Fig. 2. Crystal packing as viewed down the *b* axis. The water oxygens are numbered; broken lines represent hydrogen bonding.

References

ALTONA, C. & SUNDARALINGAM, M. (1972). *J. Am. Chem. Soc.* **94**, 8205–8212.

BROWN, J. N. & TELLER, R. G. (1976). *J. Am. Chem. Soc.* **98**, 7665–7669.

BROWN, J. N. & YANG, C.-H. (1979). *J. Am. Chem. Soc.* **101**, 455–459.

CHIANG, C. A., KARLE, I. L. & WIELAND, TH. (1982). *Int. J. Pept. Protein Res.* **20**, 414–420.

HASNOOT, C. A. G., DELEEUW, F. A. A. M., DELEEUW, H. P. M. & ALTONA, C. (1981). *Biopolymers,* **20**, 1211–1245.

International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor D. Reidel, Dordrecht.)

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

KARLE, I. L. (1975). *J. Am. Chem. Soc.* **97**, 4379–4386.

KARLE, I. L. (1976). *Macromolecules,* **9**, 61–66.

KARLE, I. L. (1977). *J. Am. Chem. Soc.* **99**, 5152–5157.

KARLE, I. L. & CHIANG, C. C. (1984). *Acta Cryst.* C**40**, 1381–1386.

KARLE, I. L. & DUESLER, E. (1977). *Proc. Natl Acad. Sci. USA,* **74**, 2602–2606.

KARLE, I. L. & FLIPPEN-ANDERSON, J. L. (1978). *Acta Cryst.* B**34**, 3237–3241.

KARTHA, G., BHANDARY, K. K., KOPPLE, K. D., ZHU, P.-P. & GO, A. (1984). *J. Am. Chem. Soc.* **106**, 3844–3850.

KOSTANSEK, E. C., LIPSCOMB, W. N. & THIESSEN, W. E. (1979). *J. Am. Chem. Soc.* **101**, 834–837.

MAIN, P., FISKE, S. J., HULL, S. E., LESSINGER, L., GERMAIN, G., DECLERCQ, J.-P. & WOOLFSON, M. M. (1980). *MULTAN80. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data.* Univs. of York, England, and Louvain, Belgium.

NORTH, A. C. T., PHILLIPS, D. C. & MATHEWS, F. C. (1968). *Acta Cryst.* A**24**, 351–359.

SMITH, G. D., DUAX, W. L., LANGS, D. A., DETITTA, G. F., EDMOND, J. W., ROHRER, D. C. & WEEKS, C. M. (1975). *J. Am. Chem. Soc.* **97**, 7242–7247.

Structure of Bis(*p*-nitrophenyl) Diselenide at 187 K

BY GERALD D. MORRIS AND F. W. B. EINSTEIN*

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

Abstract. $C_{12}H_8N_2O_4Se_2$, $M_r = 402 \cdot 1$, monoclinic, $C2/c$, $a = 7 \cdot 2771$ (12), $b = 14 \cdot 142$ (3), $c = 12 \cdot 873$ (3) Å, $\beta = 90 \cdot 51$ (2)°, $V = 1324 \cdot 7$ Å^3, $Z = 4$, $D_x = 2 \cdot 02$ g cm^{-3}, Mo $K\alpha$ radiation, $\lambda = 0 \cdot 70930$ Å, $\mu = 55 \cdot 37$ cm^{-1}, $F(000) = 776$, $T = 187$ (1) K, $R = 0 \cdot 023$ for 1104 observed reflections. The molecule has a twofold crystallographic symmetry axis passing through the Se—Se bond [$2 \cdot 3018$ (8) Å]. The phenyl group is asymmetrically coordinated with Se—C—C angles of $123 \cdot 7$ (2) and $115 \cdot 6$ (2)°, and the phenyl ring plane passes $0 \cdot 105$ (5) Å from the Se atom [Se—C $1 \cdot 920$ (3) Å].

Introduction. The structure of the title compound was determined at reduced temperature, to provide high-precision bond lengths and angles prior to attempts to study the anomeric effect in selenium compounds.

