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MAGNETIC HYPERFINE FIELD, NUCLEAR g-FACTOR, AND SPIN-LATTICE RELAXATION FOR  $^{125}\text{Sb}$  IN IRON FROM NUCLEAR ORIENTATION-NMR

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MAGNETIC HYPERFINE FIELD, NUCLEAR  $g$ -FACTOR, AND SPIN-LATTICE  
RELAXATION FOR  $^{125}\text{Sb}$  IN IRON FROM NUCLEAR ORIENTATION-NMR\*

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August 1967

It has recently been demonstrated<sup>1,2</sup> that nuclear magnetic resonance (NMR) experiments with radioactive nuclei are feasible if the non-isotropic angular distribution emitted from nuclei in an oriented state is used to detect the resonance. The only measurements so far, however, were performed with  $^{60}\text{Co}$ <sup>1,2</sup> and  $^{54}\text{Mn}$ <sup>2</sup> - classical cases in nuclear orientation work - and were primarily designed to give experimental confirmation of the method and to point out its tremendous advantages over conventional techniques. As a continuation of these earlier measurements we report here NMR experiments on 2.7-y  $^{125}\text{Sb}$ .

In recent years  $^{125}\text{Sb}$  has been the subject of several nuclear polarization experiments. The purpose was in all cases to determine the magnetic moment of  $^{125}\text{Sb}$  using the values of the internal fields at Sb nuclei in ferromagnetic hosts obtained by other methods. Hess et al<sup>3</sup> reported a nuclear moment of  $(3.55 \pm 0.3)$  nm. assuming an internal field of 200 kG for  $^{125}\text{Sb}$  in Fe. However, these authors used NaI(Tl) detectors and were observing a superposition of several  $\gamma$ -rays rather than a single transition. To avoid this difficulty Stone et al<sup>4</sup> polarized  $^{125}\text{Sb}$  in Fe and observed the anisotropy of the 462 keV  $\gamma$ -rays only, well resolved with Ge(Li) detectors. Their result,  $(2.72 \pm 0.15)$  nm., agreed well with expected

systematics. These measurements have been continued by the Oxford group<sup>5</sup> and the latest result is reported to be  $(2.55 \pm 0.10)$  nm.

The method of nuclear orientation (NO)-NMR offers a considerably higher degree of accuracy. As pointed out earlier,<sup>1</sup> This accuracy allows one to observe the shift of the resonance as a function of the external polarizing field,  $H_0$ . Provided that the resonance is observed in domains this functional dependence will be linear, the slope giving the g-factor directly and the  $H_0=0$  intercept yielding the hyperfine field times the g-factor.

In addition, NO-NMR can be employed in a very elegant manner to determine the nuclear spin-lattice relaxation time,  $T_1$ .<sup>2</sup> If  $T_L$  is the lattice temperature and  $T_s$  the nuclear spin temperature, thermal equilibrium,  $T_L \equiv T_s$ , leads to ordinary static nuclear orientation when  $T_L$  is sufficiently small.

The angular dependence of  $\gamma$ -rays emitted from oriented nuclei can be described by the equation<sup>6</sup>

$$W(0, T_s) = \sum_{k \text{ even}} B_k(T_s) U_k F_k P_k(\cos \theta), \quad (1)$$

where  $U_k$  and  $F_k$  are coefficients characteristic of the nuclear decay and  $P_k(\cos \theta)$  are the even Legendre polynomials.  $B_k$  describes the degree of orientation and is related to the spin temperature according to the relation

$$B_k = \frac{\sqrt{2I+1} \sum_M (-1)^{I-M} (IIIM-M|k 0) e^{-\frac{E_M}{kT_s}}}{\sum_M e^{-\frac{E_M}{kT_s}}} \quad (2)$$

Under the influence of an rf field at resonant frequency,  $\nu_R = g \cdot (\vec{H}_{\text{int}} + \vec{H}_O) \frac{\mu_N}{h}$ , transitions between the Zeeman levels are induced and the Boltzmann distribution is disturbed. Consequently  $T_s$  is no longer well-defined. Immediately upon changing  $\nu$  to an off-resonance value or switching off the rf-field entirely,  $T_s$  is again defined by the Boltzmann factor, but  $T_s \neq T_L$ . The rate at which  $T_s$  and  $T_L$  equilibrate is approximately characterized by  $T_1$  and described by the relation<sup>2</sup>

$$\frac{d}{dt} \left( \frac{1}{T_s} \right) = - \frac{1}{T_1} \left( \frac{1}{T_s} - \frac{1}{T_L} \right) . \quad (3)$$

