Acta Cryst. (1987). C**43**, 369–370

Bis(triphenylphosphoranediyl)ammonium 2,5,7-Tricyanoimidazo[3,4-*b*][1,2,4]-triazol-1-ide, a Salt of the $C_7N_7^-$ Anion

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(*Received* 14 *November* 1985; *accepted* 15 *August* 1986)

Abstract. $C_{36}H_{30}NP_2^+.C_7N_7^-$, $M_r = 720{\cdot}72$, monoclinic, $P2_1/c$, $a = 10{\cdot}977\ (2)$, $b = 24{\cdot}715\ (4)$, $c = 15{\cdot}210\ (3)$ Å, $\beta = 111{\cdot}77\ (2)°$, $U = 3832$ Å^3, $Z = 4$, $D_x = 1{\cdot}25$ g cm^{-3}, λ(Mo $K\alpha$) $= 0{\cdot}71069$ Å, $\mu = 1{\cdot}5$ cm^{-1}, $F(000) = 1496$, $T = 293$ K, $R = 0{\cdot}085$ for 3180 reflections. The anion is planar (r.m.s. deviation 0·016 Å); the C/N sites common to both its rings are disordered. The cation is bent at N [P—N—P 146·0 (4)°].

Experimental. Colourless equidimensional crystals with many well-formed faces, grown from methanol. Stoe–Siemens four-circle diffractometer, monochromated Mo $K\alpha$ radiation, profile-fitting mode (Clegg, 1981). $2\theta_{max}$ 45°, 5284 reflections, $+h+k\pm l$. Three check reflections, no significant intensity change. Crystal size 0·3 × 0·3 × 0·3 mm, no absorption correction. 4985 unique reflections (R_{int} 0·033), 3180 with $F > 3\sigma(F)$ used for all calculations (program system *SHELXTL*, Sheldrick, 1983). Index range: $|h| \le 10$, $|k| \le 26$, $|l| \le 16$. Cell constants refined from $\pm 2\theta$ values of 31 reflections in the range 20–23°.

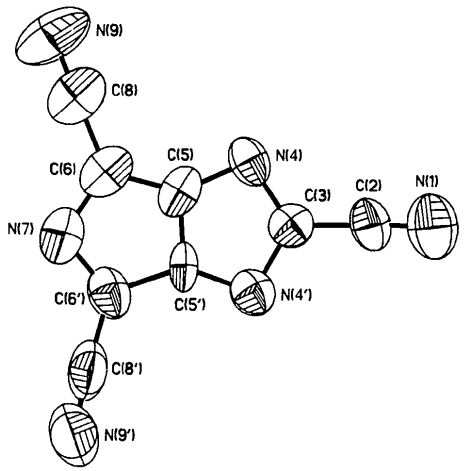

Fig. 1. Thermal ellipsoid plot of the $C_7N_7^-$ anion, showing the atom-numbering scheme. C(5) and C(5′) are the mixed C/N sites (see text).

Structure solution by routine direct methods. Refinement on F to R 0·085, wR 0·065. The rather high R value is due to the weakly diffracting nature of the

Table 1. *Atomic coordinates* (× 10^4) *and isotropic thermal parameters* (Å^2 × 10^3)

