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APPENDIX: 2π RANGE CURVES

In a 2π-range-curve measurement, the percent of the total activity of a given isotope which will penetrate a given thickness of foil t (measured in units of the mean range) will be given by

$$F(t)=100\int_{x=t}^{\infty}\int_{r=x}^{\infty}[P(r)/2r]drdx,$$

where $P(r)$ gives the distribution of ranges. We have assumed a Gaussian distribution and have evaluated this integral numerically. Values of $F(t)$ are given in Table IV for values of t from 0.95 to 1.15 and for relative full widths at half-maximum from 2 to 20%. Data in this table are convenient for the evaluation of the widths of range distribution from the activity experimentally observed in the last foil of a 2π range measurement.

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Nuclear Magnetic Resonance in Beryllium*

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NMR of ^{9}Be in beryllium metal powder has been measured from 5 to 20 kG at 295°K. The isotropic Knight shift is measured as $(-0.0027\pm0.0006)\%$; the anisotropic Knight shift $|a|<0.0003\%$; and the quadrupole coupling e^2qQ/h is measured as 56.4 ± 0.3 kc/sec.

I. INTRODUCTION

THE nearly zero Knight shift in beryllium metal as first reported by Townes *et al.*[1] has been the subject of a number of theoretical attempts[2] at explanation with no success to date. Our interest in the measurement reported here arose from our earlier measurements in metals in the presence of large quadrupole interactions where it is apparent that the position of the $(\frac{1}{2}-\frac{1}{2})$ transition is affected by that quadrupole interaction and that these apparent shifts are magnetic-field-dependent. As will be shown in the following, the quadrupolar energy in beryllium metal is so small that the apparent shift of the $(\frac{1}{2}-\frac{1}{2})$ line from the second-order broadening is less than the precision of measurement.

Our initial measurements did show a measurable Knight shift, and as a result we continued the series of measurements as a function of magnetic field. These measurements provide the opportunity to fit the second-order quadrupole broadening of the $(\frac{1}{2}-\frac{1}{2})$ line with the magnetic field dependence of that linewidth.

The treatment of NMR in polycrystalline metal samples with quadrupolar interactions has been discussed extensively in recent literature[3,4] and is not repeated here. The NMR spectrum is composed of the central $(\frac{1}{2}-\frac{1}{2})$ transition and two symetrically placed satellites that are powder pattern discontinuities of the $(\frac{1}{2}-\frac{3}{2})$ transitions. The maxima of absorption from these discontinuities occur approximately where the angle between the electric field gradient axis and the applied magnetic field is 90°. As shown in Ref. 4, some care must be taken in the measurement of the quadrupole coupling from the position of these discontinuities because of their apparent shifting from the Gaussian dipolar broadening σ.

II. EXPERIMENTAL DESCRIPTION

The NMR apparatus was a Varian broadline induction spectrometer. The electromagnets used in the measurements were Magnion instruments with FFC-4 rotating-coil field controls. The measurements were made at fixed field, and the swept rf frequencies were measured with a Hewlett-Packard 5245L frequency counter. In each run, the field was calibrated with the deuteron resonance in D_2O; the salt reference of Be in BeO was measured; the Be-metal NMR was measured; and finally the deuteron resonance was remeasured to check for any drift in the magnetic field. This procedure

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TABLE I. Purity content by spectrographic analysis of samples.

Impurity	Be metal in %	Be oxide in %
Manganese	0.0053	Nil
Magnesium	0.0023	0.0010
Silicon	0.015	0.016
Iron	0.062	<0.004
Aluminum	0.014	Nil
Copper	0.0030	0.00060
Calcium	Nil	0.0025
Chromium	0.0091	0.00093

was repeated for about ten measurements at each field point.

The powdered samples of beryllium metal and beryllium oxide were supplied by Electronic Space Products, Inc. with the reported typical purity of 99.9% for the BeO. A spectrographic analysis of the samples showed the impurities as listed in Table I. These impurity levels are expected to provide a negligible error in the NMR results especially at the room-temperature measurement.