Experimental. A single crystal measuring $0 \cdot 020 \times 0 \cdot 021 \times 0 \cdot 014$ cm was mounted within a capillary. The crystal was pale yellow in color and showed sharp extinction in polarized light. Reduced temperature was obtained using a modified Nonius Universal Low Temperature apparatus. After cooling from room temperature over a period of 4 h, low temperature was maintained until the end of data collection to avoid stressing the crystal more than necessary. Cell dimensions were determined by least-squares fit of 25 reflections ($14 \cdot 4 < \theta < 22 \cdot 4°$) using Mo $K\alpha$ radiation with graphite monochromator. Intensity data were collected on an Enraf–Nonius CAD-4F diffractometer using ω–2θ scans. 1526 independent reflections measured with 1104 considered observed with [$I/\sigma(I)$] $> 2 \cdot 5$; $h\,0 \to 9$, $k\,0 \to 18$, $l\,{-}16 \to 16$; $2\theta_{max} = 55°$. Two reflections were used as intensity standards which were measured every hour. Variations in intensity of these standards were less than $\pm 2\%$ (3σ) and

---

Table 1. *Atomic parameters and equivalent isotropic thermal parameters*

$$B_{eq} = \frac{8\pi^2}{3}(U_{11} + U_{22} + U_{33}).$$

	x	y	z	$B_{eq}(\text{Å}^2)$
Se	0·41430 (4)	0·31237 (2)	0·17443 (2)	1·81 (1)
C(1)	0·5313 (4)	0·2171 (2)	0·0917 (2)	1·6 (1)
C(2)	0·6365 (4)	0·1441 (2)	0·1330 (3)	1·9 (1)
C(3)	0·7078 (4)	0·0755 (2)	0·0680 (3)	2·0 (1)
C(4)	0·6716 (4)	0·0819 (2)	−0·0372 (2)	1·8 (1)
C(5)	0·5682 (4)	0·1542 (2)	−0·0799 (3)	2·0 (1)
C(6)	0·4986 (4)	0·2229 (2)	−0·0146 (2)	1·9 (1)
O(1)	0·8261 (4)	−0·0585 (2)	−0·0673 (2)	3·6 (1)
O(2)	0·7191 (4)	0·0183 (2)	−0·2003 (2)	3·2 (1)
N	0·7438 (4)	0·0086 (2)	−0·1065 (2)	2·3 (1)
H(2)	0·661 (4)	0·145 (2)	0·201 (3)	1·9
H(3)	0·784 (4)	0·025 (2)	0·095 (3)	2·0
H(5)	0·542 (4)	0·158 (2)	−0·150 (3)	1·9
H(6)	0·430 (4)	0·274 (2)	−0·044 (3)	1·8

were not systematic with time. Lorentz effect and crystal and monochromator polarization were corrected during data reduction. An analytical absorption correction (checked against ψ scans of four reflections) was applied (transmission factor τ 0·264–0·524). The structure was solved by Patterson and Fourier methods. Initial H-atom positions were calculated after refinement of the non-H atoms. Counter weights were applied, $w = [\sigma^2(F_o) + k(F_o)^2]^{-1}$, $k = 0·0003$. The weighting scheme was optimized by adjusting the value of k to obtain the most uniform average $\Sigma w(F_o - F_c)^2$ over segments of data grouped by $\sin\theta$ and $|F_o|$. The final difference map's largest feature was a peak of 0·47 (6) e Å^{-3}. The five largest peaks were all positive. Refined parameters were positions of all atoms, anisotropic thermal motion of all non-H atoms, scale factor and extinction [1·6 (3) ×10^{-3}]. 104 variables for the 14 crystallographically distinct atoms. $R = 0·023$, $wR = 0·029$, all (Δ/σ) were less than 0·005 in the last least-squares cycle. Scattering factors, anomalous-dispersion coefficients for all non-H atoms and mass-attenuation coefficients used were those listed in *International Tables for X-ray Crystallography* (1974). All calculations were made using the NRC VAX software package (Gabe, Larson, Lee & Le Page, 1984). Positions of atoms and mean thermal-motion parameters are listed in Table 1. Important interatomic distances and angles are given in Table 2.*

Discussion. The molecule has twofold symmetry with the axis along the line $0y\frac{1}{4}$ (Fig. 1). Comparison of the Se—Se—C(1) bond angle, 103·24 (9)°, of this structure with the corresponding angle in the pentafluorophenyl

analogue, 98·7 (6)° (Woodard, Brown, Lee & Massey, 1976), indicates a shift in bond character consistent with Bent's rule (Bent, 1961).

