of the electromagnet, to about 0.17°K, where its high surface area adsorbs gas leaking down into the chamber. Below this is a slurry of $Cr_2K_2(SO_4)_2 \cdot 12H_2O$ and glycerine, which cools to ~0.01°K and acts as a sink for heat conducted down the glass supported rod as well as a second adsorbent for gas. Finally, the CMN crystal is attached to a glass rod framework with Duco cement. The experimental chamber consists of a glass tube, sealed at the bottom, jointed to a short copper tube at the top with a Housekeeper seal.

Assembly of the apparatus consist of attaching the crystal-salt assembly, soldering the chamber on at the top, painting the chamber with Aquadag (colloidal graphite), mounting the inductance coils, and wrapping

TABLE I. Summary of results.

| Lattice | hfs const (cm⁻¹) | $\mu_{145}/\mu_{147}$ᵃ | $G_2U_2F_2$ |
|---------|------------------|------------------------|-------------|
| NES | $B=0.028(3)$ | 1.12(11) | +0.127(20) |
| CMN | $A=0.040(4)$ | 1.15(12) | +0.055(8) |

ᵃ Using $\mu_{147}=0.812\mu_N$, we have $\mu_{145}=0.92(6)\mu_N$.

it all in black paper as a precaution against radiation leaks through pinholes in the Aquadag. With this apparatus, warm-up times are typically in the 80–100-min range for a 5–10-g crystal of CMN znd ~10 h for NES.

Temperatures were determined by measuring the susceptibility of the (NES or CMN) crystal and correlating this with the magnetic field and temperature from which demagnetization was done, using the known $T$-$S$ relations.[2,3] Data for NES and CMN are shown in Figs. 2 and 3. The 61-keV γ ray was detected at 0° and 90° from the trigonal axis by 3×3-in. NaI(Tl) detectors. Care was taken to insure that data were recorded only when the crystals were at uniform temperatures, i.e., immediately after demagnetization. Thus each experimental point represents a complete demagnetization run. In Fig. 4 data taken in this way are contrasted to those taken as a CMN crystal warms up from the lowest temperature, the latter data being erroneously low.

In the CMN runs the saturation values of $W(\theta=0)$ and $W(\theta=\frac{1}{2}\pi)$ were never quite consistent with a $P_2(\cos\theta)$ distribution, the function $W(0)-1$ being slightly larger than the expected $2[1-W(\frac{1}{2}\pi)]$. This
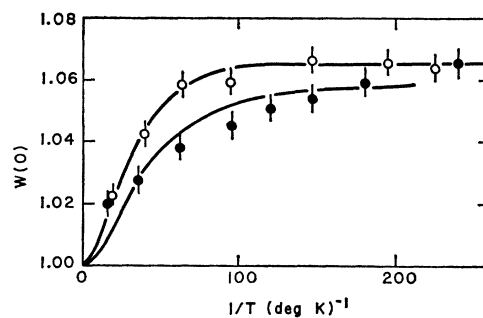


FIG. 4. Intensity along the $c$ axis of the 61-keV γ ray following the decay of Sm¹⁴⁵ oriented in CMN. Open circles were determined with the crystal at a uniform temperature. Closed circles represent data obtained as the crystal warmed up: These data give a magnetic moment that is 20% low.

effect, which was scarcely outside of experimental error, was originally attributed to scattering by the CMN crystals, which were typially 3×20×20 mm, the short dimension being along the $c$ axis. Extensive tests did not confirm a scattering mechanism, however, and the small deviation remains puzzling.

## DISCUSSION

The spin Hamiltonian relevant to this work is

$$\mathfrak{IC} = AS_zI_z + B(S_xI_x + S_yI_y).$$

In the double nitrate lattice we have $A>B$. For Sm¹⁴⁷ in samarium magnesium nitrate,[5] $A=0.0346(5)$ cm⁻¹ and $B\leq0.010$ cm⁻¹. In the ethylsufate lattice $B>A$: For Sm¹⁴⁷ in lanthanum ethylsulfate,[7] $A=0.0060(1)$ cm⁻¹ and $B=0.0251(1)$ cm⁻¹. For the lowest electronic state of Sm³⁺ in both lattices $S=\frac{1}{2}$. The nuclear spin of Sm¹⁴⁷ is[5,7] $\frac{7}{2}$: For Sm¹⁴⁵ spin-$\frac{7}{2}$ is the most probable assignment.[4] Thus rather direct comparisons of $A$ and $B$


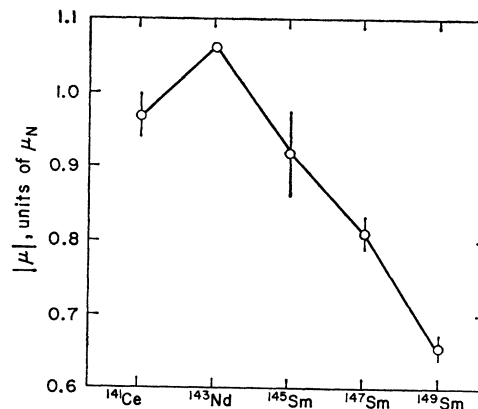
FIG. 5. Magnetic moments of Sm¹⁴⁵ and related nuclei.

