Table 3. *Classification of bond lengths and angles*

(*a*) Mean bond lengths classified according to degree of substitution (Å)

$CH-CH_2$	1·524 (2)	$C-CH_2$	1·528 (3)	CH–CH	1·537 (2)	C–CH	1·542 (2)

(*b*) Mean valency angles classified according to degree of substitution of the three C atoms (°)

CH–C–CH	107·4 (2)	C–CH–CH	109·6 (1)	$C-CH_2-CH$	111·0 (2)
$CH-C-CH_2$	109·4 (1)	CH–CH–CH (eq.)	108·2 (1)	$CH-CH_2-CH$	109·4 (1)
CH_2-C-CH_2	111·8 (2)	(axial)	111·4 (1)		
		$CH-CH-CH_2$	110·6 (1)		
		$CH_2-CH-CH_2$	109·4 (1)		

between valence angle and the nature of the C substituents alone.

We thank Professor M. A. McKervey of the University of Cork for the crystals and for initiating our interest in this work. We also thank the National Research Council of Canada for a grant (to GF).

References

Burns, W., Mitchell, T. R. B., McKervey, M. A., Rooney, J. J., Ferguson, G. & Roberts, P. (1976). *Chem. Commun.* pp. 893–895.

Sheldrick, G. M. (1976). *SHELX*-76 program system for crystal structure determination. University Chemical Laboratory, Cambridge, England.

Acta Cryst. (1977). B**33**, 2337–2339

p-Nitrobenzonitrile

By Tsuneyuki Higashi and Kenji Osaki

Faculty of Pharmaceutical Sciences, Kyoto University, Kyoto 606, *Japan*

(*Received* 10 *February* 1977; *accepted* 9 *March* 1977)

Abstract. $C_7H_4N_2O_2$, monoclinic, $P2_1$, $a = 12{\cdot}732$ (3), $b = 7{\cdot}058$ (3), $c = 3{\cdot}847$ (2) Å, $\beta = 93{\cdot}670$ (2)°, $D_m = 1{\cdot}430$, $D_c = 1{\cdot}415$ g cm^{-3}, $Z = 2$. The final R value was 0·086 for 708 independent reflexions measured photographically. The molecular dimensions are normal. There is no abnormally short intermolecular contact, but the arrangement of the molecules suggests that there is some contribution from Coulombic interactions, in addition to those involving local dipoles, to the molecular packing.

Introduction. As part of the project to study the effect of polar groups on molecular packing in crystals, the crystal structure of *p*-nitrobenzonitrile was analysed since the values of the dipole moment of benzonitrile and nitrobenzene, both equal to about 4 D, suggested a large contribution from dipolar interactions due to the cyano and nitro groups. Pale-yellow, plate-like crystals elongated along **b** were grown from an ether solution.

Reflexion data were visually estimated from equi-inclination Weissenberg photographs taken around the b and c axes with Ni-filtered Cu $K\alpha$ radiation. Application of the usual corrections gave 708 independent F values. No absorption correction was applied [μ(Cu $K\alpha$) = 9·24 cm^{-1}]. The space group is either $P2_1$ or $P2_1/m$ from the systematic absence: $0k0$, $k = 2n + 1$. The choice of $P2_1$ was verified through successful analysis.

The structure was solved by two-dimensional R-index maps based on the relative orientation of the phenyl ring, obtained from a sharpened Patterson synthesis. The model was refined first by a constrained least-squares method, and subsequently by a full-matrix least-squares method with anisotropic thermal factors, to the final R value of 0·086. The H atoms were included in the structure factor calculations with calculated positions. All the computations were carried

Table 1. *Final positional parameters* (×10⁴) *of the non-hydrogen atoms*

	x	y	z
C(1)	1933 (5)	0	2884 (16)
C(2)	3010 (5)	78 (15)	3794 (17)
C(3)	3573 (5)	1685 (15)	3030 (18)
C(4)	3045 (5)	3162 (14)	1304 (16)
C(5)	1973 (6)	3102 (15)	361 (17)
C(6)	1411 (5)	1516 (15)	1185 (18)
C(7)	1343 (6)	−1648 (18)	3761 (21)
N(8)	895 (5)	−2963 (15)	4501 (19)
N(9)	3653 (5)	4887 (14)	537 (16)
O(10)	4556 (5)	5038 (16)	1760 (19)
O(11)	3226 (5)	6041 (14)	−1377 (19)

out on the FACOM 230-75 computer at the Data Processing Center, Kyoto University, with the programs of the *KPAX* system, which includes *UNICS* programs. The atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1974). The final atomic parameters are given in Table 1.* Bond lengths and angles are shown in Fig. 1. A stereoscopic view of the molecular packing is shown in Fig. 2.

