

SHORT COMMUNICATIONS

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Acta Cryst. (1990). **C46**, 1356–1357

1:1 Molecular complex of acridine with 1,2,4,5-benzenetetracarbonitrile: refinement in space group $P\bar{1}$. By RICHARD E. MARSH,* A. A. Noyes Laboratory of Chemical Physics, California Institute of Technology, Pasadena, California 91125, USA

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Abstract

The crystal structure of $C_{13}H_9N.C_{10}H_2N_4$, which was originally described and refined in space group $P1$ [triclinic; $a = 7.447$ (4), $b = 7.885$ (5), $c = 8.072$ (9) Å, $\alpha = 73.93$ (9), $\beta = 84.59$ (9), $\gamma = 85.85$ (6)°, $Z = 1$; Toupet, Miniewicz & Ecolivet (1989). *Acta Cryst.* **C45**, 1044–1047], is better described in $P\bar{1}$. The $P\bar{1}$ description entails disorder, with the N and CH groups of the central ring of the acridine molecule being indistinguishable. Revised coordinates are given.

Toupet *et al.* (1989) (hereinafter TME) noted that some of the bond lengths 'are not in good agreement with expected values', and suggested this was due to disorder. They attempted to refine a 'static'-disordered model in space group $P\bar{1}$ (this disorder involves interchange of atoms N1 and C7 in the central ring of the acridine molecule), but reported unsatisfactory R and e.s.d. values; they then suggested that the disorder was associated with large 'thermal' motions, particularly for the acridine molecule.

I have had no problem in refining the static-disordered $P\bar{1}$ model. Observed structure factors were recovered from SUP 51832, and full-matrix refinement of the symmetrized structure routinely converged at an R of 0.039 for 1240 reflections and 152 parameters [including one for extinction; final value 16.6 (3) $\times 10^{-6}$]; for the $P1$ model, TME reported an R of 0.047 for 1241 reflections and 251 parameters. (I deleted one reflection, $17\bar{5}$, as a statistically improbable outlier; it showed equally poor agreement between F_{obs} and F_{cal} for the $P1$ model.) The $P\bar{1}$ parameters are given in Table 1.† Refinement was by full-matrix minimization of the quantity $\sum w(F_{obs}^2 - F_{cal}^2)^2$, with weights

Table 1. Coordinates ($\times 10^4$) and U_{eq} ($\times 10^4$) or B values, space group $P\bar{1}$

$$U_{eq} = (1/3)\sum_i \sum_j U_{ij} a_i^* a_j^* a_i a_j$$

	x	y	z	$U_{eq}(\text{\AA}^2)$ or $B(8\pi^2\text{\AA}^2)$
N1,C7	103 (2)	1340 (3)	799 (3)	666 (5)
C1,8	572 (2)	-359 (3)	1636 (2)	612 (5)
C2,9	1192 (3)	-814 (5)	3332 (3)	969 (9)
C3,10	1639 (4)	-2485 (8)	4129 (5)	1377 (15)
C4,11	1551 (4)	-3843 (6)	3355 (6)	1368 (15)
C5,12	987 (3)	-3499 (4)	1740 (5)	983 (9)
C6,13	470 (2)	-1725 (3)	821 (3)	614 (5)
N2,4	6479 (2)	-1839 (2)	4801 (2)	721 (5)
N3,5	6371 (3)	-5101 (2)	1833 (2)	747 (5)
C21,23	6074 (2)	-1312 (2)	3419 (2)	512 (4)
C20,22	5963 (2)	-3641 (2)	1307 (2)	523 (4)
C15,18	5461 (2)	-1788 (2)	637 (2)	439 (4)
C16,19	5536 (2)	-637 (2)	1673 (2)	432 (4)
C14,17	5074 (2)	1149 (2)	1032 (2)	477 (4)
HC7	169 (51)	2297 (52)	1385 (50)	6.6 (10)*
H2,9	1287 (41)	278 (42)	3756 (40)	13.4 (12)*
H3,10	2121 (39)	-2851 (41)	5340 (42)	13.6 (10)*
H4,11	1916 (37)	-5035 (40)	3993 (38)	11.9 (9)*
H5,12	883 (33)	-4311 (33)	1073 (32)	8.7 (8)*
H14,17	5138 (21)	1893 (21)	1722 (21)	4.3 (4)*

* Isotropic displacement parameter, B .

w according to Hughes (1941; see also Marsh & Schomaker, 1979).

The revised bond lengths are quite satisfactory, and there are no significant disparities between chemically equivalent values. As noted by TME, the U_{ij} values correspond to a large in-plane libration of the acridine molecule; the implied r.m.s. amplitude is about 8°. This large apparent libration suggests that a low-temperature analysis might well show a 'freezing-out' of the acridine molecules, into two or more discrete orientations; TME report that they are planning such a study. But it is also possible that the crystals might undergo a phase transition, into an ordered arrangement of acridine molecules.

One final comment: the situation regarding the disordered acridine molecule is, in effect, a reprise. The structure of the 1:1 complex of acridine with N,N' -dimethyldiimide was also first described in a non-

* Contribution No. 8350 from the A. A. Noyes Laboratory.