[7] G. S. Bogle and H. E. D. Scovil, Proc. Phys. Soc. (London) A65, 368 (1952).

for the two isotopes is possible. The ratio $A/B$ is of course the same for both isotopes in a given lattice. In calculating the temperature dependence of the orientation parameters $B_k$,[8] we therefore have only one independent variable to determine for each lattice.

In this work the angular distributions could be described to first approximation[9] by functions of the form

$$W(\theta) = 1 + A_2 P_2(\cos\theta),$$

where $A_2$ can be written $A_2 = B_2 U_2 G_2 g_2 F_2$.[10] The solid-angle factor $g_2$ is easily calculated, and $B_2$ may be determined from the temperature dependence of $A_2$ and divided out. Reorientation during electron-capture decay is described by $U_2$. This reorientation may be calculated in principle, but as the details of the decay are not known, we can only set the limits in $0.466 \leq U_2 \leq 1.000$, on the basis of the values 1.000, 0.810, and 0.466 for 0, 1, and 2 units of angular momentum in the lepton system. A value of $U_2 = 0.9 \pm 0.1$ is very likely. The value of $F_2$ is also *a priori* unknown, because it depends on the $E2/M1$ mixing ratio of the 61-keV $\gamma$ ray. In practice, then, we can determine $G_2 U_2 F_2$ for each lattice, and derive the ratio of attenuation factors $G_2$: The results of this analysis are set out in Table I.

The excellent agreement of the values of the magnetic moment derived from the two lattice supports the usefulness of the CMN lattice for magnetic-moment determinations. In the past, the magnetic moments derived from nuclear orientation measurements in this lattice have almost invariably been erroneously small. In our opinion, this has been due to the lack of a reliable temperature scale, to attenuation in intermediate states,

and to high heat leaks. By minimizing the heat leak it should now be possible, at least in cases for which the orientation can be saturated, to determine nuclear moments reliably.

The Sm[145] nuclear moments might be expected to follow either of two empirical trends, that of 83-neutron nuclei or that of samarium isotopes. The actual trend is shown in Fig. 5. It is possible that the $A$ dependence of $\mu$ is smoother than this figure suggests, and perhaps even monotonic, because $\mu(\text{Ce}^{141})$ is based on an extrapolated value of $\langle r^{-3} \rangle$ for $4f$ electrons.[11] Since this extrapolation is questionable, the value $\mu = 0.97$ $\mu_N$ for Ce[141] must be regarded as tentative.

From the derived values of $G_2 U_2 F_2$, we have

$$G_2(\text{CMN})/G_2(\text{NES}) = +0.44(10).$$

It is tempting to interpret this ratio as suggesting $G_2(\text{NES}) \sim 1$, $G_2(\text{CMN}) \sim 0.4$. This would be consistent with $G_2(\text{CMN})$ being a "hard core" value for time-independent perturbations in the 61-keV state and with little or no attenuation in NES. It is clear that much *more* attenuation occurs for this state in CMN than in NES, and this is consistent with evidence for other cases.[12,13] Perhaps this difference suggests that changes in oxidation state following $\beta$ decay is faster in CMN, where the rare-earth ion is surrounded by nitrate ions, than in NES, where water molecules constitute the immediate environment.

## ACKNOWLEDGMENTS

[8] R. J. Blin-Stoyle and M. A. Grace, in *Handbuch der Physik*, edited by S. Flügge (Springer-Verlag, Berlin, 1957), Vol. 42, p. 555.

[9] In the CMN case only, we found $W(0) - 1 > 2[1 - W(\frac{1}{2}\pi)]$ consistently, suggesting an $A_4 P_4 \cos\theta$ term in the angular distribution, with $A_4 \sim 1\%$ at the lowest temperatures. Because this would require a larger $E2$ admixture in the 61-keV $\gamma$ ray than the $K/L$ ratio allows (see Ref. 4), we tentatively attribute the effect to scattering in the cryostat.

[10] R. B. Frankel, D. A. Shirley, and N. J. Stone, Phys. Rev. 136, B577 (1964).

[11] I. Lindgren, in *Alpha-, Beta-, and Gamma-Ray Spectroscopy*, edited by K. Siegbahn (North-Holland Publishing Co., Amsterdam, 1965), Vol. 2, p. 1621.

[12] J. M. Daniels and S. K. Misra, Can. J. Phys. 44, 1965 (1966).

[13] P. H. Barrett and D. A. Shirley, following paper, Phys. Rev. 184, 1181 (1969).