refined parameter 1702/166 = 10·3. A *PLUTO* (Motherwell & Clegg, 1978) view of the title compound with the atomic numbering scheme is shown in Fig. 1. The final atomic coordinates and equivalent isotropic thermal parameters are given in Table 1.* Bond lengths, bond angles and selected torsion angles are given in Table 2. A *PLUTO* (Motherwell & Clegg, 1978) plot of the crystal packing is shown in Fig. 2. All calculations were performed on a Digital PDP-11/73 microcomputer using *SDP* (B. A. Frenz & Associates, Inc., 1982) and *PARST* (Nardelli, 1983).

Related literature. All bond lengths and bond angles are within the normal range (Allen, Kennard, Watson, Brammer, Orpen & Taylor, 1987). The Cremer & Pople (1975) puckering parameters for the sequence C(1′)—C(2′)—C(3′)—C(4′)—C(5′)—O(5′) are $\varphi_2 = -19\,(4)^\circ$ and $\theta_2 = 8{\cdot}9\,(5)^\circ$ with $Q_t = 0{\cdot}596\,(5)$ Å, indicating a chair conformation. The crystal packing is stabilized by hydrogen bonds involving both rings (Table 3) and by base stacking forces (mean interplanar distance between adjacent bases = 3·5 Å; dihedral angle = 0·0°).

Table 3. *Geometry* (Å, °) *of intra- and intermolecular hydrogen bonds with e.s.d.'s in parentheses*

X—H···Y	d(H···Y)	d(X···Y)	X—H···Y
O(44′)—H(4)···O(4) (1)	2·2 (1)	2·973 (6)	138 (7)
O(33′)—H(6)···O(4) (2)	1·9 (1)	2·764 (6)	140 (10)
N(3)—H(9)···O(33′) (3)	2·013 (5)	2·862 (6)	147·7 (3)

Equivalent positions: (1) $x, y-1, z+1$; (2) $x-1, y, z+1$; (3) $x, y, z-1$.

The authors wish to thank J. P. Van Cuyck for help in preparing the figures and L. Kerremans for technical assistance in synthesizing the compound.

References

ALLEN, F. H., KENNARD, O., WATSON, D. G., BRAMMER, L., ORPEN, A. G. & TAYLOR, R. (1987). *J. Chem. Soc. Perkin Trans.* 2, pp. S1–S19.

B. A. FRENZ & ASSOCIATES, INC. (1982). *SDP Structure Determination Package.* College Station, Texas, USA.

CREMER, D. & POPLE, J. A. (1975). *J. Am. Chem. Soc.* **97**, 1354–1358.

IBERS, J. A. & HAMILTON, W. C. (1964). *Acta Cryst.* **17**, 781–782.

IUPAC–IUB JOINT COMMISSION ON BIOCHEMICAL NOMENCLATURE (1983). *Pure Appl. Chem.* **55**, 1273–1280.

MOTHERWELL, W. D. S. & CLEGG, W. (1978). *PLUTO.* Program for plotting molecular and crystal structures. Univ. of Cambridge, England.

NARDELLI, M. (1983). *Comput. Chem.* **7**, 95–98.

STEWART, R. F., DAVIDSON, E. R. & SIMPSON, W. T. (1965). *J. Chem. Phys.* **42**, 3175–3187.

Stoe & Co. (1985). *REDU4. Data Reduction Program.* Stoe & Co., Darmstadt, Germany.

VAN AERSCHOT, A., EVERAERT, D., GOSSELIN, G., PEETERS, O., BLATON, N., DE RANTER, C., IMBACH, J.-L., BALZARINI, J., DE CLERCQ, E. & HERDEWIJN, P. (1990). *Antiviral Res.* **14**, 357–369.

