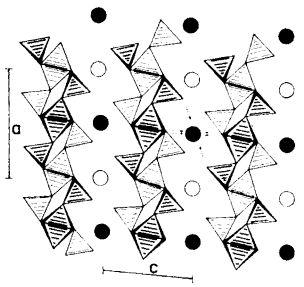

Fig. 2. The crystal structure of KCr_3O_8 projected on (010), ($a=8.569$ Å, $b=5.466$ Å, $c=7.622$ Å, $\beta=95.25°$). By shifting some of the oxygen atoms in the KCr_3O_8 type of structure in directions indicated by the arrows, rows of metal-oxygen octahedra are formed. Such octahedra are filled by Li-atoms and Cr^{III}-atoms.

structure, the coordination around the alkali atoms may be changed to an octahedral one. In the structure of $LiCr_3O_8$,[3] 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 $M_2Cr_3O_9$ can be derived from the $(Li,Cr)Cr_2O_8$ 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 $M_2Cr_3O_9$ family.

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On the Crystal Structure of Phthalimidocyclohexane

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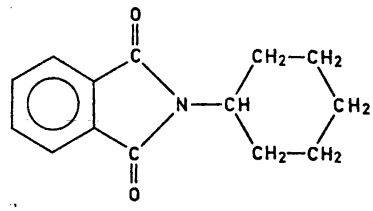
An X-ray crystallographic investigation of phthalimidocyclohexane was undertaken in order to establish to what extent the cyclohexane ring is flattened.[1]



The crystals are orthorhombic ($a=21.92_0$ Å, $b=8.04_2$ Å, $c=6.78_9$ Å) with two possible space groups: $Pnma$ and $Pna2_1$. Intensity data were obtained (at room temperature) from photometric measurements of integrated equiinclination Weissenberg diagrams (Cu$K\alpha$-radiation) corresponding to $hk0$, ··· $hk5$ and $h0l$, ··· $h5l$. The diagrams show a considerable amount of diffuse scattering. 1073 independent

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

	x	y	z	$(\text{r.m.s.})_1$	$(\text{r.m.s.})_2$	$(\text{r.m.s.})_3$
O_1	−1401	2500	5714	410	298	221
O_2	0016	2500	9913	487	312	198
N	−0821	2500	8092	406	268	192
C_1	−0905	2500	6383	314	230	218
C_2	−0286	2500	5641	217	211	205
C_3	−0114	2500	3987	286	217	211
C_4	0509	2500	3657	312	243	222
C_5	0917	2500	4895	363	249	235
C_6	0736	2500	6617	357	234	218
C_7	0130	2500	6921	261	211	205
C_8	−0196	2500	8524	380	246	189
C_{9A}	−1187	2500	9647	240	200	189
C_{9B}	−1514	2500	8833	245	223	202
C_{10A}	−2374	2500	1217	338	309	170
C_{10B}	−2196	2500	2041	363	350	214
C_{11}	−1562	0639	9757	347	248	221
C_{12}	−2094	0675	1040	409	298	218

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.

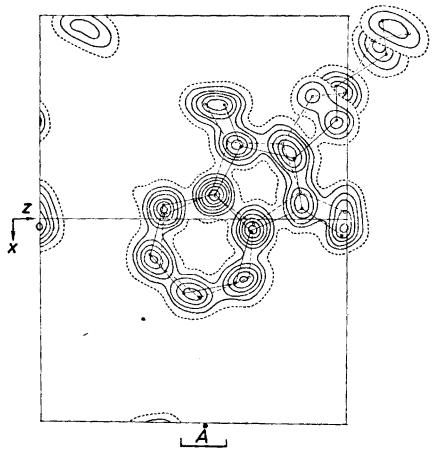


Fig. 1. The electron density in the mirror plane at $y=\frac{1}{4}$ and in a section through the two cyclohexane carbon atoms below the mirror plane.

The phase problem was solved by a computer procedure based on direct methods.[2] 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 [3] 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

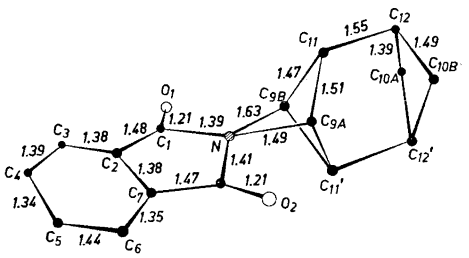


Fig. 2. Schematical drawing showing interatomic distances.

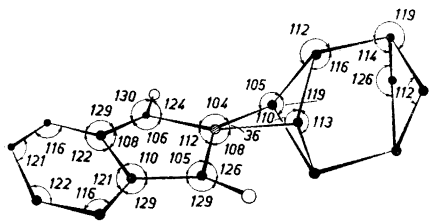


Fig. 3. Schematical drawing showing bond angles.

and 3) of the phthalimide part of the molecule are fairly resonable (σ(dist.)$\sim$0.01 Å, σ(angle)$\sim$1°), the r.m.s.-amplitudes of Table 1 and the distance $N-C_{9B}$ of 1.63 Å (Fig. 2), suggest that also this part is disordered (Fig. 1).

Several unsuccessful attempts have been made to refine disordered structures, starting with models satisfying the requirement of two equal N−C distances of about 1.49 Å. It is felt that the methods of calculations rather than wrong assumptions are responsible for the lack of convergence.

A list of observed structure factors is available by request to the author.

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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_4O_6. 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 (11.3 and 11.7 s) measured at 'ambient' temperature, are certainly too small because we have measured T_1 values at 9 MHz by the $90°-\tau-90°$ pulse technique, from 17.0 to 20.5 s in supercooled P_4O_6 (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_4O_6 might well be found to have even larger T_1 values.

The dipolar contributions to T_1 for P_4O_6 are given by [2]

$$\frac{1}{T_{1\,\mathrm{d\ intra}}} = \frac{9h^2\gamma_p^4}{8\pi^2 r_{p-p}^6}\,\tau_d \qquad (1)$$

$$\frac{1}{T_{1\,\mathrm{d\ inter}}} = \frac{6h^2\gamma_p^4 N\eta}{5kT} \qquad (2)$$

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

The correlation time, τ_d, for reorientation can be estimated from the Debye formula $\tau_d = 4\pi\eta a^3/3kT$. The 'radius', a, of the molecule is obtained from the melting point density assuming close packing. With $a^3 = 3.32\times10^{-23}$ cm^3 and $\eta_{21} = 0.025$ poise we find $\tau_d = 8.6\times10^{-11}$ s. Using eqns. (1) and (2) with $r_{p-p} = 2.98$ Å and $\varrho_{21} = 1.943$ we obtain the following contributions to T_1 at 21°C.