$wR = 0.034$
$S = 2.1$
1209 reflections
204 parameters
All H-atom parameters refined

$\Delta\rho_{max} = 0.21$ e Å^{-3}
$\Delta\rho_{min} = -0.27$ e Å^{-3}
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

H atoms were refined with a common but variable C—H bond length.

Table 1. *Fractional atomic coordinates and equivalent isotropic thermal parameters* (Å^2)

$$U_{eq} = \tfrac{1}{3}\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
N11	0.4653 (1)	0.5211 (1)	0.2257 (5)	0.0426 (9)
N12	0.4705 (1)	0.5511 (1)	0.3952 (5)	0.0407 (9)
C13	0.5138 (1)	0.5702 (1)	0.4409 (6)	0.0328 (10)
C14	0.5547 (1)	0.5568 (1)	0.3175 (6)	0.0319 (10)
N15	0.5490 (1)	0.5298 (1)	0.1395 (5)	0.0360 (8)
C16	0.5039 (1)	0.5147 (1)	0.1002 (5)	0.0325 (10)
C31	0.5135 (1)	0.6054 (1)	0.6231 (6)	0.0325 (10)
C32	0.4778 (1)	0.6015 (1)	0.7827 (6)	0.0356 (11)
C33	0.4776 (1)	0.6330 (1)	0.9575 (7)	0.0434 (12)
C34	0.5117 (2)	0.6686 (1)	0.9726 (7)	0.0533 (13)
C35	0.5461 (1)	0.6742 (1)	0.8113 (7)	0.0556 (14)
C36	0.5468 (1)	0.6425 (1)	0.6372 (7)	0.0441 (12)
C41	0.6053 (1)	0.5688 (1)	0.3713 (7)	0.0349 (11)
C42	0.6243 (1)	0.5574 (1)	0.5739 (8)	0.0487 (14)
C43	0.6726 (2)	0.5670 (2)	0.6150 (9)	0.0690 (17)
C44	0.7012 (2)	0.5886 (2)	0.4607 (10)	0.0737 (18)
C45	0.6827 (2)	0.6004 (2)	0.2605 (9)	0.0632 (16)
C46	0.6346 (2)	0.5899 (1)	0.2161 (7)	0.0456 (12)

Table 2. *Geometric parameters* (Å, °)

N11—N12	1.343 (4)	C14—N15	1.338 (5)
N11—C16	1.332 (4)	C14—C41	1.478 (4)
N12—C13	1.341 (4)	N15—C16	1.340 (4)
C13—C14	1.414 (4)	C16—C16^{i}	1.496 (4)
C13—C31	1.488 (5)		
N12—N11—C16	116.7 (3)	C13—C14—C41	125.4 (3)
N11—N12—C13	120.2 (3)	N15—C14—C41	115.0 (3)
N12—C13—C14	120.0 (3)	C14—N15—C16	115.7 (3)
N12—C13—C31	114.4 (3)	N11—C16—N15	127.0 (3)
C14—C13—C31	125.7 (3)	N11—C16—C16^{i}	115.9 (3)
C13—C14—N15	119.6 (3)	N15—C16—C16^{i}	117.0 (3)

Symmetry code: (i) $1 - x, 1 - y, -z$.

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Lists of structure factors, anisotropic thermal parameters, H-atom coordinates, complete geometry and least-squares-planes deviations have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71093 (8 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: SE1020]

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Structure of 1,4-Benzenedicyanamide Dianion Derivatives

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Abstract

The crystal structures of four tetraphenylarsonium salts of 1,4-dicyanamidebenzene derivatives, namely, bis(tetraphenylarsonium) 1,4-benzenedicyanamide(2−), bis(tetraphenylarsonium) 2,5-dimethyl-1,4-benzenedicyanamide(2−), bis(tetraphenylarsonium) 2,5-dichloro-1,4-benzenedicyanamide(2−) and bis(tetraphenylarsonium) 2,3,5,6-tetrachloro-1,4-benzenedicyanamide(2−), were determined. All of the derivatives are essentially planar with the cyanamide groups in an *anti* configuration, suggesting a significant degree of π coupling between the cyanamide groups and phenyl ring. No evidence was seen of π stacking for any of the 1,4-dicyanamidebenzene dianion derivatives.