---

* Lists of structure factors, anisotropic thermal parameters, bond lengths and angles involving H atoms, least-squares planes and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54213 (15 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Acta Cryst. (1991). C**47**, 2247–2249

Structures of Two Modifications of 2,2′:4′,4″:2″,2‴-Quaterpyridine

BY GENE E. HONEY AND PETER J. STEEL*

Chemistry Department, University of Canterbury, Christchurch, New Zealand

(*Received* 18 *March* 1991; *accepted* 26 *April* 1991)

Abstract. $C_{20}H_{14}N_4$, $M_r = 310{\cdot}4$. α-Form: monoclinic, $P2_1/c$, $a = 9{\cdot}434\,(8)$, $b = 6{\cdot}110\,(4)$, $c = 13{\cdot}322\,(10)$ Å, $\beta = 105{\cdot}02\,(6)^\circ$, $V = 741{\cdot}7\,(9)$ Å^3, $Z = 2$, $D_x = 1{\cdot}39$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 0{\cdot}08$ mm^{-1}, $F(000) = 324$, $T = 160$ K, $R = 0{\cdot}052$ for 976 independent observed reflections. β-Form: monoclinic, $P2_1/n$, $a = 7{\cdot}224\,(5)$, $b = 11{\cdot}530\,(6)$, $c = 8{\cdot}978\,(6)$ Å, $\beta = 91{\cdot}21\,(5)^\circ$, $V = 747{\cdot}7\,(8)$ Å^3, $Z = 2$, $D_x = 1{\cdot}38$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 0{\cdot}08$ mm^{-1}, $F(000) = 324$, $T = 160$ K, $R = 0{\cdot}047$ for 617 independent observed reflections. In both forms the molecules exist in a centrosymmetric planar conformation with *transoid* 2,2′- (and 2″,2‴-) linkages and pack with a common herringbone pattern but with different molecular stackings.

Experimental. Recrystallization of the title compound (1) from ethanol gave a mixture of two macroscopically distinguishable modifications, crystals of which were separated manually. The α-form

---

* Author to whom correspondence should be addressed.

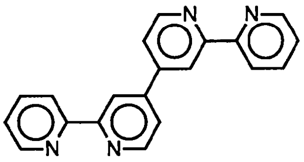
0108-2701/91/102247-03$03.00

Table 1. *Atomic coordinates* ($\times 10^4$) *and isotropic displacement parameters* (Å$^2 \times 10^3$)

U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U_{eq}
α-Form				
N(1)	2721 (2)	6172 (3)	8303 (1)	26 (1)
C(2)	2284 (3)	5881 (3)	9174 (2)	22 (1)
C(3)	1265 (3)	7245 (4)	9450 (2)	25 (1)
C(4)	713 (3)	9010 (4)	8817 (2)	29 (1)
C(5)	1172 (3)	9344 (4)	7929 (2)	29 (1)
C(6)	2173 (3)	7888 (4)	7704 (2)	28 (1)
N(1′)	2545 (2)	3743 (3)	10727 (1)	27 (1)
C(2′)	2931 (3)	3978 (3)	9836 (2)	21 (1)
C(3′)	3896 (3)	2563 (3)	9536 (2)	22 (1)
C(4′)	4491 (3)	782 (3)	10157 (2)	21 (1)
C(5′)	4081 (3)	552 (4)	11084 (2)	26 (1)
C(6′)	3129 (3)	2040 (4)	11328 (2)	28 (1)
β-Form				
N(1)	4834 (4)	7399 (3)	4967 (4)	26 (1)
C(2)	5174 (6)	8233 (4)	3951 (4)	22 (1)
C(3)	6758 (5)	8253 (4)	3114 (4)	26 (2)
C(4)	8046 (6)	7383 (4)	3334 (5)	31 (2)
C(5)	7749 (6)	6535 (4)	4382 (5)	31 (2)
C(6)	6123 (6)	6581 (4)	5162 (5)	32 (2)
N(1′)	3993 (4)	9946 (3)	2721 (3)	24 (1)
C(2′)	3716 (5)	9135 (4)	3768 (4)	22 (1)
C(3′)	2158 (5)	9129 (4)	4674 (4)	22 (1)
C(4′)	820 (5)	9987 (4)	4511 (4)	22 (1)
C(5′)	1107 (5)	10817 (4)	3414 (4)	25 (1)
C(6′)	2687 (5)	10759 (4)	2569 (5)	27 (2)

exists as plates and the more abundant β-form as rods.



