Fig. 2. Projection along b of the structure of (V).

differences found in the dimensions of the NCS groups and in the Cu—N—C angles can be attributed, respectively, to steric demand and steric strain of the organic and inorganic ligands in the linkage of the complexes. In the earlier-studied heterometal dmtp compounds the non-bridgehead pyrimidine N atom from dmtp was involved in intramolecular hydrogen bonds with coordinated water molecules which determined, apart from the packing, the orientation of the organic ligands. Although in the present compound the distance C(81)···N(32) of

$3 \cdot 37$ (2) Å is rather long, the distance H(81)···N(32) = $2 \cdot 45$ Å and the angle C(81)—H(81)—N(32) = 142°, with H in a calculated position, suggest a weak interaction between the non-bridgehead pyrimidine N atoms from dmtp bonded to Cu^I and the C—H group from dmtp linked to Hg^{II}.

References

BIAGINI CINGI, M., MANOTTI LANFREDI, A. M., TIRIPICCHIO, A., HAASNOOT, J. G. & REEDIJK, J. (1983). Inorg. Chim. Acta, 72, 81–88.

BIAGINI CINGI, M., MANOTTI LANFREDI, A. M., TIRIPICCHIO, A., HAASNOOT, J. G. & REEDIJK, J. (1984). Inorg. Chim. Acta, 86, 137–143.

BIAGINI CINGI, M., MANOTTI LANFREDI, A. M., TIRIPICCHIO, A., HAASNOOT, J. G. & REEDIJK, J. (1985). Inorg. Chim. Acta, 101, 49–61.

BÜLOW, C. & HAAS, K. (1909). Ber. Dtsch. Chem. Ges. 42, 4638–4644.

International Tables for X-ray Crystallography (1974). Vol. IV, pp. 99–101, 149–150. Birmingham: Kynoch Press. (Present distributor D. Reidel, Dordrecht.)

SHELDRICK, G. M. (1976). SHELX76. Program for crystal structure determination. Univ. of Cambridge, England.

SINGH, P. P. (1980). Coord. Chem. Rev. 32, 33–65.

Structure of 2-Oxatricyclo[6.4.0.0³,⁶]dodeca-1(12),8,10-trien-7-ol

By Dilip Kumar Nag* and Siddhartha Ray

X-ray Crystallography Laboratory, Department of Magnetism, Indian Association for the Cultivation of Science, Jadavpur, Calcutta-700 032, India

and Dipanjan Sengupta and R. V. Venkateswaran

Department of Organic Chemistry, Indian Association for the Cultivation of Science, Jadavpur, Calcutta-700 032, India

Abstract. C$_{11}$H$_{12}$O$_2$, $M_r = 176 \cdot 2$, monoclinic, $C2/c$, $a = 23 \cdot 575$ (6), $b = 7 \cdot 591$ (1), $c = 10 \cdot 056$ (4) Å, $\beta = 101 \cdot 92$ (3)°, $V = 1760 \cdot 8$ Å^3, $Z = 8$, $D_x = 1 \cdot 33$, $D_m = 1 \cdot 34$ g cm^{-3}, λ(Mo $K\alpha$) = $0 \cdot 7107$ Å, $\mu = 0 \cdot 98$ cm^{-1}, $F(000) = 752$, $T = 293$ K. Final $R = 0 \cdot 051$ for 930 observed reflections. The study establishes the stereo-chemistry at the ring junction as cis and the hydroxy group in the endo configuration. Bond lengths and angles are normal. The four-membered ring is planar to within $\pm 0 \cdot 01$ Å.

Introduction. In connection with a comprehensive programme devoted to the synthesis of A-ring aromatic tricothecane analogues (Anderson & Lee, 1980) and related sesquiterpenes, compounds of type (1) were

---

* Present address: Mineral Physics Division, Geological Survey of India, 29 Jawaharlal Nehru Road, Calcutta-700 016, India.

synthesized to study their (or their derivatives') reaction course under cationic rearrangement conditions. These were prepared by addition of ethylene to chromones (2) under UV irradiation. Addition of substituted ethylenes to chromones has precedent (Hanifin & Cohen, 1969) and the stereochemistry at the ring junction has been assigned as *cis*, based on chemical and spectral evidence. In the present case, compounds of type (1) were prepared by irradiation of a benzene solution of chromones (2) with continuous bubbling of ethylene. To establish unequivocally the stereochemistry at the ring junction, an X-ray crystallographic analysis of a crystalline alcohol (3) obtained from lithium aluminium hydride reduction of (1,R=H) was undertaken.