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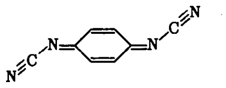
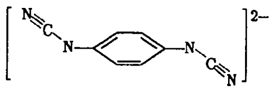
Comment

Aumuller & Hunig (1986) have recently prepared a novel series of planar acceptor molecules (Gerson, Gescheidt, Mockel, Aumuller, Erk & Hunig, 1988), the dicyanoquinonediimines [DCNQI, (I)].



(I)



(II)

Charge-transfer complexes with the stoichiometry Cu(DCNQI)$_2$, that incorporate the radical anion DCNQI, have been shown to possess metallic properties and in one instance conductivity increased with decreasing temperature reaching a maximum of 5×10^5 S m^{-1} at 3.5 K (Aumuller, Erk, Klebe, Hunig, Von Schutz & Werner, 1986). These properties are largely the result of the planarity of the radical anion DCNQI molecule and its ability to form π stacks in the solid state (Kato, Kobayashi & Kobayashi, 1989).

The DCNQI molecule is simply the two-electron oxidation product of the anionic 1,4-dicyanamidebenzene (Dicyd^{2-}) compound, (II), of this study. We are currently studying the solid-state electron-exchange mediating properties of Dicyd^{2-} and its derivatives which are expected to be maximized when planarity is maintained (Aquino, Lee, Gabe, Greedan & Crutchley, 1991; Aquino, Lee, Gabe, Bensimon, Greedan & Crutchley, 1992). While the addition of one electron to form the radical anion DCNQI molecule did not result in loss of planarity, the perturbation of two-electron addition on DCNQI geometry has yet to be evaluated.

The preparations of the tetraphenylarsonium salts of 1,4-dicyanamidebenzene dianion (Dicyd^{2-}), 1,4-dicyanamide-2,5-dimethylbenzene dianion (Me$_2$Dicyd^{2-}) and 2,5-dichloro-1,4-dicyanamidebenzene dianion (Cl$_2$Dicyd^{2-}) follow essentially the same procedure as that for 2,3,5,6-tetrachloro-1,4-dicyanamidebenzene dianion (Cl$_4$Dicyd^{2-}) which has been published (Aquino, Bostock & Crutchley, 1990). Unlike the tetrachloro derivative, these compounds can be easily isolated pure in their neutral protonated form. Yellow (Dicyd^{2-}, Cl$_2$Dicyd^{2-} and Cl$_4$Dicyd^{2-}) or red (Me$_2$Dicyd^{2-}) cubic crystals suitable for crystallographic analyses were grown under argon by the slow cooling of warm saturated DMSO solutions of the tetraphenylarsonium salts.

