structure of N-(2-hydroxyethyl)-2-thiofuramide, approximately planar, deviating from planarity within ±0·049 (5) Å. Consequently, the torsion angles along the C(1)—C(2) and C(1)—N bonds are close to 0 or 180°. The intramolecular contact N···O(1) of 2·727 (4) Å with the H(11)···O(1) separation of 2·36 (4) Å might be considered as a hydrogen bond in spite of the relatively small N—H(11)···O(1) angle of 110 (4)°.

There are N—H···O intermolecular hydrogen bonds of 2·872 (5) Å with an H···O separation of 2·17 (4) Å and N—H···O angles of 150 (4)°. They connect the molecules in chains parallel to the *c* axis (Fig. 1).

The authors thank M. Bruvo MSc for collecting the diffractometer data. This investigation was supported by the Research Council of Croatia.

References

Auerbach, C. & Moser, H. (1950). *Nature (London)*, **166**, 1019–1020.

Bodenstein, D. & Goldin, A. (1948). *J. Exp. Zool.* **108**, 75–91.

Cromer, D. T. & Liberman, D. (1970). *J. Chem. Phys.* **53**, 1891–1898.

Cromer, D. T. & Mann, J. B. (1968). *Acta Cryst.* A**24**, 321–324.

Denny, W. A. & Wilson, W. R. (1986). *J. Med. Chem.* **29**, 879–887.

Domenicano, A., Vaciago, A. & Coulson, C. A. (1975). *Acta Cryst.* B**31**, 221–234.

Dustin, P. (1947). *Nature (London)*, **159**, 794–797.

Fourme, R. (1972). *Acta Cryst.* B**28**, 2984–2991.

Galešić, N., Vlahov, A. & Galešić, M. (1987). *Acta Cryst.* C**43**, 479–482.

Jeffrey, G. A., Ruble, J. R., McMullan, R. K., DeFrees, D. J., Binkley, J. S. & Pople, J. A. (1980). *Acta Cryst.* B**36**, 2292–2299.

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

Leiserowitz, L. & Rabinovich, D. (1969). *J. Chem. Soc. A*, pp. 2367–2371.

Main, P., Fiske, S. J., Hull, S. E., Lessinger, L., Germain, G., Declercq, J.-P. & Woolfson, M. M. (1980). *MULTAN*80. *A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data.* Univs. of York, England, and Louvain, Belgium.

Maksić, Z. B. (1986). *Comput. Maths. Appl.* **12B**, 697–723.

Mata, F., Martin, M. C. & Sørensen, G. O. (1978). *J. Mol. Struct.* **48**, 157–163.

Shephard, R. & Harrp, K. R. (1982). *Br. J. Cancer*, **45**, 413–420.

Skinner, M. D. & Schartz, R. S. (1972). *New Engl. J. Med.* **287**, 221–227.

Stewart, J. M., Machin, P. A., Dickinson, C. W., Ammon, H. L., Heck, H. & Flack, H. (1976). The *XRAY*76 system. Tech. Rep. TR-446. Computer Science Center, Univ. of Maryland, College Park, Maryland, USA.

Stewart, R. F., Davidson, E. R. & Simpson, W. T. (1965). *J. Chem. Phys.* **42**, 3175–3187.

Vicković, I. & Sikirica, M. (1975). *ZOAK5. A Program for Processing X-ray Diffractometer Intensity Data.* Laboratory of General and Inorganic Chemistry, Faculty of Science, Uiv. of Zagreb, Yugoslavia.

Vlahov, A. (1976). *Synthesis of Some N-Hydroxyalkyl- and N-Chloroalkylamides Derived from Thiobenzoic and 2-Furanthiocarboxylic Acids.* MSc thesis, Univ. of Zagreb, Yugoslavia.

Vlahov, A. (1982). *The Effect of Newly Synthetized Derivatives of 2-Furancarboxylic Acid on Functional Capacity of Lymphohematopoietic Cells.* PhD thesis, Univ. of Zagreb, Yugoslavia.