The C atoms deviate up to 0·007 (4) Å from the best plane through one phenyl ring. The dihedral angle between the plane O(1)—N—O(2) and the phenyl plane is 4·1 (1)°. The torsion angle C(1)′—Se′—Se—C(1) is −87·8 (1)°. The Se atom is 0·105 (5) Å from the phenyl plane; the N atom is displaced 0·025 (5) Å on the same side as the Se. The Se—C(1) bond is

Table 2. *Important interatomic distances (Å) and angles (°)*

	Uncorrected	Rigid-body corrected*
Se—Se	2·3018 (8)	2·3024
Se—C(1)	1·920 (3)	1·922
C(1)—C(2)	1·388 (4)	1·390
C(2)—C(3)	1·386 (5)	1·387
C(3)—C(4)	1·381 (4)	1·382
C(4)—C(5)	1·381 (4)	1·383
C(5)—C(6)	1·383 (5)	1·384
C(6)—C(1)	1·390 (4)	1·390
C(2)—H(2)	0·90 (4)	—
C(3)—H(3)	0·97 (3)	—
C(5)—H(5)	0·92 (3)	—
C(6)—H(6)	0·96 (3)	—
C(4)—N	1·467 (4)	1·468
N—O(1)	1·228 (4)	1·230
N—O(2)	1·227 (3)	1·228

Se—C(1)—C(2)	123·7 (2)	Se—C(1)—C(6)	115·6 (2)
Se—Se—C(1)	103·24 (9)	C(6)—C(1)—C(2)	120·7 (3)
C(1)—C(2)—C(3)	119·9 (3)	C(2)—C(3)—C(4)	118·5 (3)
C(3)—C(4)—C(5)	122·5 (3)	C(4)—C(5)—C(6)	118·7 (3)
C(5)—C(6)—C(1)	119·8 (3)	C(3)—C(4)—N	118·9 (3)
C(5)—C(4)—N	118·6 (3)	C(4)—N—O(1)	118·2 (3)
C(4)—N—O(2)	117·9 (3)	O(1)—N—O(2)	123·9 (3)
C(1)′—Se′—Se—C(1)	−87·8 (1)	Se′—Se—C(1)—C(2)	23·3 (3)
Se′—Se—C(1)—C(6)	20·9 (2)		

* Thermal-motion correction treating the whole molecule as a rigid body. Weighted R of anisotropic U_{ij} 0·094; r.m.s. ΔU_{ij}^2 0·0014.
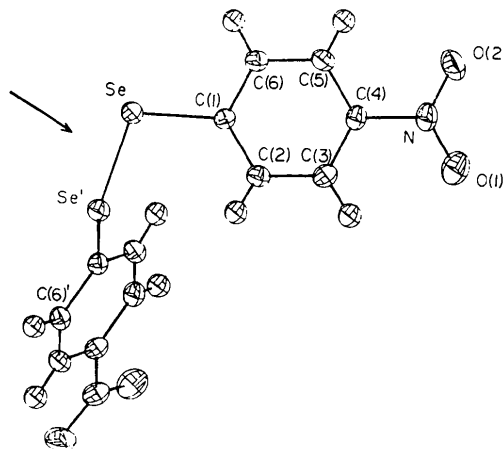

Fig. 1. *SNOOPI* (Davies, 1984) drawing of the molecule. The thermal ellipsoids represent 50% probability surfaces. The arrow indicates the twofold axis.

asymmetric in that the Se—C(1)—C(2) and Se—C(1)—C(6) bond angles are 123·7 (2) and 115·6 (2)°, respectively, and the Se—C bond is out of the phenyl plane. Studies of phenoxyalkanoic acid derivatives (Kennard, Smith & White, 1982) and hexakis-(methylthio)benzene, $C_6(SCH_3)_6$ (Peach & Burschka, 1982), showed similar behavior in O— and S—phenyl bonds. For O and S the deviation from the expected trigonal arrangement was thought to be due to crowding, but this work leads us to conclude that very weak forces can cause this type of distortion, hence the change is not chemically significant.