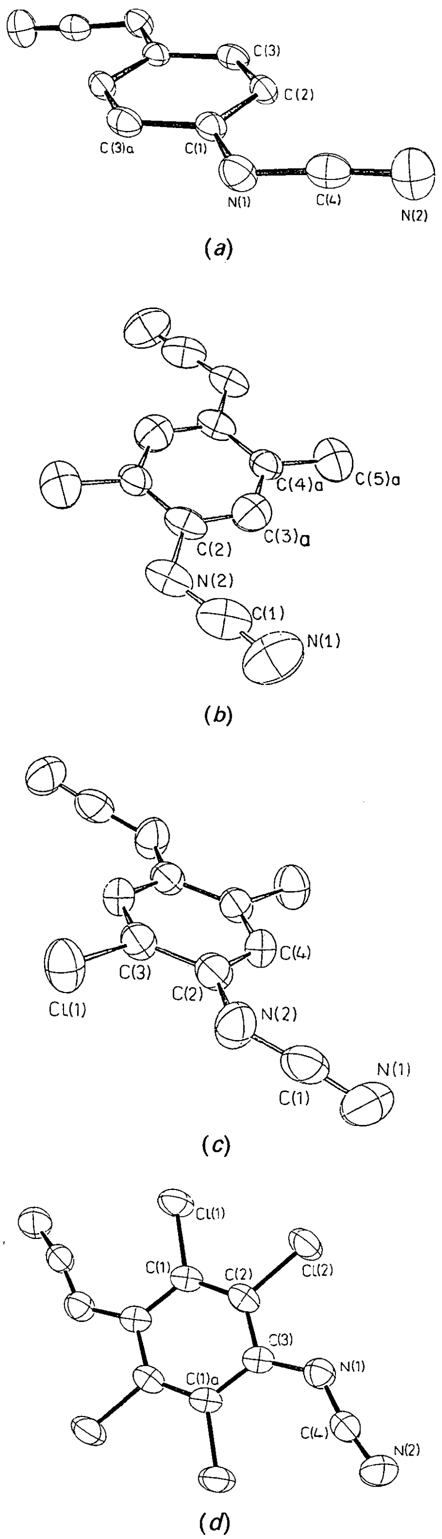


Fig. 1. *ORTEP* diagrams of (*a*) 1,4-dicyanamidebenzene dianion, (*b*) 1,4-dicyanamide-2,5-dimethylbenzene dianion, (*c*) 2,5-dichloro-1,4-dicyanamidebenzene dianion and (*d*) 2,3,5,6-tetrachloro-1,4-dicyanamidebenzene dianion. The tetraphenylarsonium ions have been excluded for clarity.

All of the dianion derivatives are almost planar, the terminal nitrile nitrogen being no more than 0.31 (13) Å out of the plane formed by the central phenyl ring. In all cases the cyanamide groups are in an *anti* configuration and are almost linear, with the NCN bond angle ranging from 168.8 (5) to 177.5 (19)°.

A slight contraction of bond lengths for the phenyl carbon to amide nitrogen bond [1.408 (19)–1.360 (6) Å is observed with increasing electron-withdrawing properties of the substituents on the phenyl ring. For Cl_4Dicyd^{2-}, the steric repulsion of the *ortho* chlorine substituents is not of sufficient magnitude to overcome the π coupling between the phenyl ring and the cyanamide groups and so force the cyanamide groups out of the phenyl ring plane. This is not the case for 1,4-dicyanamide-2,3,5,6-tetramethylbenzene dianion where the steric repulsion of *ortho* methyl groups is enough to force the cyanamide groups out of plane while still maintaining an *anti* configuration (Aquino, Lee, Gabe, Greedan & Crutchley, 1991).

The crystal lattices of alkali-metal salts of radical anion DCNQI derivatives have shown the DCNQI to be ordered into π-stacks (Kato, Kobayashi, Kobayashi, Mori & Inokuchi, 1988). This was not observed in the crystal lattices of the $Dicyd^{2-}$ derivatives of this study. We are currently investigating transition metal cations that may promote π-stack formation of $Dicyd^{2-}$ derivatives.

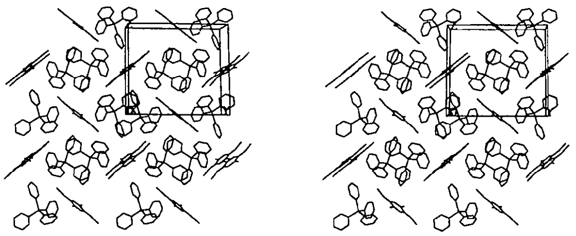


Fig. 2. Stereoview of $[AsPh_4]_2[Me_2Dicyd]$ unit cells.

