value of the spin of Ti of either  $3.1 \pm 0.4$  or  $3.7 \pm 0.4$ , depending upon whether the isotopic abundance is taken to be that of Ti<sup>47</sup> or Ti<sup>49</sup>, respectively. This would indicate that the spin is probably 7/2 and that the signals are probably due to Ti<sup>49</sup> instead of Ti47, although this measurement is not conclusive because of uncertainties in the degree of dissociation. From the observed optimum signal to noise ratios of the Ti resonances in various samples we can also estimate the spin to be  $7/2\pm 1$ . Since we have observed the magnetic moment to be negative, this indicates an  $f_{7/2}$  state for the odd neutron, which is not in disagreement with the predictions of the nuclear shell model.<sup>6</sup> Assuming, then, that the spin is 7/2 we find from (1) that the diamagnetically uncorrected value of the magnetic moment of Ti<sup>49</sup> (or perhaps Ti<sup>47</sup>) is

$$\mu_{\rm Ti} = -(1.1022 \pm 0.0003) \,\,\rm nm, \tag{2}$$

where we have taken the proton moment to be 2.7925 nm. We have searched over a wide region for the resonance signal from the other odd Ti isotope, but have as yet failed to find it, probably because the gyromagnetic ratio is small.

We have observed the nuclear magnetic resonance of As<sup>75</sup> in a 1.2 molar aqueous solution of  $\mathrm{Na_3AsS_4}$  and also in a basic aqueous solution of Na<sub>3</sub>AsO<sub>4</sub>. The Na<sub>3</sub>AsS<sub>4</sub> sample gives a large signal, with a half-width of about 0.7 gauss; the line width is limited by quadrupole broadening, which is not excessive in this case because the sample is well dissociated into symmetric  $(AsS_4)^{--}$  ions. On the other hand, it was found that in the  $Na_3AsO_4$  sample the As resonance signals were obliterated by excessive quadrupole broadening unless the sample was made basic (by the addition of NaOH) to a  $pH \cong 12$ . This is in agreement with the chemical evidence that  $Na_3AsO_4$  does not dissociate into  $(AsO_4)^{--}$  ions, except in basic solution.7 We have not detected a "chemical shift"5 between the As resonance frequencies in  $(AsS_4)^{--}$  and  $(AsO_4)^{--}$ . The ratio of the As<sup>75</sup> resonance frequency to that of protons in  $H_2O$  in the same magnetic field has been found to be

$$\nu_{\rm As75}/\nu_{\rm H} = 0.17129 \pm 0.00003.$$
 (3)

The spin<sup>8</sup> of  $As^{75}$  is known to be 3/2, and we have verified this by comparing the As<sup>75</sup> signal from Na<sub>3</sub>AsS<sub>4</sub> to the D<sup>2</sup> signal in D<sub>2</sub>O; our experimental result is  $I(As^{75}) = 1.5 \pm 0.2$ . Taking the proton moment to be 2.7925 nm, we find from (3) the diamagnetically uncorrected value of the moment of  $As^{75}$  to be

$$\mu(\text{As}^{75}) = +(1.4350 \pm 0.0003) \text{ nm.}$$
 (4)

This value is in agreement with that recently reported by Dharmatti and Weaver.9

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<sup>4</sup> We are independent to Diffigure for information discussion of the second second

# The Nuclear Magnetic Moment of Tc<sup>99\*</sup>

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HE nuclear resonance of long-lived radioactive Tc<sup>99</sup> has been observed in a nuclear induction apparatus of the type originated by Bloch and similar to that recently described by Proctor.<sup>1</sup> The sample contained 156 mg of Tc<sup>99</sup>, as NH<sub>4</sub>TcO<sub>4</sub>, in an aqueous solution. The Tc<sup>99</sup> was isolated from fission products by one of us (W.J.M.). One ml of D<sub>2</sub>O was added to the solution

giving a total volume of 6 ml, and all frequency ratio measurements were relative to deuterium. Frequency measurements were made with a Signal Corps type BC-221 frequency meter calibrated with harmonics from an external 100 kc, crystal-controlled oscillator which in turn was compared with the National Bureau of Standards radio station WWV at 5 Mc. Frequencies were measured at nominal fields of 7200 and 8300 gauss giving

#### $\nu(Tc^{99})/\nu(D) = 1.46628 \pm 0.0001.$

With Levinthal's<sup>2</sup> deuteron-to-proton frequency ratio of 0.1535059, the above ratio yields the following frequency ratio relative to the proton,

# $\nu(Tc^{99})/\nu(H) = 0.22508.$

With a Tc<sup>99</sup> diamagnetic correction of 0.411 percent,<sup>3</sup> a spin of 9/2 for  $\mathrm{Tc}^{99},\!{}^4$  and a value of 2.79268 nuclear magnetons  ${}^5$  for the proton moment, this ratio gives the following value of the nuclear magnetic moment, in units of the nuclear magneton

#### $\mu(Tc^{99}) = +5.6805 \pm 0.0004.$

The indicated estimated accuracy of the above value does not include the uncertainty in the diamagnetic correction. The sign of the Tc<sup>99</sup> magnetic moment was obtained by comparison with D, which is known to be positive.1

A previous measurement of the nuclear magnetic moment of Tc<sup>99</sup> by Kessler and Meggers<sup>6</sup> made by optical spectroscopy gave  $5.2\pm0.5$  nuclear magnetons, which may be increased 10 to 20 percent by additional corrections. This value is consistent with the more accurate value reported above.

\* This work was performed for the AEC at Oak Ridge National \* This work was performed for the fibe at call large 1 Laboratory.
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### Structural Changes in the Ferroelectric Transition of KH<sub>2</sub>PO<sub>4</sub>\*

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S the temperature is lowered through the ferroelectric Curie A<sup>S</sup> the temperature is lowered through the tetragonal  $I\overline{4}2d$ point (122°K), KH<sub>2</sub>PO<sub>4</sub> changes from the tetragonal  $I\overline{4}2d$ (or  $F\overline{4}d2$ ) symmetry to that of the orthorhombic space group Fdd. This was established in the earlier x-ray investigation of de Ouervain<sup>1</sup> and of Ubbelohde and Woodward.<sup>2</sup> These authors also accurately measured the lattice deformation and noted that changes in intensity were observable in some of the diffraction maxima. De Quervain measured a few of the intensities from room temperature down past the transition. Working on the basis of the well-known room temperature determination of the tetragonal structure by West,3 he suggested a model for the ferroelectric structure involving displaced P's in deformed PO4 groups; however, he had an insufficient number of observations (and these were not from the most favorable class of reflections) to test his model.

We undertook a more complete attack on this problem with the purpose of determining the atomic rearrangements associated with the transition. Of specific interest was the detection of relative displacements of the constituent ions parallel to the polar c-axial direction. It should be mentioned here that the Slater<sup>4</sup> theory of the transition, while very probably playing an important role, cannot in itself account for the polarization of the crystal. It is reasonable to suppose that his scheme of hydrogen bond orientation does occur, but this must either "trigger" other structural changes or must itself be the result of the other changes. Of course, all of the contributing factors are not amenable to x-ray analysis,