Analysis of the thermal-motion parameters (*THMV*9, Trueblood, 1985; Rosenfield, Trueblood & Dunitz, 1978) was carried out in an attempt to identify librating groups of atoms. Neither the nitro group nor the phenyl ring was indicated to be librating separately from the rest of the molecule. The relative motions of atoms in different groups of the molecule were not significantly greater than those within a group, hence separately librating groups were not suggested by the analysis. Interatomic distances corrected for thermal motion were calculated treating the whole molecule as a rigid body and are listed in Table 2. The corrections were all less than σ.

A weak intermolecular H bond may exist between H(6) and O(1). The H—O distance is 2·45 (4) Å, the O(1)—H(6)—C(6) angle 157 (3)° and the N—O(1)—H(6) angle 158 (1)°. The interacting molecules are related by the C centering.

References

BENT, H. A. (1961). *J. Chem. Phys.* **33**, 1259–1260.
DAVIES, E. K. (1984). *SNOOPI* plot program. Chemical Crystallography Laboratory, Oxford Univ.
GABE, E. J., LARSON, A. C., LEE, F. L. & LE PAGE, Y. (1984). *NRC VAX Crystal Structure System.* National Research Council of Canada, Ottawa.
International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor D. Reidel, Dordrecht.)
KENNARD, C. H. L., SMITH, G. & WHITE, A. H. (1982). *Acta Cryst.* B38, 868–875.
PEACH, M. E. & BURSCHKA, C. (1982). *Can. J. Chem.* 60, 2029–2037.
ROSENFIELD, R. E. JR, TRUEBLOOD, K. N. & DUNITZ, J. D. (1978). *Acta Cryst.* A34, 828–829.
TRUEBLOOD, K. N. (1985). *THMV*9 thermal-motion-analysis program. Private communication.
WOODARD, C. M., BROWN, D. S., LEE, J. D. & MASSEY, A. G. (1976). *J. Organomet. Chem.* 121, 333–344.

Structure of 3-Benzyl-2-methylindole

BY D. K. CHAKRABORTY AND S. K. TALAPATRA*

X-ray Laboratory, Department of Physics, Jadavpur University, Calcutta 700032, India

Abstract. $C_{16}H_{15}N$, $M_r = 221·3$, monoclinic, $P2_1/n$, $a = 12·458$ (1), $b = 5·904$ (1), $c = 17·293$ (2) Å, $\beta = 102·75$ (1)°, $V = 1240·6$ (3) Å^3, $Z = 4$, $D_m = 1·180$, $D_x = 1·184$ g cm^{-3}, $\lambda(Mo\ K\alpha) = 0·7107$ Å, $\mu = 0·56$ cm^{-1}, $F(000) = 472$, $T = 296$ K, $R = 0·054$ for 1084 observed reflections. The indole system is nearly planar. The methyl group is slightly out of the plane of the indole system. The substituent benzene ring makes a dihedral angle of 79·8 (1)° with the pyrrole ring.

Introduction. Indole and its various substituted products have long been known for their interesting chemical and biological activities. Our interest in the structural properties of 3-benzyl-2-methylindole is twofold: (1) its structural study might aid in the elucidation of its mechanistic action; (2) there is, somewhat surprisingly, relatively little knowledge about the crystal chemistry of non-steroidal indole-based derivatives. As an aid in understanding the above mentioned mechanistic studies and to provide further structural data on substituted indoles, we have investigated the solid-state structure of the title compound by X-ray diffraction methods and give a full report of the results here.

Experimental. Single crystals of the title compound obtained by slow evaporation of the powder substance in chloroform. The approximate dimensions of the

---

* To whom correspondence should be addressed.