(1)

The crystals used measured 0·58 × 0·46 × 0·06 mm for the α-form and 0·79 × 0·08 × 0·06 mm for the β-form. Lattice parameters were determined with a Nicolet *R*3*m* diffractometer, using 25 reflections with $20 < 2\theta < 32°$ in each case. Data collection employed ω scans to 55° in both cases, with $-12 \le h \le 12$, $0 \le k \le 7$, $0 \le l \le 17$ for the α-form and $-9 \le h \le 9$, $0 \le k \le 14$, $0 \le l \le 11$ for the β-form; standard reflections (and intensity variations) were 600, $\bar{1}06$, 032 (1·5%) for the α-form, and $\bar{3}01$, 041, 004 (4·0%) for the β-form and were monitored every 100 measurements. The reflections measured, unique reflections, $R_{\rm int}$ and observed reflections [$I > 3\sigma(I)$] are 1941, 1699, 0·024, 976 for the α-form, and 1904, 1705, 0·025, 617 for the β-form. Corrections were applied for Lorentz and polarization effects but not for absorption. Both structures were solved by direct methods and refined on $|F|$ by blocked-cascade least-squares procedures. All non-H atoms were

Table 2. *Bond lengths* (Å) *and angles* (°)

	α-Form	β-Form
N(1)—C(2)	1·340 (3)	1·352 (5)
N(1)—C(6)	1·338 (3)	1·334 (5)
C(2)—C(3)	1·392 (4)	1·383 (6)
C(2)—C(2′)	1·491 (3)	1·487 (6)
C(3)—C(4)	1·385 (3)	1·379 (6)
C(4)—C(5)	1·376 (4)	1·377 (6)
C(5)—C(6)	1·386 (4)	1·381 (6)
N(1′)—C(2′)	1·336 (3)	1·344 (5)
N(1′)—C(6′)	1·341 (3)	1·335 (5)
C(2′)—C(3′)	1·389 (3)	1·402 (5)
C(3′)—C(4′)	1·394 (3)	1·389 (6)
C(4′)—C(5′)	1·393 (4)	1·392 (6)
C(4′)—C(4′*a*)	1·490 (5)	1·490 (7)
C(5′)—C(6′)	1·375 (4)	1·385 (6)
C(2)—N(1)—C(6)	117·5 (2)	117·1 (4)
N(1)—C(2)—C(3)	122·5 (2)	122·9 (4)
N(1)—C(2)—C(2′)	116·4 (2)	115·9 (3)
C(3)—C(2)—C(2′)	121·0 (2)	121·4 (4)
C(2)—C(3)—C(4)	118·8 (2)	118·3 (4)
C(3)—C(4)—C(5)	119·1 (3)	120·0 (4)
C(4)—C(5)—C(6)	118·3 (2)	117·8 (4)
N(1)—C(6)—C(5)	123·6 (2)	124·0 (4)
C(2′)—N(1′)—C(6′)	116·7 (2)	116·4 (3)
C(2)—C(2′)—N(1′)	116·5 (2)	116·7 (3)
C(2)—C(2′)—C(3′)	120·9 (2)	120·5 (4)
N(1′)—C(2′)—C(3′)	122·6 (2)	122·8 (4)
C(2′)—C(3′)—C(4′)	120·5 (2)	120·0 (4)
C(3′)—C(4′)—C(5′)	116·3 (2)	116·8 (3)
C(3′)—C(4′)—C(4′*a*)	121·5 (3)	120·8 (5)
C(5′)—C(4′)—C(4′*a*)	122·1 (2)	122·4 (5)
C(4′)—C(5′)—C(6′)	119·4 (2)	119·4 (4)
N(1′)—C(6′)—C(5′)	124·3 (2)	124·5 (4)