Vlahov, A. & Volf, D. (1983). *Topics in Furan Chemistry. Proc. Fourth Symp. Furan Chem. Bratislava*, edited by J. Kovač, pp. 237–239. Slovak Tech. Univ., Czechoslovakia.

Acta Cryst. (1987). C**43**, 2353–2355

Structure of 1,6-Cyclodecanedione Bis[(*o*-nitrophenyl)hydrazone]

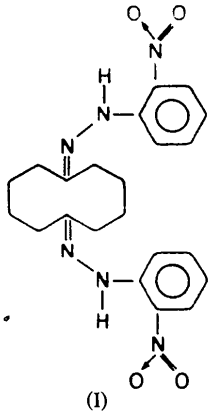
By C. A. Kavounis and P. J. Rentzeperis

Applied Physics Laboratory, Department of Physics, Aristotle University of Thessaloniki, Thessaloniki, Greece

(*Received* 4 *November* 1985; *accepted* 13 *July* 1987)

Abstract. $C_{22}H_{26}N_6O_4$, $M_r = 438·49$, monoclinic, $P2_1/c$, $a = 7·889$ (2), $b = 14·335$ (4), $c = 10·117$ (2) Å, $\beta = 110·83$ (2)°, $V = 1069·3$ Å^3, $Z = 2$, $D_m = 1·356$, $D_x = 1·362$ Mg m^{-3}, m.p. = 451–455 K, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 0·1039$ mm^{-1}, $F(000) = 464$, $T = 298$ K, final $R = 0·080$ for 1107 independent reflections. The molecule has crystallographic $\bar{1}$ (C_i) symmetry and is made up of two planar (*o*-nitrophenyl)hydrazone ligands attached at the 1,6-positions of the ten-membered ring in *trans* form. The central ten-membered ring has approximate $2/m$ symmetry and boat–chair–boat conformation, which is energetically the most stable. As in other compounds with a ten-membered ring, the endocyclic bond angles [114·7 (10)–119·1 (8)°] are considerably larger than the usually accepted value of 112·7° in normal paraffins, indicating strain in the ring. The bond distances and angles are in good agreement with literature values.

Introduction. Molecules containing ten-membered rings are of great interest, because of physical and chemical properties due to the dynamics of medium rings. The behaviour of such a molecule is explained by considering the large strain contained in the stereochemical form of the ring. The proximity effect of the ring causes transannular reactions between the two opposite 1,6 atoms and the formation of arylazodecalins such as 9,10-bis(*p*-chlorobenzeneazo)-*cis*-decalin (PCPAD) (Kavounis & Rentzeperis, 1983) and 9,10-bis(*p*-nitrobenzeneazo)-*cis*-decalin (PNPAD) (Kavounis & Rentzeperis, 1984). The title compound (I, ONPCD) was prepared at the Organic Chemistry Laboratory of the University of Thessaloniki (Malamidou-Xenikaki & Alexandrou, 1982), and its structure was determined within the framework of a systematic investigation of analogous compounds.



(I)

Table 1. *Atomic coordinates and equivalent isotropic temperature factors* (Å^2) *for the non*-H *atoms in ONPCD*