The sources used in these experiments were prepared by depositing 1-2  $\mu\text{C}$  of  $^{125}\text{Sb}$  onto iron foils and melting the samples to obtain solid solutions of less than 1 part Sb in  $10^4$  parts Fe. The Fe Sb alloy melts were then flattened in a rolling mill, with repeated intermediate annealing steps, to produce foils of 6000-10000  $\text{\AA}$  thickness. After final annealing two foils of ca. 9  $\text{mm}^2$  size were soldered to a copper fin system using Wood's metal. The copper fins were in good thermal contact with the demagnetization salt, chromium-alum. The salt pill was demagnetized adiabatically from an initial H/T of ca. 27 kG/deg K to cool the alloy to about 0.015<sup>o</sup>K. Accurate temperature measurements were made by observing W(0) for the 426-keV transition with a 30  $\text{cm}^3$  Ge(Li) detector and comparing it to the anisotropy vs T curve obtained using the polarization data from Ref. (4). In a typical run the final temperature measured in this way was 0.014<sup>o</sup>K. For the NMR-measurements, however, a 3"x3" NaI(Tl) detector was used (distance ~12 cm) to provide greater counting efficiency. We thus obtained for the combined 426-462 keV peak a net effect, W(0)-1, of +13%.

The Fe Sb alloy was polarized by a superconducting Helmholtz pair capable of producing fields up to 55 kG.  $H_1$  was applied perpendicular to  $H_0$  by an rf coil of approximately Helmholtz configuration. An rf field amplitude of about 10 mG was used throughout the frequency range indicated in Fig. 1. The rf source was a Marconi TF 1066 B/6 FM signal generator. The present measurements were carried out with a 1 kHz internal frequency modulation of  $\pm 400$  kHz bandwidth. With this arrangement we were able to destroy typically about 25% of the orientation leading to a net resonance effect of approximately 2% along the quantization axis.

The raw data are shown in Fig. 1 for two opposite frequency sweep directions. The sloping background represents the warm-up curve. The anisotropy of the 426-462 keV  $\gamma$ -ray group is positive. From these curves the line width is found to be about 2.0 MHz, representing a tremendous inhomogeneous broadening of the natural linewidth even when the broadening effect of the  $\pm 400$  kHz FM bandwidth is taken into account. This inhomogeneous broadening characterizes the field distribution in a dilute Fe Sb alloy.

The frequency sweep in Fig. 1 was slow compared to the relaxation time  $T_1$ , leading to symmetrical resonance curves. Still, in order to eliminate any possible frequency shift in sweep direction, the two curves of Fig. 1 were added together to give the center frequency of the resonance. The added data, shown in Fig. 2, yield  $\nu_R = 132.15 \pm 0.10$  MHz for  $H_0 = 787$  G.

The observed resonance must be due to nuclei situated in domains, since only a small fraction of  $^{125}\text{Sb}$  nuclei are situated in walls, even in an incompletely polarized sample. Furthermore, nuclei in walls will



orient randomly since the external fields do not penetrate the walls. Such random orientation gives rise to only a small anisotropy and could not account for the observed effect. The unambiguous proof that, in fact, the domain resonance is measured is the shift of the resonance with varying external field  $H_0$ . In Fig. 3 the observed resonance frequency for various  $H_0$  is shown. The linear behavior is described by the relation

$$\nu_R = \frac{\mu_N}{h} g (H_{hf} \pm H_0).$$

The slope of the line thus gives the  $g$ -factor and with the known  $g$ -factor the  $H_0=0$  intercept yields  $H_{hf}$ . It is important to notice that the sign of the  $g$ -factor cannot be obtained (for symmetry reasons) from this type of measurement. The sign of the hyperfine field, however, determines the sign of the slope. From Fig. 3 it is clear that the hyperfine field of  $^{125}\text{Sb}$  in Fe is positive, as measured previously by Samoilov.<sup>7</sup> The data of Fig. 3 are listed in Table I. From a least-squares fit we obtain

