SHORT-FORMAT PAPERS

Contributions intended for publication under this heading should follow the format given in the Checklist for Authors [*Acta Cryst.* (1985). C**41**, 1–4].

Acta Cryst. (1985). C**41**, 1548–1549

Structure of a 2,2′-Bipyridine Containing Dioxolane and 6-Bromopyridine Subunits

BY FRANK R. FRONCZEK, HELLEN C. R. TAYLOR, VINOD K. GUPTA AND GEORGE R. NEWKOME*

Department of Chemistry, Louisiana State University, Baton Rouge, Louisiana 70803-1804, *USA*

(*Received* 28 *May* 1985; *accepted* 19 *June* 1985)

Abstract. 6,6′-Bis[2-(6-bromo-2-pyridyl)-1,3-dioxolan-2-yl]-2,2′-bipyridine, $C_{26}H_{20}Br_2N_4O_4$, M_r = 612·3, triclinic, $P\bar{1}$, a = 6·231 (1), b = 9·859 (2), c = 10·299 (3) Å, α = 88·45 (2), β = 84·65 (2), γ = 77·09 (2)°, V = 613·9 (3) Å^3, Z = 1, D_x = 1·656 g cm^{-3}, Mo $K\alpha$, λ = 0·71073 Å, μ = 33·1 cm^{-1}, $F(000)$ = 306, T = 302 K, R = 0·037 for 1795 observations (of 2158 unique data). The molecule has crystallographic $\bar{1}$ symmetry, with the bipyridine moiety in the *anti* conformation, the dioxolane rings in the half-chair conformation, and pyridine nitrogen atoms oriented *anti* to dioxolane oxygen atoms.

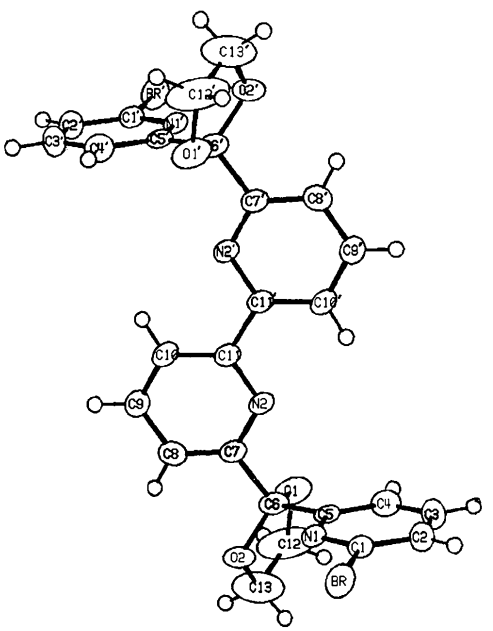

Fig. 1. Numbering scheme and conformation.

* To whom correspondence should be addressed.

0108-2701/85/101548-02$01.50

Experimental. Material synthesized by Taylor (1983), colorless crystals from CH_3CN. Crystal size 0·28 × 0·32 · 0·48 mm, space group by successful refinement of centrosymmetric model; cell dimensions from setting angles of 25 reflections having $14 < \theta < 16°$. Data collection on Enraf–Nonius CAD-4 diffractometer, Mo $K\alpha$ radiation, graphite monochromator, ω–2θ scans designed for $I = 25\sigma(I)$. Scan rates varied 0·49–5·0° min^{-1}. Reflections having $1 < \theta < 25°$, $0 \le h \le 7$, $-11 \le k \le 11$, $-12 \le l \le 12$ measured, corrected for background, Lorentz and polarization effects. No redundant data measured. Absorption corrections by ψ scans of reflections near $\chi = 90°$, minimum relative transmission coefficient 69·4%. Standard reflections 300, 030, 006, ±2·1% random variation. Structure solved by heavy-atom methods, refined by full-matrix least squares based on F, using data for which $I > 3\sigma(I)$, weights $w = \sigma^{-2}(F_o)$, with Enraf–Nonius