† Tables of U_{ij} 's and bond lengths and angles and thermal-ellipsoid plots of the molecules with atom numbering have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52572 (5 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

centrosymmetric space group, *Pn* (Yamaguchi & Ueda, 1984), and was later refined more satisfactorily as a disordered model in the centrosymmetric *P2/n* (Marsh, 1986); the disordered model again required that the central N and CH groups of the acridine molecule be equivalent. And the conclusion is the same: on the basis of the diffraction data, there is little choice but to opt for the centrosymmetric, disordered description.

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Structure of 1,2,5,6-tetramethyltricyclo[3.1.0.0^{2,6}]hexane-3,4-dione. By A. L. SPEK AND P. VAN DER SLUIS, *Vakgroep Algemene Chemie, Afdeling Kristal- en Structuurchemie, University of Utrecht, Padualaan 8, 3584 CH Utrecht, The Netherlands*

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Abstract

The crystal structure of the title compound, which was originally described in the non-centrosymmetric space group *Pnc2* [Spek (1977). *Cryst. Struct. Commun.* **6**, 259–262], has been refined successfully in the centrosymmetric space group *Pnca*. The molecular geometry has been improved significantly. The cell parameters are: $a = 12.084$ (2), $b = 11.295$ (2), $c = 6.659$ (2) Å, with $R = 0.049$.

The structure of the title compound ($C_{10}H_{12}O_2$, $M_r = 164.2$, $\mu = 0.9 \text{ cm}^{-1}$) has been reported (Spek, 1977) in space group *Pnc2* [CAD-4, Zr-filtered Mo $K\alpha$, $\theta_{\text{max}} = 20^\circ$, 341 reflections with $I > 2.5(I)$, $R = 0.08$]. Molecules are located in this space group on crystallographic twofold axes resulting in two independent half molecules. Examination of the symmetry of the refined structural parameters with the *MISSYM* algorithm (Le Page, 1987) as implemented in the program *PLATON* (Spek, 1982) revealed an additional inversion centre at 0.254, -0.509 , -0.244 . This inversion centre relates the previously reported two independent half molecules. The correct space group is *Pnca* (non-standard setting of *Pbcn*) [$a = 12.084$ (2), $b = 11.295$ (2), $c = 6.659$ (2) Å, $Z = 4$]. The structure was refined on *F* by full-matrix least squares with *SHELX76* (Sheldrick, 1976). Hydrogen atoms on the methyl groups were included at calculated positions ($C-H = 0.98$ Å) and refined as rigid groups with two common isotropic thermal parameters. Convergence was reached at $R = 0.049$ ($wR = 0.077$, $w^{-1} = \sigma^2(F) + 0.0002F^2$, $S = 0.58$, 333 reflections [$I > 2.5\sigma(I)$], 63 parameters, $(\Delta/\sigma)_{\text{max}} = 0.3$). No residual density outside $\pm 0.15 \text{ e } \text{Å}^{-3}$. Final parameters are given in Table 1.* Fig. 1 gives the atom numbering. The additional

* Lists of structure factors, anisotropic thermal parameters, H-atom parameters, bond distances, bond angles and torsion angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52626 (6 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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 YAMAGUCHI, Y. & UEDA, I. (1984). *Acta Cryst.* **C40**, 113–115.

Table 1. Final coordinates, space group *Pnca*, and equivalent isotropic thermal parameters, with their e.s.d.'s in parentheses

$$U_{\text{eq}} = \frac{1}{3} \sum_i \sum_j U_{ij} a_i^* a_j^* a_i a_j$$

	<i>x</i>	<i>y</i>	<i>z</i>	U_{eq} (Å ²)
O(1)	0.3524 (3)	0.0732 (3)	0.2877 (6)	0.070 (2)
C(2)	0.3031 (3)	0.0390 (4)	0.1373 (9)	0.049 (2)
C(3)	0.3261 (3)	0.0566 (3)	−0.0746 (8)	0.046 (2)
C(4)	0.2841 (3)	−0.0524 (3)	−0.1908 (7)	0.047 (2)
C(5)	0.4230 (4)	0.1282 (4)	−0.1511 (8)	0.065 (2)
C(6)	0.3377 (4)	−0.1373 (4)	−0.3320 (7)	0.067 (2)

Table 2. Bond distances (Å), space group *Pnca*

Primed atoms are related by $\frac{1}{2} - x, -y, z$.

O(1)—C(2)	1.228 (7)	C(3)—C(4')	1.540 (6)
C(2)—C(2')	1.557 (6)	C(3)—C(5)	1.512 (6)
C(2)—C(3)	1.452 (8)	C(4)—C(4')	1.442 (5)
C(3)—C(4)	1.540 (6)	C(4)—C(6)	1.491 (6)

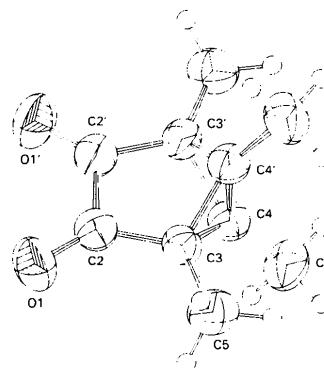


Fig. 1. Thermal motion ellipsoid plot (50% probability level) showing the twofold crystallographic symmetry and atom labelling.