$$g \cdot \frac{\mu_N}{h} = 0.570 \pm 0.014 \text{ (MHz kG}^{-1}\text{)}$$

and

$$g \cdot \frac{\mu_N}{h} \cdot H_{int} = 131.711 \pm 0.032 \text{ (MHz)}.$$

From this the  $g$ -factor of  $^{125}\text{Sb}$  is found to be

$$|g| = 0.748 \pm 0.018.$$

With spin  $7/2$ , the magnetic moment is

$$|\mu| = 2.62 \pm 0.06 \text{ nm.},$$

in excellent agreement with the latest

value reported by Stone.<sup>5</sup> We note that this agreement is possible only if the spin is  $7/2$ , and this constitutes a spin determination for  $^{125}\text{Sb}$ .

This result is a basic consequence<sup>1</sup> of the fact that the two methods, NMR and nuclear orientation, measure  $gH$  and  $\mu H$ , respectively.

The internal field value is

$$H_{\text{int}} = + 231 \pm 6 \text{ kG},$$

slightly larger than the field reported by Samoilov,<sup>7</sup> but in excellent agreement with NMR results of Kontani and Itoh,<sup>8</sup> who found +230 kG.

Finally the spin-lattice relaxation time for  $^{125}\text{Sb}$  in iron was measured as described elsewhere,<sup>2</sup> and a least-squares fit was made to

$$W(0) \cong A + B e^{-t/T_1},$$

which is the first-order solution of Eqs. 1-3. We found  $T_1 = 145 \pm 20$  sec at  $0.015^\circ\text{K}$ , or  $T_1 T = 2.2 \text{ sec } ^\circ\text{K}$ . This large value indicates that neither the orbital relaxation mechanism<sup>9</sup> nor additional relaxation due to localized moments<sup>2</sup> is present. The positive hyperfine field on Sb in Fe is thought to arise from the s band, which is consistent with this large  $T_1 T$ .

Footnote and References

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Table I. Resonance frequency for various values of the polarizing field.

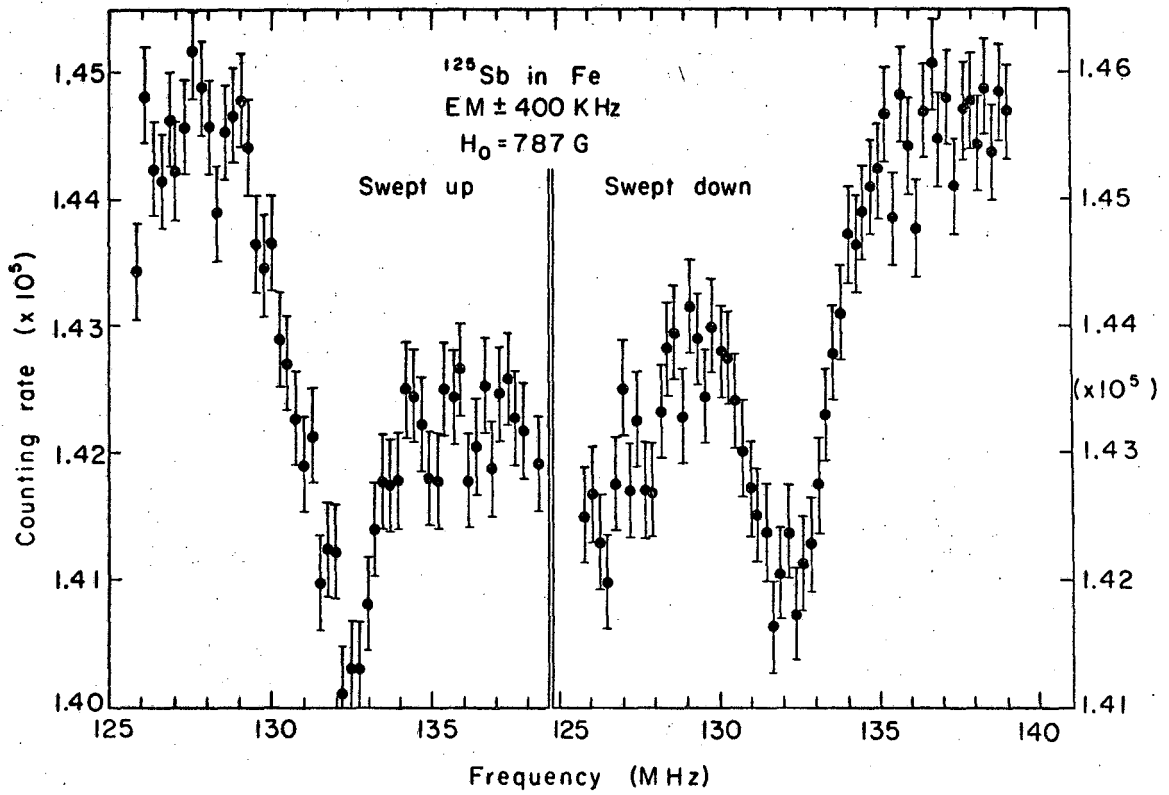
$H_0$ (gauss)	$\nu_R$ (MHz)
787	$132.15 \pm 0.20$
1560	$132.60 \pm 0.20$
4000	$134.05 \pm 0.20$
6000	$135.05 \pm 0.20$

