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## Molecular complexes exhibiting polarization bonding. V. The crystal structure of the azulene-*s*-trinitrobenzene complex.

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As part of a series of investigations of complexes of trinitrobenzene (*e.g.* Part IV: Brown, Wallwork & Wilson, 1964), the crystal structure of the complex with azulene has been determined in order to investigate the possible influence of dipole–dipole interactions on the molecular arrangement.

The crystal data are as follows:

$C_{10}H_8 . C_6H_3(NO_2)_3$, $M=341{\cdot}3$. Monoclinic, $a=14{\cdot}05$, $b=6{\cdot}76$, $c=16{\cdot}41$, all $\pm 0{\cdot}02$ Å, $\beta=96{\cdot}0\pm 0{\cdot}5°$, $U=1548$ Å$^3$, $D_m=1{\cdot}45$ g.cm$^{-3}$ (by flotation), $Z=4$, $D_c=1{\cdot}46$ g.cm$^{-3}$, $F(000)=624$, Cu $K\alpha$, $\lambda=1{\cdot}542$ Å, $\mu=11{\cdot}2$ cm$^{-1}$. Absent reflexions $h0l$ when $l$ is odd, $0k0$ when $k$ is odd. Space group $P2_1/c$.

The positions and orientations of the molecules were obtained by consideration of the pleochroism, the weighted reciprocal lattice and a three-dimensional sharpened Patterson summation. The structure was refined initially by electron-density projections and three-dimensional electron-density sections and finally by six cycles of least-squares analysis.

Throughout the refinement, difficulty was experienced in distinguishing between two possible orientations of the azulene molecule, related to each other by rotating the molecule through approximately 180° in its own plane. The orientation used in the later stages was that which gave a slightly better reliability index, $R$. However, convergence of the least-squares refinement was obtained with $R=0{\cdot}19$, suggesting some disorder in the structure. Further evidence of a disordered structure, in which a certain proportion of the azulene molecules is approximately reversed, was obtained from the electron-density sections (Fig. 1), the difference Fourier sections, the high apparent temperature factors of the transannular carbon atoms and an impossibly large distance (approximately 1·8 Å) between them obtained from the coordinates.

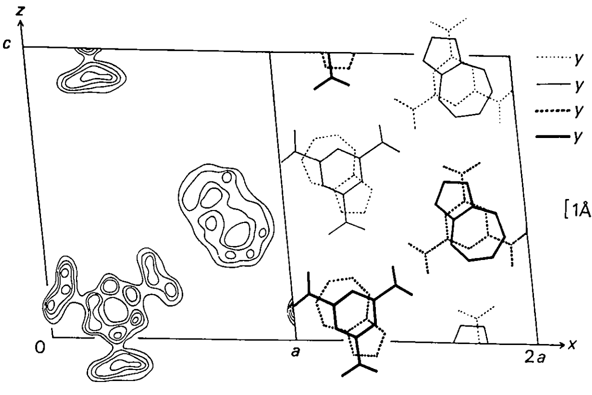

Fig. 1. Electron-density section at $y=\frac{1}{8}$ and structure of the azulene–*s*-trinitrobenzene complex.

The structure (Fig. 1) consists of approximately planar molecules arranged alternately in a plane-to-plane manner in infinite columns parallel to the $b$ axis, the planes making an angle of 87½° with this axis, and having an average perpendicular separation of 3·38 Å. No sideways short contacts have been observed and there is no evidence of localized dipole–dipole interactions, either sideways or between molecular planes.

In view of the accurate low-temperature structure analysis of this complex now in progress (Hanson, 1964) a more detailed investigation of the disorder and further refinement of the present structure is not being attempted, and no atomic coordinates or molecular dimensions are reported here.

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## Crystallographic data for some testosterone esters.

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The crystallographic data for the testosterone esters given in Table 1 were obtained from oscillation and Weissenberg photographs (Cu $K\alpha$ radiation). The densities were determined by flotation, using aqueous solutions of cadmium chloride.

Brief notes on the appearance of the crystals and the space-group determinations are given in the text. The maxi-

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