Fig. 2. The crystal structure of KCr$_3$O$_4$ projected on (010), (a=8.569 Å, b=5.466 Å, c=7.622 Å, β=95.25°). By shifting some of the oxygen atoms in the KCr$_3$O$_4$ type of structure in directions indicated by the arrows, rows of metal-oxygen octahedra are formed. Such octahedra are filled by Li-atoms and Cr$^{III}$-atoms.

structure, the coordination around the alkali atoms may be changed to an octahedral one. In the structure of LiCr$_3$O$_4$, these positions are occupied by lithium and chromium atoms in a random way, forming somewhat staggered strings of octahedra connected by sharing edges in the c-direction. By sharing corners the strings are linked via CrO$_4$ tetrahedra to form a three-dimensional framework. Each CrO$_4$ tetrahedron is in contact with three separate octahedral strings.

From a formal point of view the structure of M$_2$Cr$_3$O$_8$ can be derived from the (Li,Cr)Cr$_3$O$_4$ structure by introducing a big cation in one half of the octahedral sites in an ordered way as well as adding another cation for each chain this resulting in a split up of the three-dimensional framework structure to the one-dimensional chain structure of the M$_2$Cr$_3$O$_8$ family.

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On the Crystal Structure of Phthalamidocyclohexane

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An X-ray crystallographic investigation of phthalamidocyclohexane was undertaken in order to establish to what extent the cyclohexane ring is flattened.

\[
\begin{align*}
\text{O} & \quad \text{C} \\
\text{N} \quad \text{C} \quad \text{O} & \quad \text{CH}_2-\text{CH}_2 \\
\text{C} \quad \text{O} & \quad \text{CH}_2-\text{CH}_2
\end{align*}
\]

The crystals are orthorhombic (a=21.92 Å, b=8.042 Å, c=6.789 Å) with two possible space groups: Pnma and Pna2$_1$. Intensity data were obtained (at room temperature) from photometric measurements of integrated equiinclination Wiessenberg diagrams (CuK$\alpha$-radiation) corresponding to $h0l$, $...$, $hk5$ and $h0l$, $...$, $h5l$. The diagrams show a considerable amount of diffuse scattering.
reflections were strong enough to be measured.

The intensities were statistically put on absolute scale. Wilson-ratio and N(z)-plot strongly indicated the centrosymmetrical space group (Pnma) which requires that the molecules retain a mirror plane in the crystals.

The phase problem was solved by a computer procedure based on direct methods. Signs for 250 large structure factors were determined and used in the calculation of a three-dimensional Fourier synthesis which closely resembles the final synthesis, of which Fig. 1 shows the electron density in the mirror plane at \( y = \frac{1}{4} \), and a section through the two carbon atoms below the mirror plane.

By postulating the disorder of the cyclohexane ring suggested by the Fourier map, and introducing anisotropic thermal vibration parameters, full-matrix least squares refinement gave a final R-value of 12.8 % for 1073 observed reflections.

Final coordinates and r.m.s.-amplitudes, obtained by analysis of thermal parameters, are given in Table 1. Although interatomic distances and angles (Figs. 2

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**Table 1.** Final fractional coordinates and root mean square amplitudes (multiplied by \( 10^4 \) and \( 10^3 \), respectively).

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Phosphorus Spin-Lattice Relaxation in Phosphorus Trioxide

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1. The liquid state. Recently Mowthorpe and Chapman \(^1\) presented a spin-lattice relaxation study on phosphorus trioxide, P\(_2\)O\(_5\). They concluded that the \(^{31}\)P spin-lattice relaxation time is controlled by the spin-rotation interaction. This might well be the case; however, their conclusion rests on few experimental results, and apparently on erroneous formulae for the contribution to \(T_1\) from the dipolar and spin-rotation interactions. Furthermore, the two reported \(T_1\) values (11.5 s and 11.7 s) measured at ‘ambient’ temperature, are certainly too small because we have measured \(T_1\) values at 9 MHz by the \(90^\circ-\tau-90^\circ\) pulse technique, from 17.0 to 20.5 s in supercooled P\(_2\)O\(_5\) (see Fig. 1). The change in \(T_1\) was small in the limited temperature range studied, but \(T_1\) appeared to decrease slowly with temperature.

All possible care was taken in preparing the sample. In spite of this, it showed traces of red phosphorus after having been exposed to light for some time. An extremely pure sample of P\(_2\)O\(_5\) might well be found to have even larger \(T_1\) values.

The dipolar contributions to \(T_1\) for P\(_2\)O\(_5\) are given by \(^2\)

\[
\frac{1}{T_{1d \text{ intra}}^d} = \frac{9h^2\gamma_p^4}{8\pi^2\tau_{\text{d}P-P}}\ \ \ \ \ \ (1)
\]

\[
\frac{1}{T_{1d \text{ inter}}} = \frac{6h^2\gamma_p^2N\eta}{5kT}\ \ \ \ \ \ (2)
\]

These equations are based on the assumptions of Brownian motion of molecules and incoherence of the various spin-spin interactions.

The correlation time, \(\tau_d\), for reorientation can be estimated from the Debye formula \(\tau_d = 4\pi \eta \alpha a^2 / 3kT\). The ‘radius’, \(a\), of the molecule is obtained from the melting point density assuming close packing. With \(a^2 = 3.32 \times 10^{-23} \text{ cm}^2\) and \(\eta_{31} = 0.025\) poise we find \(\tau_d = 8.6 \times 10^{-11} \text{ s}\). Using eqns. (1) and (2) with \(r_{\text{P-P}} = 2.98 \text{ Å}\) and \(\xi_{31} = 1.943\) we obtain the following contributions to \(T_1\) at 21°C.

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