Figure Captions

Fig. 1. Nuclear orientation NMR for  $^{125}\text{Sb}$  in Fe. The data represent  $W(0^\circ)$  for the combined 426-462 keV gamma group which shows a positive net anisotropy. The frequency was swept in opposite directions in the left and right part of the figure, causing the opposite slopes in the warm-up curve.

Fig. 2. Resonance for  $^{125}\text{Sb}$  in Fe. The data points represent the sum of the frequency scans shown in Fig. 1. The center of the resonance appears at  $\nu_R = 132.15 \pm 0.10$  MHz.

Fig. 3. Shift of the resonance frequency with external polarizing field. The linear behavior proves that domain resonances were observed. These data determine unambiguously  $g$ ,  $H_{hf}$  and the sign of  $H_{hf}$ .



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Fig. 1

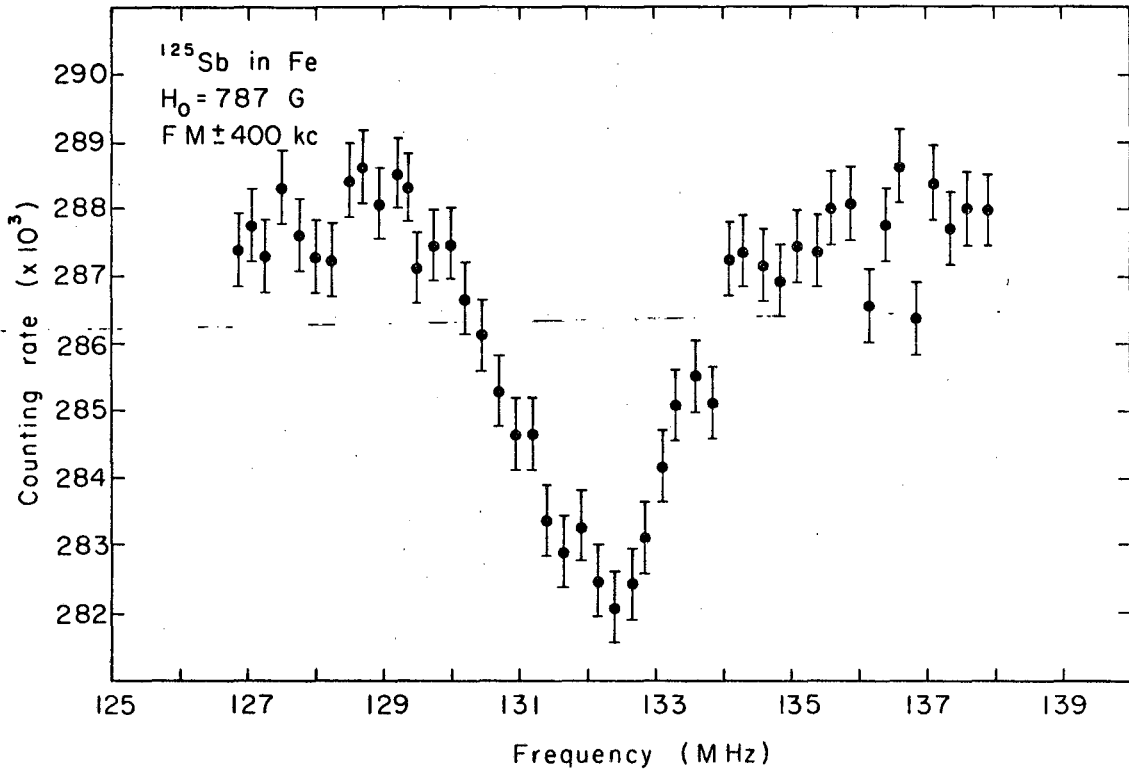


Fig. 2

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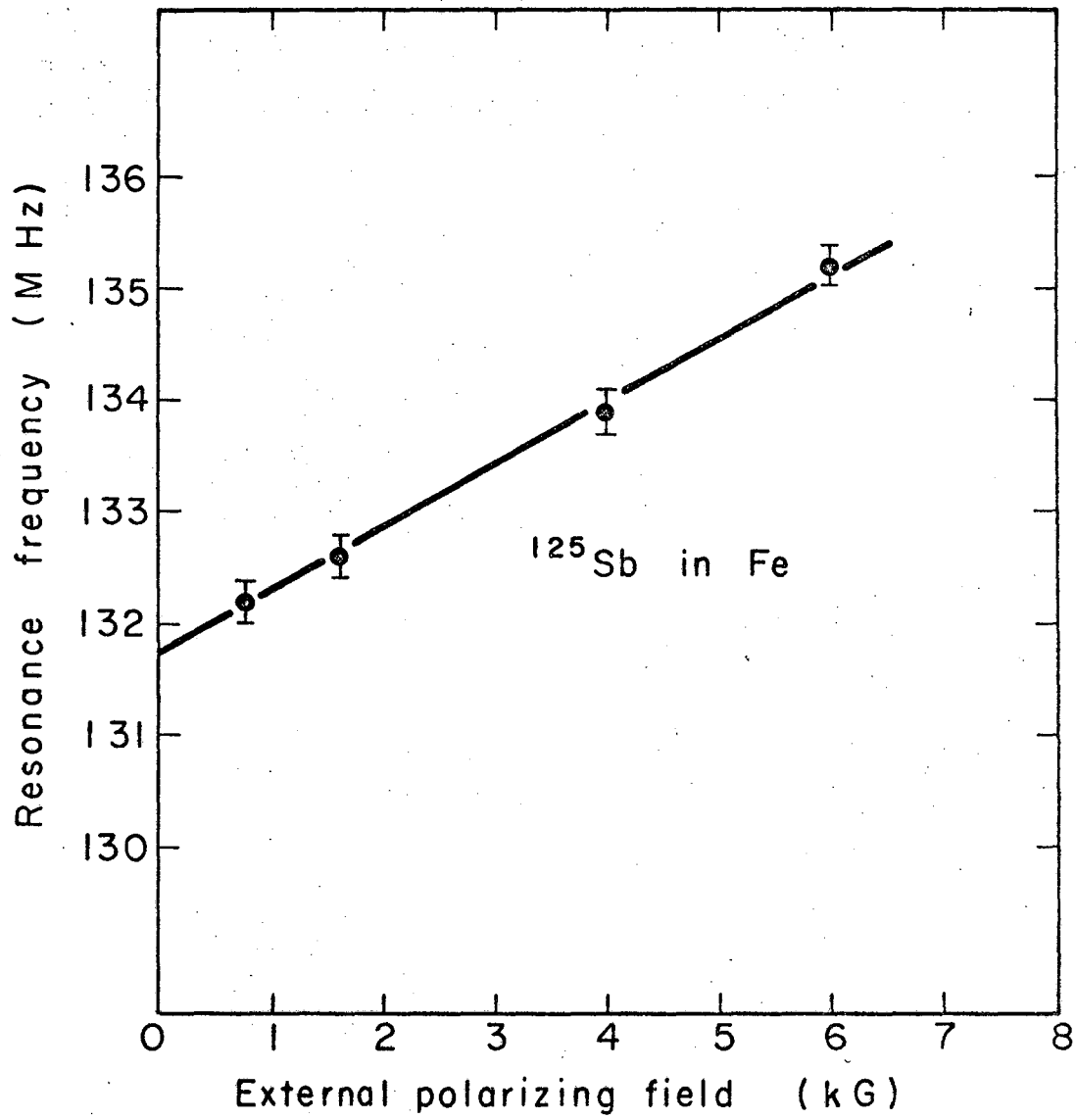


Fig. 3

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