	x	*y*	*z*	B_{eq}*
O(1)	0·2982 (11)	−0·1309 (6)	0·5680 (9)	7·5
O(2)	0·2652 (13)	−0·2190 (6)	0·7305 (9)	11·1
N(1)	−0·0733 (11)	−0·0150 (6)	0·2392 (9)	4·4
N(2)	0·0248 (11)	−0·0675 (7)	0·3580 (10)	5·6
N(3)	0·2056 (15)	−0·1720 (7)	0·6222 (12)	6·8
C(1)	0·0263 (14)	0·0266 (7)	0·1822 (10)	4·0
C(2)	0·2297 (14)	0·0212 (9)	0·2248 (11)	7·1
C(3)	0·3131 (12)	0·0208 (8)	0·1102 (11)	5·8
C(4)	0·2766 (14)	−0·0665 (7)	0·0208 (12)	4·5
C(5)	0·0764 (15)	−0·0894 (6)	−0·0606 (10)	4·3
C(6)	−0·0726 (15)	−0·1192 (6)	0·4179 (10)	3·4
C(7)	0·0133 (16)	−0·1723 (8)	0·5422 (12)	4·3
C(8)	−0·0842 (20)	−0·2257 (9)	0·6042 (12)	6·4
C(9)	−0·2714 (21)	−0·2293 (8)	0·5440 (13)	7·1
C(10)	−0·3634 (14)	−0·1774 (7)	0·4206 (14)	6·0
C(11)	−0·2605 (15)	−0·1236 (6)	0·3641 (11)	4·3

* $B_{eq} = \frac{8}{3}\pi^2\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j$.

Table 2. *Interatomic distances* (Å) *and angles* (°) *in ONPCD*

(*a*) Interatomic distances

C(1)–C(2)	1·51 (1)	C(7)–C(8)	1·38 (2)
C(2)–C(3)	1·52 (2)	C(8)–C(9)	1·38 (2)
C(3)–C(4)	1·51 (1)	C(9)–C(10)	1·41 (2)
C(4)–C(5)	1·53 (1)	C(10)–C(11)	1·38 (2)
C(5)–C(1)′	1·51 (1)	C(11)–C(6)	1·39 (2)
C(1)–N(1)	1·28 (2)	C(7)–N(3)	1·44 (2)
N(1)–N(2)	1·40 (1)	N(3)–O(1)	1·21 (2)
N(2)–C(6)	1·36 (2)	N(3)–O(2)	1·23 (1)
C(6)–C(7)	1·42 (1)		

(*b*) Bond angles

C(1)–C(2)–C(3)	119·1 (8)	C(6)–C(7)–C(8)	122·1 (10)
C(2)–C(3)–C(4)	114·7 (10)	C(7)–C(8)–C(9)	119·9 (11)
C(3)–C(4)–C(5)	116·1 (9)	C(8)–C(9)–C(10)	120·2 (13)
C(4)–C(5)–C(1)′	117·5 (9)	C(9)–C(10)–C(11)	117·8 (10)
C(5)′–C(1)–C(2)	118·2 (10)	C(10)–C(11)–C(6)	124·4 (9)
C(5)′–C(1)–N(1)	114·2 (9)	C(11)–C(6)–C(7)	115·5 (10)
N(1)–C(1)–C(2)	127·6 (9)	C(6)–C(7)–N(3)	124·9 (12)
C(1)–N(1)–N(2)	113·5 (8)	C(8)–C(7)–N(3)	112·9 (10)
N(1)–N(2)–C(6)	116·7 (8)	C(7)–N(3)–O(1)	115·4 (10)
N(2)–C(6)–C(11)	123·1 (9)	C(7)–N(3)–O(2)	119·5 (12)
N(2)–C(6)–C(7)	121·4 (10)	O(1)–N(3)–O(2)	124·7 (11)

(*c*) Torsion angles

C(1)–C(2)–C(3)–C(4)	−68·7 (13)	C(4)–C(5)–C(1)′–C(2)′	−155·5 (13)
C(2)–C(3)–C(4)–C(5)	59·5 (11)	C(5)–C(1)′–C(2)′–C(3)′	39·2 (13)
C(3)–C(4)–C(5)–C(1)′	69·7 (13)		