Table 1. *Coordinates and equivalent isotropic thermal parameters*

	x	y	z	B_{eq}*(Å^2)
Br	0·15335 (6)	0·32311 (3)	0·06789 (3)	3·800 (7)
O(1)	0·1375 (5)	0·8394 (2)	0·4299 (2)	4·31 (6)
O(2)	−0·1155 (4)	0·7050 (2)	0·4298 (2)	3·67 (5)
N(1)	0·1299 (4)	0·5556 (2)	0·2148 (2)	2·42 (5)
N(2)	0·0327 (4)	0·8917 (2)	0·1445 (2)	2·60 (5)
C(1)	0·2741 (5)	0·4529 (3)	0·1559 (3)	2·62 (6)
C(2)	0·5012 (5)	0·4301 (4)	0·1551 (4)	3·61 (8)
C(3)	0·5823 (5)	0·5237 (4)	0·2220 (4)	4·21 (8)
C(4)	0·4352 (6)	0·6323 (3)	0·2858 (4)	3·81 (7)
C(5)	0·2111 (5)	0·6462 (3)	0·2801 (3)	2·53 (6)
C(6)	0·0382 (5)	0·7623 (3)	0·3472 (3)	2·94 (6)
C(7)	−0·0875 (5)	0·8641 (3)	0·2521 (3)	2·66 (6)
C(8)	−0·3075 (6)	0·9261 (3)	0·2800 (3)	3·45 (7)
C(9)	−0·4124 (6)	1·0200 (4)	0·1897 (4)	3·64 (8)
C(10)	−0·2915 (6)	1·0506 (3)	0·0795 (3)	3·20 (7)
C(11)	−0·0692 (5)	0·9855 (3)	0·0591 (3)	2·55 (6)
C(12)	0·0974 (12)	0·7937 (6)	0·5585 (4)	8·8 (2)
C(13)	−0·0469 (11)	0·6965 (6)	0·5552 (4)	7·7 (2)

* $B_{eq} = \frac{4}{3}(a^2\beta_{11} + b^2\beta_{22} + c^2\beta_{33} + ab\beta_{12}\cos\gamma + ac\beta_{13}\cos\beta + bc\beta_{23}\cos\alpha)$.

© 1985 International Union of Crystallography

Table 2. *Bond distances* (Å), *angles* (°) *and torsion angles* (°)

Br–C(1)	1·909 (3)	C(3)–C(4)	1·380 (5)
O(1)–C(6)	1·425 (3)	C(4)–C(5)	1·379 (4)
O(1)–C(12)	1·405 (5)	C(5)–C(6)	1·517 (4)
O(2)–C(6)	1·422 (3)	C(6)–C(7)	1·522 (3)
O(2)–C(13)	1·394 (5)	C(7)–C(8)	1·378 (4)
N(1)–C(1)	1·314 (3)	C(8)–C(9)	1·394 (4)
N(1)–C(5)	1·343 (3)	C(9)–C(10)	1·368 (4)
N(2)–C(7)	1·336 (3)	C(10)–C(11)	1·390 (4)
N(2)–C(11)	1·352 (3)	C(11)–C(11′)	1·484 (4)
C(1)–C(2)	1·381 (4)	C(12)–C(13)	1·456 (7)
C(2)–C(3)	1·376 (5)		
C(6)–O(1)–C(12)	107·4 (3)	O(1)–C(6)–C(7)	107·9 (2)
C(6)–O(2)–C(13)	107·5 (3)	O(2)–C(6)–C(5)	109·8 (2)
C(1)–N(1)–C(5)	116·9 (2)	O(2)–C(6)–C(7)	108·9 (2)
C(7)–N(2)–C(11)	117·6 (2)	C(5)–C(6)–C(7)	113·3 (2)
Br–C(1)–N(1)	115·9 (2)	N(2)–C(7)–C(6)	115·3 (2)
Br–C(1)–C(2)	118·3 (2)	N(2)–C(7)–C(8)	123·4 (3)
N(1)–C(1)–C(2)	125·8 (3)	C(6)–C(7)–C(8)	121·3 (3)
C(1)–C(2)–C(3)	116·7 (3)	C(7)–C(8)–C(9)	118·5 (3)
C(2)–C(3)–C(4)	118·9 (3)	C(8)–C(9)–C(10)	118·9 (3)
C(3)–C(4)–C(5)	119·8 (3)	C(9)–C(10)–C(11)	119·3 (3)
N(1)–C(5)–C(4)	121·8 (3)	N(2)–C(11)–C(10)	122·2 (3)
N(1)–C(5)–C(6)	114·9 (2)	N(2)–C(11)–C(11′)	116·3 (3)
C(4)–C(5)–C(6)	123·3 (3)	C(10)–C(11)–C(11′)	121·5 (3)
O(1)–C(6)–O(2)	106·1 (2)	O(1)–C(12)–C(13)	107·4 (4)
O(1)–C(6)–C(5)	110·6 (2)	O(2)–C(13)–C(12)	106·1 (3)