Discussion. The benzene ring is planar within experimental errors, but both cyano and nitro groups deviate significantly from the plane to the same side (Table 2). The nitro group is rotated through 10·3° out of the benzene plane. Other molecular dimensions are normal within the standard deviations; C(1)–C(7), C(7)–N(8), and C(4)–N(9) bonds are 1·438 (12), 1·155 (15), and 1·483 (13) Å, respectively, and are in good agreement with generally accepted values, 1·44 (1), 1·158 (2), and 1·475 (10) Å respectively (*Molecular Structures and Dimensions*, 1972). The average length of the central C–C bonds of the benzene ring parallel to the long molecular axis, 1·380 Å, is shorter by almost one standard deviation than the average of all six ring bonds. However, contribution of a quinonoid resonance structure, as found in p-cyanophenol (Higashi & Osaki, 1977), is not possible in this molecule because both its *para* substituents are electron-withdrawing, therefore preventing occurrence of the cooperative effect found between electron-donating and electron-withdrawing groups (Mak & Trotter, 1965). The shortening of the central bonds found here may be similar to that in terephthalic acid (Bailey & Brown, 1967) or in p-nitrobenzoic acid (Tavale & Pant, 1971).

The molecules are stacked along **c**, the shortest period, with a perpendicular distance of 3·30 Å. Also, there is a belt-shaped, almost planar, chain of molecules, limited by the planes at $x = 0$ and $\frac{1}{2}$, which runs infinitely along $[01\bar{1}]$ because of some degree of tilt of the molecular plane from the *ab* plane.

The short intermolecular distances, shown in Table 3, are comparable with the sum of the van der Waals radii. O···N, O···C and C···N contacts predominate while N···N, O···O, or H···H distances are relatively long. The shortest contact, O(10)···C(3) 3·24 Å, results from O(10) approaching the edges of the benzene rings related by screw axes at $x = \frac{1}{2}$. Similarly, the cyano N(8) at the opposite end of the molecule approaches the edges of the benzene rings

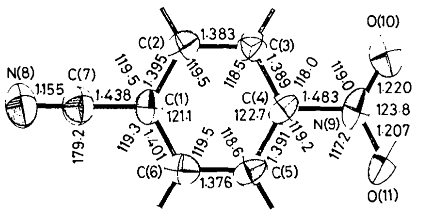

Fig. 1. An *ORTEP* drawing (Johnson, 1965) of p-nitrobenzonitrile showing bond lengths and angles (e.s.d.'s are 0·010–0·015 Å for lengths and 0·6–1·0° for angles).

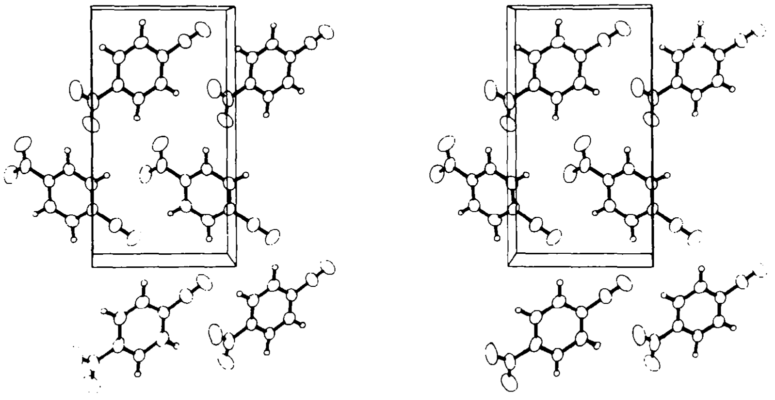

Fig. 2. Stereoscopic view of the crystal structure viewed along **c**; **a** is vertical and **b** horizontal. The two molecules at the bottom of the figure represent part of a belt-shaped chain.

Table 2. *Deviations* (Å) *from the least-squares plane of the benzene ring*

The plane equation, referred to orthogonal axes a^*, b and c, is $0{\cdot}2495X - 0{\cdot}4034Y - 0{\cdot}8803Z + 0{\cdot}3865 = 0$, where X, Y, Z are in Å.

C(1)	0·001	C(4)	0·002	C(7)	−0·021	O(10)	−0·210
C(2)	0·006	C(5)	0·005	N(8)	−0·054	O(11)	0·169
C(3)	−0·008	C(6)	−0·007	N(9)	−0·030		

---

* Lists of structure factors and thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 32565 (7 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

Table 3. *Intermolecular distances shorter than* 3·6 Å

Roman numerals represent equivalent points, (i) x,y,z; (ii) $-x$, $\frac{1}{2}+y$, $-z$; and the other symbols represent cell translations.