Experimental

$Dicyd^{2-}$

Crystal data

$[As(C_6H_5)_4]_2.C_8H_4N_4$
$M_r = 922.82$
Monoclinic
$P2_1/a$
$a = 9.7659$ (6) Å
$b = 21.6128$ (13) Å
$c = 11.1040$ (7) Å
$\beta = 110.560$ (10)°
$V = 2194.42$ (23) Å^3
$Z = 2$
$D_x = 1.397$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.70930$ Å
Cell parameters from 21 reflections
$\theta = 17.5$–$22.5°$
$\mu = 1.56$ mm^{-1}
$T = 295$ K
Cubic
0.25 × 0.20 × 0.20 mm
Red
Crystal source: recrystallized from DMSO

Data collection

Enraf–Nonius diffractometer
$\theta/2\theta$ scans
Absorption correction: none
4910 measured reflections
2848 independent reflections
1804 observed reflections [$I > 2.5\sigma(I)$]
$R_{\rm int} = 0.020$

$\theta_{\rm max} = 22.4°$
$h = -10 \rightarrow 9$
$k = 0 \rightarrow 22$
$l = 0 \rightarrow 11$
3 standard reflections
frequency: 150 min
intensity variation: 0.902%

Refinement

Refinement on F
Final $R = 0.040$
$wR = 0.034$
$S = 1.290$
1804 reflections
347 parameters

All H-atom parameters refined
$(\Delta/\sigma)_{\rm max} = 0.633$
$\Delta\rho_{\rm max} = 0.340$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.360$ e Å^{-3}
Extinction coefficient: 0.150 (67) μm

Me_2Dicyd^{2-}

Crystal data

$[As(C_6H_5)_4]_2.C_{10}H_8N_4$
$M_r = 950.88$
Monoclinic
$P2_1/n$
$a = 9.8860$ (14) Å
$b = 16.416$ (5) Å
$c = 14.909$ (3) Å
$\beta = 99.391$ (17)°
$V = 2387.2$ (10) Å^3
$Z = 2$
$D_x = 1.323$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.70930$ Å
Cell parameters from 25 reflections
$\theta = 20$–$23.5°$
$\mu = 1.44$ mm^{-1}
$T = 295$ K
Cubic
0.20 × 0.20 × 0.20 mm
Red
Crystal source: recrystallized from DMSO

Data collection

Rigaku diffractometer
$\theta/2\theta$ scans
Absorption correction: ψ-scan
$T_{\rm min} = 0.6129$, $T_{\rm max} = 0.7251$
3759 measured reflections
3530 independent reflections
1648 observed reflections [$I > 2.5\sigma(I)$]

$R_{\rm int} = 0.05$
$\theta_{\rm max} = 23.45°$
$h = -11 \rightarrow 10$
$k = 0 \rightarrow 18$
$l = 0 \rightarrow 16$
3 standard reflections
frequency: 150 min
intensity variation: 0.757%

Refinement

Refinement on F
Final $R = 0.072$
$wR = 0.051$
$S = 3.63$
1648 reflections
290 parameters

H-atom parameters not refined
$(\Delta/\sigma)_{\rm max} = 0.105$
$\Delta\rho_{\rm max} = 0.570$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.680$ e Å^{-3}
Extinction coefficient: 0.183 (130) μm

Cl_2Dicyd^{2-}

Crystal data

$[As(C_6H_5)_4]_2.C_8H_4Cl_2N_4$
$M_r = 991.71$

Mo $K\alpha$ radiation
$\lambda = 0.70930$ Å

Monoclinic
$P2_1/n$
$a = 9.8673$ (23) Å
$b = 18.3696$ (19) Å
$c = 13.1791$ (11) Å
$\beta = 102.875$ (14)°
$V = 2328.8$ (16) Å^3
$Z = 2$
$D_x = 1.414$ Mg m^{-3}

Cell parameters from 23 reflections
$\theta = 20$–22.5°
$\mu = 1.59$ mm^{-1}
$T = 295$ K
Cubic
0.40 × 0.30 × 0.20 mm
Yellow
Crystal source: recrystallized from DMSO

Data collection

Enraf–Nonius diffractometer
$\theta/2\theta$ scans
Absorption correction: none
3068 measured reflections
3041 independent reflections
2685 observed reflections [$I > 2.5\sigma(I)$]

