Acta Cryst. (1965). **19**, 149

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

By D. S. Brown* and S. C. Wallwork, *Department of Chemistry, University of Nottingham, England*

(*Received* 18 *September* 1964 *and in revised form* 19 *October* 1964)

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, $\beta=96{\cdot}0\pm0{\cdot}5°$, $U=1548$ 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.

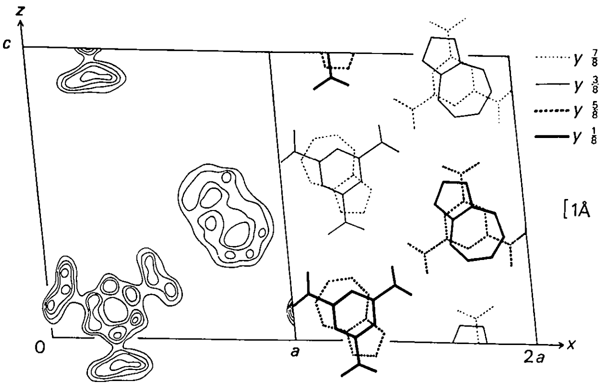

Fig. 1. Electron-density section at $y=\frac{1}{8}$ and structure of the azulene–*s*-trinitrobenzene complex.

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.

We are indebted to the Director of the Oxford University Computing Laboratory for computing facilities and to the authors of programs used, particularly J. S. Rollett, O. S. Mills and R. A. Sparks. We are grateful to the Royal Society for a grant towards the cost of apparatus, and one of us (D.S.B.) thanks the Department of Scientific and Industrial Research for a maintenance grant. We also wish to thank Mrs D. H. Thomas for assistance with the diagram.

References

Brown, D. S., Wallwork, S. C. & Wilson, A. (1964). *Acta Cryst.* **17**, 168.
Hanson, A. W. (1964). Private communication.

---

* Present address: Department of Chemistry, Loughborough College of Technology, Loughborough, England.

Acta Cryst. (1965). **19**, 149

Crystallographic data for some testosterone esters.

By P. J. F. Griffiths†, K. C. James* and M. Rees*, *Welsh College of Advanced Technology, Cardiff, Wales*

(*Received* 10 *February* 1965)

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-

---

† Department of Chemistry and Biology.
* Welsh School of Pharmacy.

Table 1. *Crystallographic data for testosterone esters*

Compound	Space group	a(Å)	b(Å)	c(Å)	β	D_m (g.cm^{-3})	D_x (g.cm^{-3})	z
Testosterone formate	$P2_1$	21·7	12·5	12·9	96°	1·22	1·21	8
Testosterone acetate	$P2_12_12_1$	12·6	18·1	7·8	—	1·22	1·22	4
Testosterone propionate	$P2_12_12_1$	12·6	20·3	7·6	—	1·17	1·17	4
Testosterone butyrate	$P2_12_12_1$	12·3	16·3	10·3	—	1·16	1·15	4
Testosterone valerate	$P2_12_12_1$	12·3	16·7	10·3	—	1·18	1·17	4

mum error in the cell dimensions and observed densities is of the order of 1 % and the interaxial angle is accurate to within 1°.

Testosterone formate, $C_{20}H_{28}O_3$

This ester can be recrystallized from n-hexane or ethanol as colourless needles elongated along [001]. The Laue symmetry is $2/m$ and reflexions $0k0$ are systematically absent when k is odd, so that the space group is either $P2_1$ or $P2_1/m$. Since testosterone and its esters are optically active (*British Pharmacopœia*, 1963) the space group is determined as $P2_1$.

Testosterone acetate, propionate, butyrate and valerate,
$C_{21}H_{30}O_3$, $C_{22}H_{32}O_3$, $C_{23}H_{34}O_3$ *and* $C_{24}H_{36}O_3$

All four esters can be recrystallized from ethanol as colourless needles elongated along [001]. For each ester, the Laue symmetry is *mmm* and the space group is determined uniquely as $P2_12_12_1$ by the systematic absences.

The authors wish to thank the Department of Applied Physics of this College for the loan of X-ray diffraction equipment.

References

British Pharmacopœia (1963). p. 812.

Acta Cryst. (1965). **19**, 150

Crystallographic data for YCo_3 and Y_2Co_{17}*.

By Werner Ostertag, *Air Force Materials Laboratory, Materials Physics Division, Wright-Patterson Air Force Base, Ohio, U.S.A.*

(*Received* 26 *October* 1964)

In the study of the yttrium and cobalt phase diagram YCo_3 and Y_2Co_{17} have been found to exist as single-phase compounds. The samples were prepared by levitation melting from stoichiometric amounts of yttrium and cobalt (both 99·5 % pure). To homogenize the samples, they were wrapped with tantalum foil, sealed in quartz capsules under vacuum and annealed for 200 hours to 800 °C. The compounds were then ground in an agate mortar under carbon

Table 1. *Powder diffraction data for* YCo_3

hkl	d_o	d_c	I_o	I_c	*hkl*	d_o	d_c	I_o	I_c
006	2·710	2·714	*m*	5·0	1,1,10	1·365	1·366	*m–s*	4·3
110	2·506	2·508	*s*	12·0	304		1·364		5·4
112	2·400	2·397	*w*	1·5	0,0,12	1·356	1·357	*w–m*	2·0
200	2·158	2·171	*m*	3·1	127	1·341	1·341	*vw*	0·8
201		2·153		2·5	2,0,10	1·302	1·303	*w–m*	2·9
114	2·134	2·135	*vs*	14·0	218	1·278	1·278	*w*	1·2
202	2·096	2·099	*w–m*	3·8	306		1·277		1·0
107	2·051	2·050	*w*	1·3	220	1·251	1·254	*s*	21·0
008	2·038	2·035	*m–s*	7·1	221		1·250		1·0
203	2·018	2·016	*w–m*	3·1	222	1·241	1·239	*w*	2·4
204	1·916	1·916	*m–s*	7·4	2,0,11	1·224	1·223	*w*	1·5
108	1·841	1·843	*s*	11·4	129	1·215	1·216	*m*	3·5
106		1·843		8·4	130	1·204	1·205	*s*	13·2
205	1·804	1·807	*m–s*	7·5	131		1·201		1·3
206	1·695	1·695	*w*	2·7	224	1·197	1·198	*s*	21·1
109	1·669	1·670	*w–m*	3·5	1,1,12	1·193	1·193	*s*	28·8
210	1·641	1·642	*m–s*	8·0	225	1·170	1·170	*m–w*	5·4
211		1·635		1·7	0,0,14	1·162	1·163	*m–w*	*n.c.*
0,0,10	1·627	1·628	*m*	6·4	1,2,10	1·156	1·156	*m*	n.c.
1,0,10	1·528	1·525	*w*	1·3	2,0,12	1·150	1·151	*m*	n.c.
125	1·466	1·466	*m*	3·6	226	1·139	1·138	*w*	2·5
300	1·446	1·448	*m*	5·1	135	1·130	1·130	*w–m*	3·4
209	1·391	1·390	*w–m*	2·8	1,0,14	1·122	1·123	*m*	n.c.

n.c. = not calculated

---

* This work was performed in the Air Force Materials Laboratory, Materials Physics Division (MAYA), under Project No. 7360.