O(10)···C(3, ii + a + c)	3·24 Å
O(10)···C(3, ii + a)	3·32
O(10)···C(2, ii + a + c)	3·45
O(11)···N(9, i − c)	3·32
O(11)···O(10, i − c)	3·32
O(11)···C(4, i − c)	3·48
O(11)···C(7, i + b − c)	3·37
O(11)···N(8, i + b − c)	3·39
O(11)···C(2, i + b − c)	3·41 Å
O(11)···C(2, i + b)	3·51
N(8)···C(6, ii − b + c)	3·46
N(8)···C(6, ii − b)	3·55
N(8)···C(5, i − b)	3·54
C(3)···C(4, i + c)	3·48
C(1)···C(6, i + c)	3·49

related by screw axes at $x = 0$. In the belt-shaped chain, O(11) and the cyano C(7) of the successive molecules are arranged closely together, while in the molecular column along **c** O(11) and the nitro N(9) are in close contact.

From the viewpoint of intermolecular interactions, it is worth noting that there is no close contact suggesting dipole–dipole interactions between cyano groups or between nitro groups. But there is an anti-parallel close contact between the cyano and nitro groups of successive molecules within the belt-shaped chain, suggesting interactions between local dipoles.

The molecular arrangement described above seems better explained by Coulombic interactions, if we assume some residual charge on each atom: negative charges on O and N(cyano), positive charges on N(nitro), C and H. It may also be added that this assumption is supported qualitatively by a CNDO/2 calculation.

References

Bailey, M. & Brown, C. J. (1967). *Acta Cryst.* **22**, 387–391.

Higashi, T. & Osaki, K. (1977). *Acta Cryst.* B**33**, 607–609.

International Tables for X-ray Crystallography (1974). Vol. IV, p. 72. Birmingham: Kynoch Press.

Johnson, C. K. (1965). *ORTEP.* Report ORNL-3794. Oak Ridge National Laboratory, Tennessee.

Mak, T. C. W. & Trotter, J. (1965). *Acta Cryst.* **18**, 68–74.

Molecular Structures and Dimensions (1972). Vol. A1, p. S2. Utrecht: Oosthoek.

Tavale, S. S. & Pant, L. M. (1971). *Acta Cryst.* B**27**, 1479–1481.

Acta Cryst. (1977). B**33**, 2339–2342

Perfluorododecahydrotetracyclopentacyclooctene

By R. E. Cobbledick and F. W. B. Einstein

Department of Chemistry, Simon Fraser University, Burnaby, BC, Canada V5A 1S6

(*Received* 5 *March* 1977; *accepted* 19 *March* 1977)

Abstract. $C_{20}F_{24}$, $M_r = 696\cdot2$, monoclinic, space group $C2/c$, $a = 15\cdot368$ (7), $b = 15\cdot326$ (7), $c = 10\cdot066$ (4) Å, $\beta = 105\cdot75$ (3)°, $V = 2281\cdot8$ Å^3, $Z = 4$, $D_c = 2\cdot026$, $D_o = 2\cdot01$ g cm^{-3}. The structure was solved by direct methods from Mo $K\alpha$ X-ray diffractometer intensities and refined by full-matrix least squares to a final R of 0·077 for 1200 observed reflexions (assuming space group $C2/c$). The molecule has crystallographic twofold symmetry and approximate $\bar{4}2m$ symmetry with the cyclooctatetraene ring in a boat conformation.

Introduction. Initial unit-cell dimensions were determined from photographs (Cu $K\alpha$ radiation) and absent reflexions hkl when $(h + k) = 2n + 1$ and $h0l$ when $l = 2n + 1$ indicated the space group to be either Cc or $C2/c$. Crystals sublimed at room temperature and were sealed in Lindemann-glass capillaries. Accurate cell dimensions were obtained by least-squares refinement of the setting angles of nine reflexions with 2θ values in the range 35–41°, centred on a Picker FACS-I four-circle diffractometer [λ(Mo $K\alpha_1$) = 0·70926 Å]. The crystal used for intensity measurements was a rectangular parallelepiped 0·18 × 0·30 × 0·25 mm and was mounted on the diffractometer with **b** off-set from the φ axis. Nb-filtered Mo radiation and a scintillation counter with pulse-height analysis were used. A θ–2θ scan was used with a scan speed of 2° min^{-1} and a scan width of 1·4° for reflexions with $2\theta < 35°$ and