$R_{\rm int} = 0.006$
$\theta_{\rm max} = 22.45°$
$h = -10 \rightarrow 10$
$k = 0 \rightarrow 19$
$l = 0 \rightarrow 14$
3 standard reflections
frequency: 150 min
intensity variation: 0.55%

Refinement

Refinement on F
Final $R = 0.026$
$wR = 0.020$
$S = 4.02$
2685 reflections
374 parameters

All H-atom parameters refined
$(\Delta/\sigma)_{\rm max} = 280$
$\Delta\rho_{\rm max} = 0.260$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.290$ e Å^{-3}
Extinction coefficient: 0.842 (32) μm

$\mathbf{Cl_4Dicyd^{2-}}$

Crystal data

$[\mathrm{As(C_6H_5)_4}]_2.\mathrm{C_8Cl_4N_4}$
$M_r = 1060.60$
Triclinic
$P\bar{1}$
$a = 10.0609$ (10) Å
$b = 10.2590$ (11) Å
$c = 12.0028$ (10) Å
$\alpha = 101.936$ (11)°
$\beta = 103.436$ (8)°
$\gamma = 90.783$ (10)°
$V = 1176.42$ (22) Å^3
$Z = 4$
$D_x = 1.497$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.70930$ Å
Cell parameters from 112 reflections
$\theta = 20$–22.5°
$\mu = 3.38$ mm^{-1}
$T = 295$ K
Cubic
0.40 × 0.20 × 0.20 mm
Yellow
Crystal source: recrystallized from DMSO

Data collection

Enraf–Nonius diffractometer
$\theta/2\theta$ scans
Absorption correction: none
8199 measured reflections
5627 independent reflections
3606 observed reflections [$I > 2.5\sigma(I)$]
$R_{\rm int} = 0.040$

$\theta_{\rm max} = 22.45°$
$h = -12 \rightarrow 12$
$k = 0 \rightarrow 13$
$l = -15 \rightarrow 15$
3 standard reflections
frequency: 150 min
intensity variation: 0.731%

Refinement

Refinement on F
Final $R = 0.048$
$wR = 0.029$
$S = 1.974$
3606 reflections
379 parameters

H-atom parameters not refined
$(\Delta/\sigma)_{\rm max} = 0.506$
$\Delta\rho_{\rm max} = 0.900$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.790$ e Å^{-3}
Extinction coefficient: 0.052 (13) μm

Table 1. *Fractional atomic coordinates and equivalent isotropic thermal parameters* (Å^2)

$B_{\rm iso}$ is the mean of the principal axes of the thermal ellipsoid.