N(1)–C(5)–C(6)–O(1)	170·9 (4)
N(2)–C(7)–C(6)–O(2)	−159·5 (4)
O(1)–C(12)–C(13)–O(2)	−9·3 (6)
C(12)–C(13)–O(2)–C(6)	20·5 (5)
C(13)–O(2)–C(6)–O(1)	−24·0 (5)
O(2)–C(6)–O(1)–C(12)	17·9 (4)
C(6)–O(1)–C(12)–C(13)	−5·5 (6)

SDP (Frenz & Okaya, 1980), scattering factors of Cromer & Waber (1974). Non-H atoms anisotropic; H atoms located by ΔF synthesis, placed in calculated positions with C–H 0·95 Å, $B = 5{\cdot}0$ Å^2. Final $R = 0{\cdot}037$ (0·053 for all data), $wR = 0{\cdot}049$, $S = 1{\cdot}563$ for 163 variables. Max. shift $0{\cdot}01\sigma$ in final cycle, largest residual density 0·62 e Å^{-3}, min. −0·45 e Å^{-3}. The molecule is depicted in Fig. 1, coordinates are given in Table 1, bond distances, angles and torsion angles are listed in Table 2.*

Related literature. Chisholm, Huffman, Rothwell, Bradley, Kress & Woodruff (1981); James & Williams (1973); Kvick (1976); Newkome, Taylor, Fronczek & Delord (1984).

This work was supported, in part, by the National Science Foundation and the Center for Energy Studies, LSU.

* Lists of H-atom coordinates, anisotropic thermal parameters and structure factor amplitudes have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42311 (13 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

CHISHOLM, M. H., HUFFMAN, J. C., ROTHWELL, I. P., BRADLEY, P. G., KRESS, N. & WOODRUFF, W. H. (1981). *J. Am. Chem. Soc.* **103**, 4945–4947.

CROMER, D. T. & WABER, J. T. (1974). *International Tables for X-ray Crystallography*, Vol. IV, Table 2.2B. Birmingham: Kynoch Press. (Present distributor D. Reidel, Dordrecht.)

FRENZ, B. A. & OKAYA, Y. (1980). *Enraf–Nonius Structure Determination Package.*

JAMES, M. N. G. & WILLIAMS, G. J. B. (1973). *Acta Cryst.* B**29**, 1172–1174.

KVICK, Å. (1976). *Acta Cryst.* B**32**, 220–224.

NEWKOME, G. R., TAYLOR, H. C. R., FRONCZEK, F. R. & DELORD, T. J. (1984). *J. Org. Chem.* **49**, 2961–2971.

TAYLOR, H. C. R. (1983). PhD dissertation, Louisiana State Univ., Baton Rouge, Louisiana, USA.

Acta Cryst. (1985). C**41**, 1549–1550

Structure of Tetraphenylphosphonium Bromide

BY NATHANIEL W. ALCOCK, MARK PENNINGTON AND GERALD R. WILLEY

Department of Chemistry, University of Warwick, Coventry CV4 7AL, England

(*Received* 25 *April* 1985; *accepted* 15 *July* 1985)

Abstract. $C_{24}H_{20}P^+.Br^-$, $M_r = 419{\cdot}3$, tetragonal, $I\bar{4}$, $a = 11{\cdot}960$ (2), $c = 6{\cdot}967$ (2) Å, $V = 996{\cdot}6$ (3) Å^3, $Z = 2$, $D_m = 1{\cdot}45$, $D_x = 1{\cdot}40$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 21{\cdot}24$ cm^{-1}, $F(000) = 428$, ambient temperature. $R = 0{\cdot}066$ for 1006 observed reflections. The cation has $\bar{4}$ symmetry with P–C bond lengths of 1·800 (6) Å. The dimensions of the phenyl rings agree with standard values.

Experimental. Title compound recrystallized from MeOH/MeCN to yield large colourless block-like crystals. Density measured by flotation. Data collected with a Syntex $P2_1$ four-circle diffractometer. Maximum 2θ 60°, scan range ±1° (2θ) around $K\alpha_1$–$K\alpha_2$ angles; scan speed 2–29° min^{-1} depending on intensity of 2 s prescan; backgrounds measured at each end of the scan for 0·25 of scan time. Three standard reflections

0108-2701/85/101549-02$01.50

© 1985 International Union of Crystallography