Table 1. *Final atomic coordinates* ($\times 10^4$) *with e.s.d.'s in parentheses and equivalent isotropic thermal parameters for the non-*H *atoms*

$$B_{eq} = \tfrac{4}{3}\Sigma_i\Sigma_j\beta_{ij}\mathbf{a}_i\cdot\mathbf{a}_j.$$

	x	y	z	B_{eq}(Å^2)
O(1)	205 (1)	4264 (3)	1313 (2)	3·96
O(2)	1975 (1)	3198 (3)	1642 (2)	4·03
C(1)	1569 (1)	2374 (4)	646 (3)	2·74
C(2)	1768 (1)	1274 (4)	−253 (3)	3·28
C(3)	1377 (1)	379 (5)	−1226 (3)	3·55
C(4)	789 (2)	602 (4)	−1307 (4)	3·72
C(5)	589 (1)	1708 (5)	−398 (3)	3·34
C(6)	979 (1)	2607 (4)	607 (3)	2·52
C(7)	805 (1)	3756 (4)	1677 (3)	2·82
C(8)	1194 (1)	5366 (4)	1971 (3)	3·00
C(9)	1179 (1)	6686 (5)	788 (3)	3·59
C(10)	1822 (2)	6337 (5)	820 (4)	4·51
C(11)	1844 (1)	4987 (5)	1962 (3)	3·36

(1) R=H, Me; R₁=R₂=O   (2) R=H, Me

(3) R=R₁=H; R₂=OH

Experimental. Colourless needle-shaped specimen, $0.45 \times 0.15 \times 0.20$ mm. CAD-4F diffractometer, graphite-monochromated Mo $K\alpha$ radiation. 25 reflections ($9 < \theta < 17.5°$) used for measurement of lattice parameters. Data collected by ω–2θ scan, $\theta_{max} = 25°$, h −24 to 24, k 0 to 9, l 0 to 12. 1513 unique reflections measured; 930 with $I > 2\sigma(I)$ considered observed. Variation in intensity of three periodically monitored reflections ($\overline{12},0,2$, $\overline{2}21$ and 022) <1%. No decay correction, no absorption correction. Density measured by flotation in aqueous zinc bromide. Structure solved by $MULTAN78$ (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978). Least-squares refinement (on F) with weights $w = \sigma^{-2}(F)$, $\sigma(F)$ based on intensity statistics alone. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974). Block-diagonal approximation after introduction of anisotropic thermal parameters. 11 H atoms, assigned isotropic temperature factors equivalent to those of the respective contiguous C atoms, kept fixed at their calculated positions. Refinement using 930 observed reflections converged (shift < corresponding e.s.d.) at R=0·051, wR=0·045, S=1·43. The H atom belonging to the OH group could not be located. Final difference map showed max. and min. peaks 0·3 and −0·3 e Å^{-3} respectively. All calculations carried out on a Burroughs 6700 computer. Programs used from $XRAY ARC$ (*World List of Crystallographic Computer Programs*, 1973), locally modified for the B6700 computer.

Table 2. *Bond distances* (Å) *and angles* (°) *with e.s.d.'s in parentheses*

C(1)–C(2)	1·382 (5)	C(7)–C(8)	1·519 (4)
C(2)–C(3)	1·377 (5)	C(8)–C(11)	1·561 (4)
C(3)–C(4)	1·383 (5)	C(11)–O(2)	1·443 (4)
C(4)–C(5)	1·392 (5)	O(2)–C(1)	1·385 (4)
C(5)–C(6)	1·396 (4)	C(8)–C(9)	1·550 (5)
C(6)–C(1)	1·393 (4)	C(9)–C(10)	1·534 (5)
C(6)–C(7)	1·506 (4)	C(10)–C(11)	1·532 (5)
C(7)–O(1)	1·439 (4)		

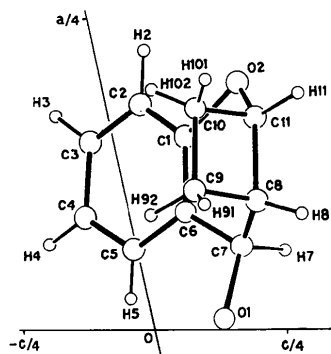| C(1)–C(2)–C(3)	119·7 (3)	C(6)–C(1)–O(2)	120·0 (3)
C(2)–C(3)–C(4)	119·9 (3)	C(8)–C(9)–C(10)	90·4 (3)
C(3)–C(4)–C(5)	120·4 (3)	C(9)–C(10)–C(11)	90·6 (3)
C(4)–C(5)–C(6)	120·6 (3)	C(10)–C(11)–C(8)	90·0 (3)
C(5)–C(6)–C(1)	117·5 (3)	C(11)–C(8)–C(9)	88·9 (2)
C(6)–C(1)–C(2)	122·0 (3)	C(2)–C(1)–O(2)	117·9 (3)
C(1)–O(2)–C(11)	116·0 (2)	C(5)–C(6)–C(7)	124·4 (3)
O(2)–C(11)–C(8)	115·5 (3)	C(10)–C(11)–O(2)	115·9 (3)
C(11)–C(8)–C(7)	113·8 (3)	C(6)–C(7)–O(1)	111·8 (2)
C(8)–C(7)–C(6)	111·3 (3)	O(1)–C(7)–C(8)	110·8 (2)
C(7)–C(6)–C(1)	118·1 (3)	C(7)–C(8)–C(9)	117·0 (3)

Fig. 1. View of the molecule (3) along **b**.