	x	y	z	$B_{\rm iso}$
$\mathrm{Dicyd^{2-}}$				
N(1)	0.5426 (5)	0.39489 (22)	0.3650 (4)	3.8 (3)
N(2)	0.7843 (6)	0.36206 (24)	0.3644 (5)	5.2 (3)
C(1)	0.5270 (6)	0.4465 (3)	0.4335 (5)	3.1 (3)
C(2)	0.3900 (7)	0.4595 (3)	0.4413 (5)	3.5 (3)
C(3)	0.3636 (6)	0.5106 (3)	0.5051 (5)	3.1 (3)
C(4)	0.6726 (9)	0.3799 (3)	0.3684 (5)	3.7 (3)
H(2)	0.312 (5)	0.4301 (22)	0.397 (4)	4.0
H(3)	0.269 (5)	0.5181 (23)	0.503 (5)	3.8
$\mathrm{Me_2Dicyd^{2-}}$				
N(1)	0.5626 (20)	0.7084 (11)	0.8075 (12)	7.5 (10)
N(2)	0.4000 (14)	0.6329 (8)	0.8856 (8)	4.8 (6)
C(1)	0.4889 (21)	0.6721 (10)	0.8441 (11)	5.1 (9)
C(2)	0.4485 (16)	0.5672 (8)	0.9425 (9)	4.0 (6)
C(3)	0.4150 (17)	0.4621 (10)	1.0487 (11)	4.6 (7)
C(4)	0.3618 (18)	0.5258 (9)	0.9932 (9)	3.7 (6)
C(5)	0.2147 (18)	0.5552 (11)	0.9909 (12)	5.6 (8)
H(3)	0.346	0.433	1.090	5.4
$\mathrm{Cl_2Dicyd^{2-}}$				
Cl	0.31768 (7)	0.96919 (4)	1.05046 (6)	4.88 (4)
N(1)	−0.0486 (3)	0.75236 (13)	0.93366 (21)	6.16 (15)
N(2)	0.10354 (22)	0.85947 (13)	0.97190 (18)	4.64 (13)
C(1)	0.0179 (3)	0.80482 (17)	0.95150 (22)	4.30 (16)
C(2)	0.0495 (3)	0.92818 (15)	0.98368 (18)	3.55 (14)
C(3)	0.1394 (3)	0.98515 (15)	1.02200 (19)	3.49 (13)
C(4)	−0.0916 (3)	0.94632 (16)	0.96122 (21)	3.75 (14)
H(4)	−0.1556 (21)	0.9144 (11)	0.9427 (16)	3.0 (6)
$\mathrm{Cl_4Dicyd^{2-}}$				
Cl(1)	0.88530 (12)	0.54855 (12)	0.24951 (19)	4.34 (6)
Cl(2)	1.18638 (12)	0.48099 (13)	0.32588 (10)	4.78 (7)
C(1)	0.9483 (4)	0.5234 (4)	0.3896 (3)	2.86 (21)
C(2)	1.0816 (4)	0.4915 (4)	0.4242 (4)	3.01 (21)
C(3)	0.8569 (4)	0.5354 (4)	0.4654 (4)	2.91 (22)
N(1)	0.7241 (4)	0.5678 (3)	0.4438 (3)	3.66 (19)
C(4)	0.6503 (5)	0.6087 (4)	0.3551 (4)	3.35 (23)
N(2)	0.5684 (4)	0.6489 (4)	0.2865 (3)	5.09 (25)

Table 2. *Selected bond lengths* (Å), *angles* (°) *and out of plane distances*† (Å)

$\mathrm{Dicyd^{2-}}$			
N(1)—C(1)	1.388 (8)	C(1)—C(2)	1.399 (9)
N(1)—C(4)	1.299 (10)	C(2)—C(3)	1.385 (9)
N(2)—C(4)	1.172 (10)	C(2)—H(2)	0.98 (5)
C(1)—N(1)—C(4)	118.7 (5)	C(1)—C(2)—C(3)	123.1 (5)
N(1)—C(1)—C(2)	119.1 (5)	C(1)—C(2)—H(2)	115 (3)
C(2)—C(1)—C(3)*a*	114.8 (5)	N(1)—C(4)—N(2)	174.1 (6)
N(1)	0.31 (13)	N(2)	0.201 (19)
C(4)	0.097 (16)		
$\mathrm{Me_2Dicyd^{2-}}$			
C(3)—H(3)	1.100 (16)	N(2)—C(2)	1.408 (19)
N(1)—C(1)	1.15 (3)	C(4)—C(5)	1.527 (22)
N(2)—C(1)	1.32 (3)	C(2)—C(4)	1.406 (21)
C(2)—C(4)—C(5)	119.8 (13)	N(2)—C(2)—C(4)	121.1 (14)
C(3)*a*—C(2)—C(4)	116.2 (13)	C(1)—N(2)—C(2)	117.9 (14)
C(2)—C(4)—C(3)	118.8 (13)	N(1)—C(1)—N(2)	177.5 (19)
N(2)	0.001 (19)	N(2)	0.00 (3)
C(1)	−0.005 (23)		