Experimental. Small orange crystals, size 0·15 × 0·10 × 0·08 mm. D_m measured by flotation in KBr solution, computer-controlled Philips PW 1100 four-circle single-crystal diffractometer; graphite-monochromated Mo *K*α radiation, $\theta/2\theta$ scan mode; lattice parameters and standard deviations by least-squares analysis with *LATCON* (Stewart, Machin, Dickinson, Ammon, Heck & Flack, 1976) of the θ angles of 48 strong reflections ($\theta = 5$–$12°$). Systematic absences indicated the space groups *P*2/*c* and *Pc*, but $P2_1/c$ could not be excluded since the 0*k*0 reflections were not discernible, except for a very faint 040 reflection. Finally, $P2_1/c$ gave a plausible structure. 1935 reflections, of which 1720 independent, were measured in the range $\theta = 3$–$25°$, index range $h = -9 \rightarrow 9$, $k = 0 \rightarrow 16$, $l = 0 \rightarrow 9$; because of the relatively small number of reflections with $I > 2\sigma(I)$ in relation to the number of parameters, the data set was extended by considering all the reflections with $I > 1{\cdot}7\sigma(I)$ and from the unobserved, those with $F_c > F_o$; finally 1107 reflections were used; no decrease in intensity in the three standard reflections measured every 100 min; correction for Lorentz and polarization factors with *DATRED* (Main, 1970). $R_{int} = 0{\cdot}068$, no absorption correction. Structure initially solved in space group *Pc* ($R = 0{\cdot}107$) using *MULTAN*77 (Main, Lessinger, Woolfson, Germain & Declercq, 1977), but better convergence was attained in space group $P2_1/c$ after moving the centre of the centrosymmetric molecule to the symmetry centre (0,0,0). Refinement based on *F* magnitudes, full-matrix with *CRYLSQ* of the *XRAY*72 system (Stewart, Kruger, Ammon, Dickinson & Hall, 1972). 145 parameters refined. All the non-H atoms were refined anisotropically and the H atoms were kept fixed at their positions found with a difference Fourier synthesis; weighting scheme $w = 1/\sigma^2$; $R = 0{\cdot}080$, $wR = 0{\cdot}036$, $S = 1{\cdot}19$, $(\Delta/\sigma)_{max} = 0{\cdot}080$, residual density from −0·87 to 0·58 e Å^{-3}; the rather high value of the final *R* results from the high percentage of very weak reflections used; atomic scattering factors from

International Tables for X-ray Crystallography (Ibers & Hamilton, 1974). The best-plane parameters (deposited) were calculated with *BP*70 (Ito & Sugawara, 1983).

Discussion. Final coordinates for the non-H atoms are given in Table 1.* Interatomic distances and angles are in Table 2. A clinographic projection of the molecule with atomic numbering is shown in Fig. 1. The geometry of the C atoms about the two =N—N— groups of the ligands is *anti*. The ten-membered central ring shows approximate $2/m$ symmetry with the twofold axis passing through the middle of the C(5)—C(1′) and C(1)—C(5′) bonds. The C—C distances vary between 1·51 (1) and 1·53 (1) Å with a mean value of 1·52 Å. As with various other compounds containing a ten-membered ring, the C—C—C angles are considerably larger than the value of 112·7° in normal paraffins. In particular, the mean value of the ten C—C—C angles is 117·1° with a minimum of 114·7 (10)° and a maximum of 119·1 (8)° in excellent agreement with the values given for other compounds by Dunitz (1968) and Ermer, Dunitz & Bernal (1973). This conformation of the ring gives a C(1)···C(5) separation of 3·11 (2) Å, in good agreement with the value 3·13 Å for 1,6-cyclodecanedione (Dunitz, 1968). The torsion angles of the ten-membered ring (Table 2) agree well with the corresponding values in 1,6-cyclodecanedione and other cyclodecane derivatives (Dunitz, 1968).

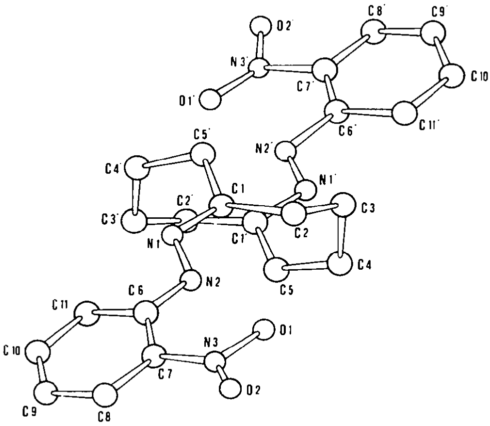


Fig. 1. Clinographic projection of the ONPCD molecule.