	x	y	z	U_{eq}*
N(1)	−159 (5)	4814 (2)	3796 (4)	106 (3)
C(2)	807 (6)	5005 (2)	4174 (4)	73 (3)
C(3)	2017 (6)	5268 (3)	4629 (4)	61 (3)
N(4)	3078 (5)	5009 (2)	5254 (4)	63 (3)
N(4′)	2171 (4)	5801 (2)	4454 (3)	66 (2)
C(5)	3950 (6)	5420 (3)	5487 (4)	62 (3)
C(5′)	3400 (5)	5871 (2)	5012 (4)	50 (2)
C(6)	5197 (7)	5539 (3)	6052 (4)	74 (4)
C(6′)	4362 (6)	6275 (3)	5317 (4)	70 (3)
N(7)	5450 (5)	6079 (3)	5942 (4)	82 (3)
C(8)	6141 (6)	5172 (3)	6687 (4)	91 (4)
C(8′)	4180 (8)	6819 (4)	4993 (6)	97 (4)
N(9)	6893 (6)	4885 (3)	7180 (4)	130 (4)
N(9′)	3967 (8)	7257 (3)	4696 (6)	146 (5)
P(1)	1527 (1)	2047 (1)	5307 (1)	40 (1)
P(2)	3801 (1)	1370 (1)	5302 (1)	43 (1)
N(11)	2940 (4)	1855 (2)	5412 (3)	46 (2)
C(12)	−382 (5)	1690 (2)	5966 (4)	49 (3)
C(13)	−822 (6)	1483 (2)	6633 (5)	58 (3)
C(14)	61 (6)	1315 (2)	7485 (5)	66 (3)
C(15)	1381 (6)	1358 (2)	7685 (4)	66 (3)
C(16)	1829 (5)	1575 (2)	7023 (4)	54 (3)
C(11)	951 (4)	1743 (2)	6155 (4)	42 (2)
C(22)	2541 (5)	3068 (2)	5347 (4)	56 (3)
C(23)	2614 (6)	3618 (3)	5515 (4)	68 (3)
C(24)	1767 (6)	3863 (3)	5859 (4)	71 (3)
C(25)	843 (7)	3566 (3)	6031 (5)	87 (4)
C(26)	755 (5)	3011 (2)	5865 (4)	73 (3)
C(21)	1610 (5)	2761 (2)	5520 (4)	43 (2)
C(32)	−246 (6)	2325 (2)	3506 (4)	59 (3)
C(33)	−1152 (7)	2199 (3)	2611 (5)	77 (4)
C(34)	−1508 (6)	1674 (3)	2376 (4)	67 (3)
C(35)	−975 (5)	1266 (3)	3005 (4)	61 (3)
C(36)	−91 (5)	1381 (2)	3898 (4)	52 (3)
C(31)	301 (4)	1918 (2)	4163 (4)	40 (2)
C(42)	2590 (5)	462 (3)	5663 (4)	59 (3)
C(43)	1820 (7)	7 (3)	5411 (6)	81 (4)
C(44)	1367 (7)	−177 (3)	4498 (7)	91 (5)
C(45)	1711 (7)	86 (3)	3829 (6)	89 (4)
C(46)	2499 (6)	543 (3)	4078 (5)	65 (3)
C(41)	2938 (5)	737 (2)	4995 (4)	46 (3)
C(52)	5620 (5)	1729 (3)	6981 (4)	63 (3)
C(53)	6653 (6)	1678 (3)	7831 (4)	77 (3)
C(54)	7220 (5)	1180 (3)	8109 (4)	81 (3)
C(55)	6777 (5)	737 (3)	7561 (4)	83 (3)
C(56)	5744 (5)	782 (3)	6699 (4)	64 (3)
C(51)	5146 (5)	1282 (2)	6406 (4)	46 (3)
C(62)	3819 (5)	1887 (3)	3713 (4)	67 (3)
C(63)	4270 (6)	1985 (3)	2988 (5)	92 (4)
C(64)	5309 (6)	1702 (3)	2949 (5)	99 (4)
C(65)	5943 (7)	1329 (3)	3638 (5)	112 (4)
C(66)	5490 (6)	1232 (3)	4356 (4)	86 (4)
C(61)	4434 (4)	1514 (2)	4402 (4)	48 (2)

* Equivalent isotropic U defined as one third of the trace of the orthogonalized U_{ij} tensor.

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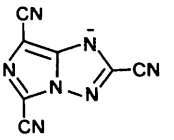
Table 2. *Selected bond lengths* (Å) *and angles* (°)

N(1)–C(2)	1·108 (8)	C(2)–C(3)	1·409 (9)
C(3)–N(4)	1·360 (7)	C(3)–N(4′)	1·367 (8)
N(4)–C(5)	1·351 (8)	N(4′)–C(5′)	1·312 (6)
C(5)–C(5′)	1·344 (8)	C(5)–C(6)	1·351 (8)
C(5′)–C(6′)	1·400 (8)	C(6)–N(7)	1·387 (11)
C(6)–C(8)	1·445 (9)	C(6′)–N(7)	1·312 (7)
C(6′)–C(8′)	1·421 (12)	C(8)–N(9)	1·134 (8)
C(8′)–N(9′)	1·164 (13)	P(1)–N(11)	1·572 (5)
P(1)–C(11)	1·797 (6)	P(1)–C(21)	1·791 (5)
P(1)–C(31)	1·790 (4)	P(2)–N(11)	1·573 (5)
P(2)–C(41)	1·799 (6)	P(2)–C(51)	1·790 (5)
P(2)–C(61)	1·786 (6)		