Discussion. The refined positional parameters are in Table 1* and bond distances and angles in Table 2. A view of the molecule along **b** is shown in Fig. 1.

The aromatic ring is, as usual, planar. The ring C(1)–C(6)–C(7)–C(8)–C(11)–O(2) has a boat conformation. In the four-membered ring C(8)–C(9)–C(10)–C(11), only one of the angles deviates from 90° by more than 2σ. The ring junction H atoms, H(8) and H(11), are *cis*. The stereochemistry is thus similar to that previously assigned to the photocycloaddition

products of substituted ethylenes to chromones (Hanifin & Cohen, 1969).

---

* Lists of structure factors, anisotropic thermal parameters, H-atom parameters, bond lengths involving H and mean-planes' calculations have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42619 (11 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

ANDERSON, W. K. & LEE, G. E. (1980). *J. Org. Chem.* **45**, 501–506.
HANIFIN, J. W. & COHEN, E. (1969). *J. Am. Chem. Soc.* **91**, 4494–4499.
International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor D. Reidel, Dordrecht.)
MAIN, P., HULL, S. E., LESSINGER, L., GERMAIN, G., DECLERCQ, J.-P. & WOOLFSON, M. M. (1978). *MULTAN78. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data.* Univs. of York, England, and Louvain, Belgium.
World List of Crystallographic Computer Programs (1973). *J. Appl. Cryst.* **6**, 309–346.

4'-Nitro-2-biphenylamine

BY H. H. SUTHERLAND AND Z. ALI-ADIB

Physics Department, The University of Hull, Hull HU6 7RX, England

Abstract. $C_{12}H_{10}N_2O_2$, $M_r = 214 \cdot 2$, monoclinic, $C2/c$, $a = 20 \cdot 66$ (5), $b = 7 \cdot 86$ (3), $c = 13 \cdot 29$ (1) Å, $\beta = 104 \cdot 6$ (2)°, $V = 2088$ Å^3, $Z = 8$, $D_m = 1 \cdot 37$, $D_x = 1 \cdot 36$ Mg m^{-3}, Cu $K\alpha$, $\lambda = 1 \cdot 5418$ Å, $\mu = 0 \cdot 69$ mm^{-1}, $F(000) = 896$, $T = 290$ K, $R = 0 \cdot 102$ for 1175 observed reflexions. The average C–C bond in the phenyl rings is $1 \cdot 391$ Å. The molecule is non-planar; the angle between the phenyl rings is $54 \cdot 7$ (6)°, the angle between the nitro group and the ring to which it is bonded is $7 \cdot 1$ (6)° and the bond N–C(4') is $1 \cdot 453$ (9) Å.

Introduction. The structure determination of 4'-nitro-2-biphenylamine forms part of an investigation into liquid-crystal compounds and their chemical precursors.

Experimental. D_m measured by flotation in aqueous cadmium *n*-dodecatungstoborate; orange opaque crystals, $0 \cdot 6 \times 0 \cdot 02 \times 0 \cdot 05$ mm and $0 \cdot 45 \times 0 \cdot 016 \times 0 \cdot 004$ mm, respectively, for *b*-axis, $k = 0$–6 and *c*-axis *hk*0 Weissenberg data. 1221 reflexions measured from multiple-film photographs, Cu $K\alpha$ radiation: $-26 \le h \le 24$; $0 \le k \le 6$; $0 \le l \le 16$. Data merged to give 1175 unique observed reflexions; $R_{int} = 0 \cdot 06$; structure solved by direct methods with *SHELX*76 (Sheldrick, 1976) and refined by least squares (*F* values) with anisotropic thermal parameters for non-hydrogen atoms. H-atom positions, initially obtained from dif-

ference synthesis and placed at geometrically reasonable positions, refined with isotropic thermal parameters; $R = 0 \cdot 102$; unit weights. $(\Delta/\sigma)_{max}$ in final refinement cycle $0 \cdot 03$ for positional and $0 \cdot 04$ for thermal parameters. Max. and min. heights in final $\Delta \rho$ map $+0 \cdot 30$ and $-0 \cdot 40$ e Å^{-3}. Scattering factors from *International Tables for X-ray Crystallography* (1974). Computer programs used: *SHELX*76 (Sheldrick, 1976) and locally written programs supplied by HHS and Drs C. Morgan and M. J. Mottram.

Discussion. Table 1* gives atomic parameters and Table 2 bond lengths and angles. The atomic numbering is shown in Fig. 1.

The phenyl ring with the attached amino group is planar to within $\pm 0 \cdot 004$ Å; in the other phenyl ring deviations of up to $\pm 0 \cdot 01$ Å from the mean plane occur. The average C–C bond in the two rings is $1 \cdot 391$ Å. The C(1)–C(7) bond length, $1 \cdot 465$ (9) Å, although slightly shorter than in some biphenyl com-

---

* Lists of structure factors, anisotropic thermal parameters, H atom parameters, intermolecular contact distances and mean-plane calculations have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42634 (20 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.