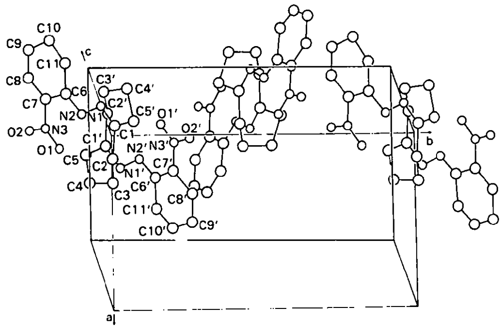


Fig. 2. Clinographic projection of the unit cell showing the molecular packing in ONPCD.

The bond distances in the benzene ring are normal, with a mean value of 1·39 Å.

A clinographic projection of the unit cell showing the molecular packing of ONPCD is given in Fig. 2. A very probable intramolecular hydrogen bond of the form N—H···O exists between the atoms N(2)—H(N2)···O(1) [H···O(1) = 1·95 Å, angle N(2)—H···O(1) = 123°] and also between their symmetry-equivalent atoms. A probable intermolecular hydrogen bond occurs between O(2) and atoms H(C10) and C(10) of the molecule at $(1+x,\ -\frac{1}{2}-y,\ \frac{1}{2}+z)$ with O(2)···H = 2·39 Å and O(2)···H—C(10) = 140°.

Thanks are due to Professor N. E. Alexandrou and to Dr E. Malamidou-Xenikaki for providing the ONPCD crystals. We also thank the University of Thessaloniki for the use of its computing facilities.

References

Dunitz, J. D. (1968). *Perspectives in Structural Chemistry*, Vol. 2, edited by J. D. Dunitz & J. A. Ibers, pp. 21*ff.* New York: John Wiley.

Ermer, O., Dunitz, J. D. & Bernal, I. (1973). *Acta Cryst.* **B29**, 2278–2285.

Ibers, J. A. & Hamilton, W. C. (1974). *International Tables for X-ray Crystallography*, Vol. IV, pp. 71–151. Birmingham: Kynoch Press. (Present distributor D. Reidel, Dordrecht.)

Ito, T. G. & Sugawara, Y. (1983). *Best-Plane Program BP*70. Institute of Physical and Chemical Research, RIKEN, Wako, Japan.

Kavounis, C. A. & Rentzeperis, P. J. (1983). *Z. Kristallogr.* **164**, 297–305.

Kavounis, C. A. & Rentzeperis, P. J. (1984). *Acta Cryst.* **C40**, 1434–1436.

Main, P. (1970). *A Computer Program for the Reduction of Raw Diffractometer Data to F_{obs}*. Univ. of York, England.

Main, P., Lessinger, L., Woolfson, M. M., Germain, G. & Declercq, J.-P. (1977). *MULTAN77. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data.* Univs. of York, England, and Louvain-La-Neuve, Belgium.

Malamidou-Xenikaki, E. & Alexandrou, N. E. (1982). *Tetrahedron Lett.* **23**, 3957–3958.

Stewart, J. M., Kruger, G. J., Ammon, H. L., Dickinson, C. W. & Hall, S. R. (1972). The *XRAY*72 system–version of June 1972. Tech. Rep. TR-192. Computer Science Center, Univ. of Maryland, College Park, Maryland, USA.

Stewart, J. M., Machin, P. A., Dickinson, C. W., Ammon, H. L., Heck, H. & Flack, H. (1976). The *XRAY*76 system. Tech. Rep. TR-446. Computer Science Center, Univ. of Maryland, College Park, Maryland, USA.

---

* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and least-squares-plane calculations have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44216 (12 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.