N(1)–C(2)–C(3)	177·5 (6)	C(2)–C(3)–N(4)	122·2 (6)
C(2)–C(3)–N(4′)	120·7 (5)	N(4)–C(3)–N(4′)	117·1 (5)
C(3)–N(4)–C(5)	99·7 (5)	C(3)–N(4′)–C(5′)	99·9 (4)
N(4)–C(5)–C(5′)	110·5 (5)	N(4)–C(5)–C(6)	141·5 (6)
C(5′)–C(5)–C(6)	108·0 (6)	N(4′)–C(5′)–C(5)	112·8 (5)
N(4′)–C(5′)–C(6′)	140·9 (5)	C(5)–C(5′)–C(6′)	106·2 (5)
C(5)–C(6)–N(7)	109·5 (6)	C(5)–C(6)–C(8)	126·5 (7)
N(7)–C(6)–C(8)	124·0 (6)	C(5′)–C(6′)–N(7)	110·5 (6)
C(5′)–C(6′)–C(8′)	124·6 (5)	N(7)–C(6′)–C(8′)	124·9 (6)
C(6)–N(7)–C(6′)	105·8 (5)	C(6)–C(8)–N(9)	179·0 (9)
C(6′)–C(8′)–N(9′)	176·5 (7)	N(11)–P(1)–C(11)	113·4 (2)
N(11)–P(1)–C(21)	107·3 (2)	C(11)–P(1)–C(21)	106·9 (3)
N(11)–P(1)–C(31)	113·7 (3)	C(11)–P(1)–C(31)	106·5 (2)
C(21)–P(1)–C(31)	108·8 (2)	N(11)–P(2)–C(41)	114·2 (3)
N(11)–P(2)–C(51)	108·2 (2)	C(41)–P(2)–C(51)	108·0 (3)
N(11)–P(2)–C(61)	110·5 (3)	C(41)–P(2)–C(61)	106·9 (3)
C(51)–P(2)–C(61)	108·9 (3)	P(1)–N(11)–P(2)	146·0 (3)

crystal. The C–N moiety common to both rings of the anion is disordered and both atoms were therefore refined as C with site occupation factor $1\frac{1}{12}$. All non-H atoms anisotropic; H atoms included using a riding model with C–H 0·96 Å, $U(H) = 1{\cdot}2\ U_{eq}(C)$; weighting scheme $w^{-1} = \sigma^2(F) + 0{\cdot}0003F^2$; 478 parameters; $S = 1{\cdot}56$; max. Δ/σ 0·04; max. features in final $\Delta\rho$ map 0·45, −0·35 e Å^{-3}. Atom scattering factors from *SHELXTL*. Final atomic coordinates are given in Table 1, and selected bond lengths and angles in Table 2.* Fig. 1 shows the atom-numbering scheme.

Related literature. Synthesis of other salts of the same anion (1) and correct suggestion of its structure: Wiley, Webster & Blanchard (1976).



(1)

We thank the Verband der Chemischen Industrie for financial support, and Professor H. W. Roesky and Mr H. Hofmann for providing the crystals.

* Lists of structure factors, H-atom coordinates, bond lengths and angles, and anisotropic thermal parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43070 (35 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Acta Cryst. (1987). C**43**, 370–372

α-[2′,5′-Bis(methoxymethyl)phenyl]-3,4,5-trimethoxyphenylacetonitrile

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(*Received* 18 *April* 1986; *accepted* 27 *August* 1986)

Abstract. $C_{21}H_{25}NO_5$, $M_r = 371{\cdot}43$, triclinic, $P\bar{1}$, $a = 7{\cdot}876\ (4)$, $b = 10{\cdot}435\ (5)$, $c = 12{\cdot}242\ (4)$ Å, $\alpha = 84{\cdot}87\ (3)$, $\beta = 83{\cdot}44\ (3)$, $\gamma = 106{\cdot}65\ (3)°$, $V = 949{\cdot}7\ (6)$ Å^3, $Z = 2$, $D_x = 1{\cdot}299$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, μ(Mo $K\alpha$) = 0·86 cm^{-1}, $F(000) = 396$, $T = 295$ K, final $R = 0{\cdot}043$ for 2556 observed reflections. There are no unusual bond lengths or angles.

Experimental. Colorless rectangular plates, unit-cell parameters by least-squares fit of 15 reflections in the range $17 < 2\theta < 25°$. Crystal 0·54 × 0·33 × 0·18 mm, automatic Syntex $P2_1$ diffractometer, graphite-monochromated Mo $K\alpha$ radiation, $\theta/2\theta$ scan mode, 3363 independent reflections in the range $3 < 2\theta < 50°$, hkl range h −9→9, k −12→12, l 0→14, 2556 observed reflections with $I > 3\sigma(I)$, $\sigma(I)$ from counting statistics; two standard reflections, 100 and 022, remeasured after every 100 reflections did not show any significant change in intensity; Lorentz–polarization correction, no absorption or extinction correction. Direct-methods *MULTAN*78 (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978), refinement by full-matrix least squares using *SHELX*76 (Sheldrick, 1976), anisotropic, H atoms located in difference Fourier map, H

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0108-2701/87/020370-03$01.50