tric. This latter assumption requires that $z=(3/2)^{\frac{1}{2}}(a/c)x$, and reduces the determination of the Ti–F distance to the determination of the single parameter x. For the calculation of the second moment all F–F internuclear distances out to 10 Å and all K–F and Ti–F distances out to 5 Å as a function of x were calculated for the symmetric model; the sums in equation (2) over distances longer than these were obtained by integration. The following value of x and the distances corresponding to it were calculated from the observed second moment:

$$x = 0{\cdot}1580 \pm 0{\cdot}0016 \quad (z = 0{\cdot}2376 \pm 0{\cdot}0024);$$
$$\text{Ti–F} = 1{\cdot}916 \pm 0{\cdot}020 \text{ Å}, \quad \text{F–F} = 2{\cdot}709 \pm 0{\cdot}028 \text{ Å}.$$

The contributions of the various terms in (2) to the moment are F–F 98·5%, Ti–F 1·3%, and K–F 0·2%.

The limits of error given above are those which arise from the errors in the measurement of the second moment, and do not reflect the errors due to the assumption of a symmetric model. It is reasonable to ask how large these latter errors might be. Siegel's structure corresponds to a calculated second moment of 6·48 gauss^2. The symmetric model which corresponds to this moment has Ti–F = 1·904 Å, some 0·013 Å shorter than Siegel's value. It should be noted, however, that the two F–F distances derived by Siegel are not significantly different on the basis of the usual statistical tests (Cruickshank & Robertson, 1953); hence, the symmetric model cannot be eliminated on the basis of Siegel's results. It is thus unreasonable to require that the Ti–F distance derived above should be corrected because it is based on the symmetric model. Moreover, the Ti–F distances of 1·917± 0·026 Å (X-ray) and 1·916±0·020 Å (n.m.r.) are in excellent agreement.

The value of the Ti–F distance derived above from the nuclear magnetic resonance data is believed to be reasonably accurate and precise. What is perhaps more important, it was obtained with considerably less time and effort than would be required to obtain similar precision from X-ray methods. Thus, nuclear magnetic resonance techniques are powerful ones for the elucidation of certain, rather specific, structural information. The case of K_2TiF_6 is perhaps atypical, for the structure is so simple that the Ti–F distance could have been derived from the second moment without the aid of the X-ray information. In general, however, this is not the case, and it must be emphasized that nuclear magnetic resonance techniques are essentially complementary with X-ray techniques.

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The unit cell and space group of the compound $TiNb_2O_7$.*

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The compound $TiNb_2O_7$ was first recorded by Roth & Coughanour (1955) in a paper dealing with phase equilibrium in the system TiO_2–Nb_2O_5. Its existence has been confirmed in the course of a similar investigation in these Laboratories.

When fired at 1350° C., pressed blocks of the mixed oxides, of appropriate composition, yield a somewhat porous, pale yellow mass of crystals of the compound, up to $\frac{1}{4}$ mm. in size.

The ease of crushing indicates a hardness of about 4 on Mohs scale.

There are two good cleavages which make a large angle with one another. On account of this, crushed fragments often show an elongated prismatic form parallel to the edge common to the cleavage traces. Between crossed nicols the extinction is parallel to this edge. The refractive indices are much higher than 1·74 (methylene iodide) and the birefringence, in sections parallel to a cleavage direction, is low.

The above observations indicate that the symmetry is at least monoclinic, with the b axis parallel to the cleavage traces.

Single-crystal rotation and zero- and first-layer Weissenberg photographs of a cleavage fragment rotated about the b axis give the following dimensions for the unit cell:

$$a_0 = 11{\cdot}9, \; b_0 = 3{\cdot}77, \; c_0 = 10{\cdot}1 \text{ Å}, \; \beta = 120°.$$

The Weissenberg photographs show no systematic absences of reflexions. The space group is thus one of three, namely $P2$, Pm or $P2/m$.

An approximate density measurement suggested 4·38 ($TiNb_2O_7$) per unit cell. If the true value is assumed to be 4 ($TiNb_2O_7$), the calculated density is 5·89 g.cm.$^{-3}$.

The dielectric constant shows no anomalies with change of temperature and does not establish the presence or absence of a centre of symmetry.

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