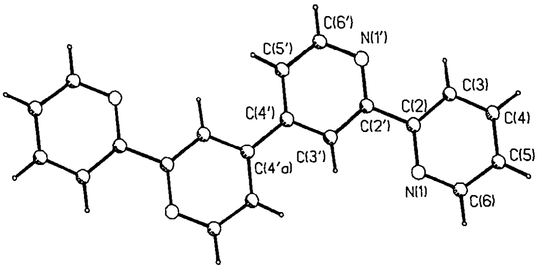


Fig. 1. Structure and atom labelling of 2,2′:4′,4″:2″,2‴-quaterpyridine.

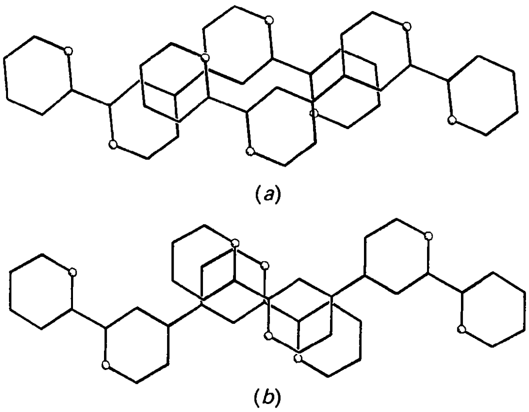


Fig. 2. Stacking of adjacent molecules for (*a*) the α-form and (*b*) the β-form.

refined with anisotropic displacement parameters; the H atoms were located in calculated positions with isotropic displacement parameters equal to the isotropic equivalent of their carrier C atom (C—H = 0·96 Å). The refinement converged at $R = 0{\cdot}052$, $wR = 0{\cdot}071$ ($g = 0{\cdot}0012$) and $S = 1{\cdot}49$ with 109 parameters for the α-form and at $R = 0{\cdot}047$, $wR = 0{\cdot}051$ ($g = 0{\cdot}0005$) and $S = 1{\cdot}26$ with 109 parameters for the β-form, with $w^{-1} = [\sigma^2(F) + g(F)^2]$ and $(\Delta/\sigma)_{\max} =$ 0·005 and 0·001 for the α- and β-forms, respectively, and $(\Delta\rho)_{\max}$, $(\Delta\rho)_{\min} = +0{\cdot}26$, $-0{\cdot}26$ and $+0{\cdot}21$, $-0{\cdot}22\ \text{e Å}^{-3}$ for the α- and β-forms, respectively. The atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV). All calculations, including the drawing of diagrams, were performed on a Nova 4X computer using *SHELXTL* (Sheldrick, 1983).

Table 1 lists the atom coordinates and Table 2 the bond lengths and angles.* Fig. 1 shows the structure and atom labelling. The two forms differ only in the crystal packing which shows the common herringbone pattern in both cases but with different interlayer stacking. Fig. 2 highlights the stacking differences and shows the overlap of adjacent molecules projected through their mean planes which are separated by 3·428 and 3·458 Å for the α- and β-forms respectively.

Related literature. The synthesis and a diruthenium(II) complex of the title compound have been described by Downard, Honey, Phillips & Steel (1991) and the structure and conformation of 2,2′-bipyridine by Almenningen, Bastiansen, Gundersen & Samdal (1989). For two preparations and the crystal structure of 2,2′:6′,2″:6″,2‴-quaterpyridine see Constable, Elder, Healy & Tocher (1990) and Uenishi, Tanaka, Wakabayashi, Oae & Tsukube (1990), and for 4,4′:2′,2″:4″,4‴-quaterpyridine see Morgan & Baker (1990). A survey of related binucleating ligands is given by Steel (1990).