Both the metal and the salt samples were annealed at about 400°C for 32 h in sealed Pyrex ampules. The $(\frac{1}{2}-\frac{1}{2})$ transition in the beryllium metal narrowed by about 10% from this annealing.

III. RESULTS AND DISCUSSION

The average values obtained for the isotropic Knight shift in the beryllium metal in respect to the ^{9}Be resonance in beryllium oxide at 295°K is $K=(-0.0027\pm 0.0006\%)$. These isotropic Knight shifts are measured by determining the zero-derivative center of the $(\frac{1}{2}-\frac{1}{2})$ resonance. Since this resonance is broadened in second order by the quadrupole interaction, it is important to notice that this broadening is asymmetric and can lead to an error in determining the center of this $(\frac{1}{2}-\frac{1}{2})$ transition. We calculate that this asymmetry in line shape, though not readily observable directly, should provide a field-dependent increase of the apparent Knight shift from -0.0033% at 5 kG to -0.0028% at 12 kG. Although these variations are less than our standard deviation, we have included the appropriate corrections to our measured shifts at each field point. The same type of corrections were made for the BeO reference using $e^2qQ/h=41$ kc/sec as reported by Hon.[5]

The nuclear quadrupole interaction is determined from the splitting of powder-pattern discontinuities of the $(\frac{1}{2}-\frac{3}{2})$ transition. Without static (Gaussian) dipolar broadening these discontinuities occur where the angle between the electric-field-gradient (EFG) axis and the applied magnetic field is 90° and their separation is $\nu_Q=\frac{1}{2}e^2qQ/h$ for beryllium where eq is the axial component of the EFG and eQ is the quadrupole moment of the beryllium nucleus. In the presence of Gaussian broadening σ, it is necessary to correct for the shifted position of the zero derivative of discontinuity position as explained in Ref. 4. With this correction we find $e^2qQ/h=56.4\pm 0.3$ kc/sec.

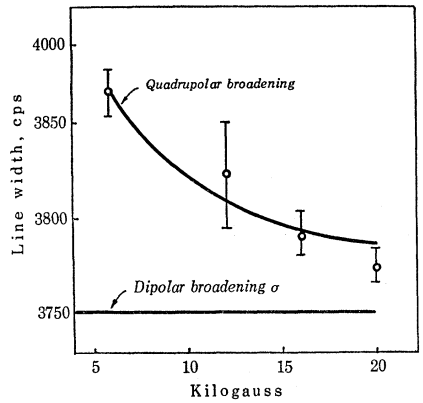

FIG. 1. Magnetic field dependence of the linewidth of beryllium NMR showing the dipolar broadening and the quadrupolar broadening components.

The best test for the anisotropic Knight shift in this powder pattern is a measurement of the linewidth of the $(\frac{1}{2}-\frac{1}{2})$ transition as a function of magnetic field. In Fig. 1 we have plotted our measurements of the $(\frac{1}{2}-\frac{1}{2})$ transition showing the correction of the width from the quadrupolar second-order broadening and the resultant field-independent dipolar width σ. Our value for σ is 3.75 ± 0.01 kc/sec. As seen from Fig. 1, there is no apparent broadening of the $(\frac{1}{2}-\frac{1}{2})$ linewidth with increasing field but the field dependence of the width is well accounted for by the static dipolar width and quadrupolar broadening that is proportional to H^{-1}. Since our precision of measurement of the linewidth should allow the observation of a 100-cps broadening at 20 kG, we use this value as an upper limit on the possible anisotropic Knight shift a; that is, $|a|<0.0003\%$.

This measurement of K has been referred to BeO and in respect to a deuteron resonance in aqueous solution. Other measurements have recently been performed independently of these using an aqueous solution of $BeCl_2$.[6] These values of the beryllium Knight shift are in excellent agreement with the measurement reported here.

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