Cl_2Dicyd^{2-}			
Cl—C(3)	1.740 (3)	C(2)—C(3)	1.392 (4)
N(1)—C(1)	1.160 (4)	C(3)—C(4)*a*	1.379 (4)
N(2)—C(1)	1.301 (4)	C(4)—H(4)	0.857 (21)
N(2)—C(2)	1.392 (3)		
C(1)—N(2)—C(2)	118.38 (22)	C(3)—C(2)—C(4)	114.96 (24)
N(1)—C(1)—N(2)	174.2 (3)	Cl—C(3)—C(2)	118.92 (21)
N(2)—C(2)—C(3)	119.53 (23)	C(2)—C(3)—C(4)*a*	122.10 (24)
N(2)—C(2)—C(4)	125.51 (25)		
N(2)	0.003 (3)	N(1)	0.305 (6)
C(1)	0.216 (5)		
Cl_4Dicyd^{2-}		C(1)—C(3)	1.426 (6)
Cl(1)—C(1)	1.728 (4)	C(3)—N(1)	1.360 (6)
C(1)—C(2)	1.374 (6)	N(1)—C(4)	1.299 (6)
		C(4)—N(2)	1.167 (7)
Cl(1)—C(1)—C(2)	120.1 (3)	C(2)*a*—C(3)—N(1)	118.9 (4)
C(2)—C(1)—C(3)	122.3 (4)	C(3)—N(1)—C(4)	130.1 (4)
Cl(2)—C(2)—C(3)*a*	115.7 (3)	N(1)—C(4)—N(2)	168.8 (5)
N(1)	0.015 (6)	N(2)	0.290 (16)
C(4)	0.153 (12)		

† Distance from cyanamide atoms to the phenyl ring plane.

All chemicals and solvents were reagent grade or better. Tetraphenylarsonium chloride monohydrate (Strem), phenylenediamine (Aldrich), 2,5-dichloro-1,4-phenylenediamine (Aldrich) and 2,5-dimethyl-1,4-phenylenediamine (Aldrich) were used as received.

A $\theta/2\theta$ scan technique was employed with profile analysis (Grant & Gabe, 1978). The Me_2Dicyd^{2-} salt is easily oxidized and so measurements were carried out on a crystal sealed in a tube. No crystal decay was observed during measurement for any of the compounds of this study. The space groups were determined either by systematic absences or during the analysis.

The structures were solved by direct methods and refined by full-matrix least squares using weights based on counting statistics. H-atom positions were calculated. All the calculations were performed with the *NRCVAX* package (Gabe, Lee, Le Page, Charland & White, 1989). Atomic scattering factors were obtained from *International Tables for X-ray Crystallography* (1974, Vol. IV).

Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71067 (108 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: CD1008]

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Structure of a Chiral Bicyclic 1-Hydroxyalkyl Phosphonamide

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Abstract

The crystal structure of a chiral racemic 1-hydroxyalkyl phosphonamide, 1,3-(2,2-dimethylpropyl)-2-[1-hydroxy-3-phenyl-(*E*)-2-propenyl]-2,3,3a,4,5,6,7,-7a-octahydro-1*H*-1,3,2-benzodiazaphosphole 2-oxide, m.p. 455-456 K, has been determined by X-ray diffraction. The title compound consists of a five-membered ring with a tetrahedrally coordinated phosphorus atom. In addition to the two N atoms of the five-membered ring, the phosphorus is bonded to an oxygen and the 1-carbon of a hydroxyalkyl chain. The five-membered ring is *trans* fused to a cyclohexane in the chair conformation. The solid-state structure contains an intermolecular hydrogen-bonded enantiomeric pair.

Comment

We recently reported the preparation and reactions of chiral phosphorous acid diamides (Koeller & Spilling, 1991). Treatment of phosphorous acid diamide (1) with butyllithium in tetrahydrofuran (THF) solution followed by addition of cinnamaldehyde to the resulting anion gave the 1-hydroxy phosphonamides (2) in an 8:1 ratio of diastereoisomers (Koeller & Spilling, 1993). Crystallization from an ethyl acetate/hexane mixture yielded only the major isomer. Evidence for the relative configuration of C(17) was not available by standard spectroscopic methods. Single-crystal X-ray diffraction analysis allowed assignment of the relative configuration of the new chiral centre as *R* resulting from the (*S*,*S*)-diamide.

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