---

* Lists of structure factors, anisotropic displacement parameters and calculated H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54200 (13 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Almenningen, A., Bastiansen, O., Gundersen, S. & Samdal, S. (1989). *Acta Chem. Scand.* **43**, 932–937.
Constable, E. C., Elder, S. M., Healy, J. & Tocher, D. A. (1990). *J. Chem. Soc. Dalton Trans.* pp. 1669–1674.
Downard, A. J., Honey, G. E., Phillips, L. F. & Steel, P. J. (1991). *Inorg. Chem.* **30**, 2259–2260.
Morgan, R. J. & Baker, A. D. (1990). *J. Org. Chem.* **55**, 1986–1993.
Sheldrick, G. M. (1983). *SHELXTL Users Manual.* Revision 4. Nicolet XRD Corporation, Madison, Wisconsin, USA.
Steel, P. J. (1990). *Coord. Chem. Rev.* **106**, 227–265.
Uenishi, J., Tanaka, T., Wakabayashi, S., Oae, S. & Tsukube, H. (1990). *Tetrahedron Lett.* **31**, 4625–4628.

Acta Cryst. (1991). C**47**, 2249–2252

Structures of Two *N*-Acyl Triethyl Ammonium Salts and One Simple Triethyl Ammonium Salt

By Joseph A. King Jr and Garold L. Bryant Jr

General Electric Company, Corporate Research and Development, Schenectady, NY 12301, *USA*

(*Received* 20 *November* 1990; *accepted* 29 *April* 1991)

Abstract. *N*-Benzoyl-*N*-triethylammonium tetraphenylborate (I), $C_{13}H_{20}NO^+.C_{24}H_{20}B^-$, $M_r = 525{\cdot}5$, monoclinic, $P2_1$, $a = 9{\cdot}666\,(2)$, $b = 9{\cdot}933\,(2)$, $c = 16{\cdot}023\,(3)$ Å, $\beta = 106{\cdot}01\,(2)°$, $V = 1478{\cdot}7$ Å^3, $Z = 2$, $D_x = 1{\cdot}180\ \text{g cm}^{-3}$, $\lambda(\text{Mo}\ K\alpha) = 0{\cdot}71069$ Å, $\mu = 0{\cdot}64\ \text{cm}^{-1}$, $F(000) = 564$, $T = 191$ K, $R = 0{\cdot}0483$ for 2844 unique reflections with $I > 4\sigma(I)$. *N*-Triethyl-*N*-phenoxycarbonylammonium tetrafluoroborate (II), $C_{13}H_{20}NO_2{}^+.BF_4^-$, $M_r = 309{\cdot}1$, monoclinic, $P2_1/c$, $a = 9{\cdot}196\,(3)$, $b = 13{\cdot}442\,(3)$, $c = 12{\cdot}718\,(3)$ Å, $\beta = 104{\cdot}46\,(2)°$, $V = 1522{\cdot}3$ Å^3, $Z = 4$, $D_x = 1{\cdot}349\ \text{g cm}^{-3}$, $\lambda(\text{Mo}\ K\alpha) = 0{\cdot}71069$ Å, $\mu = 1{\cdot}14\ \text{cm}^{-1}$, $F(000) = 648$, $T = 205$ K, $R = 0{\cdot}0477$ for 2853 unique reflections with $I > 3\sigma(I)$. Triethylammonium tetraphenylborate (III), $C_6H_{15}N^+.C_{24}H_{20}B^-$, $M_r = 421{\cdot}4$, monoclinic, $P2_1/m$, $a = 9{\cdot}291\,(4)$, $b = 14{\cdot}368\,(13)$, $c = 9{\cdot}559\,(7)$ Å, $\beta = 109{\cdot}76\,(5)°$, $V = 1200{\cdot}9$ Å^3, $Z = 2$, $D_x = 1{\cdot}165\ \text{g cm}^{-3}$, $\lambda(\text{Mo}\ K\alpha) = 0{\cdot}71069$ Å, $\mu = 0{\cdot}61\ \text{cm}^{-1}$, $F(000) = 456$, $T = 208$ K, $R = 0{\cdot}0743$ for 1274 unique reflections with $I > 4\sigma(I)$. (I) and (II) are *N*-acyl ammonium salts; (III) is an ammonium

0108-2701/91/102249-04$03.00