Table 3. *Comparison of observed (X-ray) and calculated molecular geometry parameters (Å, °) for (I) and (II)*

(I) was constrained in the molecular-orbital calculations to C_{2v} symmetry and (II) to C_s symmetry.

(I)	X-ray	SCF	MP2	(II)	X-ray	SCF	MP2
N4—C4*M*	1.463 (2)	1.4481	1.4537	N1—C1*M*	1.455 (2)	1.4480	1.4524
N4—C3	1.345 (3)	1.3537	1.3624	N1—N2	1.344 (2)	1.3234	1.3411
C3—N2	1.309 (3)	1.2802	1.3199	N2—N3	1.299 (2)	1.2512	1.3180
N2—N1	1.395 (2)	1.3605	1.3783	N3—N4	1.360 (2)	1.3356	1.3555
N1—C5	1.300 (3)	1.2802	1.3199	C5—N4	1.315 (2)	1.2898	1.3220
C5—N4	1.348 (2)	1.3537	1.3624	C5—N1	1.331 (2)	1.3273	1.3465
C4*M*—N4—C5	127.6 (2)	128.33	127.98	C1*M*—N1—C5	130.78 (15)	131.25	130.73
C4*M*—N4—C3	127.9 (2)	128.33	127.98	C1*M*—N1—N2	120.81 (13)	121.56	120.86
C3—N4—C5	104.5 (2)	103.34	104.03	N2—N1—C5	108.42 (14)	107.91	108.41
N2—C3—N4	111.3 (2)	110.99	111.04	N3—N2—N1	106.23 (12)	107.33	106.21
N1—N2—C3	106.2 (2)	107.34	106.94	N4—N3—N2	110.78 (14)	111.19	110.71
C5—N1—N2	106.5 (2)	107.34	106.94	C5—N4—N3	105.39 (14)	105.48	105.71
N4—C5—N1	111.5 (2)	110.99	111.04	N1—C5—N4	109.2 (2)	108.71	108.96

All quantum-mechanical calculations were performed with the *GAMESS-UK* package (Dupuis, Spangler & Wendoloski, 1980; Guest, Kendrick, van Lenthe, Schoeffel & Sherwood, 1995); the MP2 total energies for compounds (I) and (II) were −280.95197 and −296.95764 a.u., respectively (1 a.u. = 2626 kJ mol^{-1}).

For both compounds, data collection: *DIF*4 (Stoe & Cie, 1990*a*); cell refinement: *DIF*4; data reduction: *REDU*4 (Stoe & Cie, 1990*b*); program(s) used to solve structures: *SIR*92 (Altomare *et al.*, 1994); program(s) used to refine structures: *SHELXTL/PC* (Sheldrick, 1995); molecular graphics: *SHELXTL/PC*; software used to prepare material for publication: *SHELXTL/PC*.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: CF1129). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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17β-Isopropylsparteine and 17β-Isopropyllupanine Perchlorates

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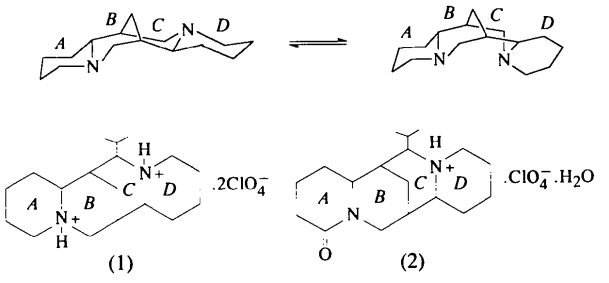
Abstract

In the sparteine derivative, (1), $C_{18}H_{34}N_2^{2+}.2ClO_4^-$ {sparteine = [7*S*-(7α,7aα,14α,14a*β*)]-dodecahydro-7,14-methano-2*H*,6*H*-dipyrido[1,2-*a*:1′,2′-*e*][1,5]diazocine}, the quinolizidine skeletons (*A*/*B* and *C*/*D* systems) have a *trans*/*trans* configuration with chair, chair, distorted boat and chair conformations for the *A*, *B*, *C* and

D rings, respectively. In the lupanine derivative, (2), $C_{18}H_{31}N_2O^+.ClO_4^-.H_2O$ {lupanine = dodecahydro-7,14-methano-4*H*,6*H*-dipyrido[1,2-*a*:1′,2′-*e*][1,5]diazocin-4-one}, the configuration of the cation is quasi-*trans* and *trans* for the *A/B* and *C/D* ring systems, respectively, and the ring conformations are intermediate between sofa and half-chair, chair, distorted boat and chair for the *A*, *B*, *C* and *D* rings, respectively. In both (1) and (2), bulky isopropyl substituents are responsible for large deformations of the bisquinolizidine skeletons. Hydrogen bonds play a major role in crystal packing.

Comment

Lupine alkaloids form a group of chiral bisquinolizidine compounds with a semi-rigid stereostructure. This semi-rigidity enables them to act as bidentate ligands, provided the N16 atom can invert its configuration, converting ring *C* into a chair conformation in contrast to its boat form in the free base (see reaction scheme). In particular, the chelating properties of sparteine have been widely used in enantioselective organic syntheses (Borowiak & Wolska, 1996).



One of the factors influencing the configuration and conformation of bisquinolizidine alkaloids is the introduction of substituents to their labile *C/D* fragment. The methyl group introduced on C17 in a β-orientation stabilizes the 'transoidal' arrangement of both N atoms (ring *C* has a boat form in the 'transoidal' arrangement). This is the case for 17β-methylsparteine which does not form a monocation on protonation as sparteine does, but easily transforms into a dication (Wiewiórowski, Pieczonka & Skolik, 1977; Pyżalska, Gawron & Borowiak, 1979). The conformation of the dication remains the same as that of the free base.

Lupanine is a more complicated case. In the lactam group in ring *A*, the free electron pair of the N1 atom is involved in a strongly conjugated system, N1···C2···O2, and for this reason N1 is inaccessible to protonation, so only monosalts of lupanine can be formed. The configuration and conformation of the lupanine free base are retained in its hydrated salts, *e.g.* in (+)-lupanine hydrochloride dihydrate (Skrzypczak-Jankun & Kałuski, 1978) or (+)-lupanine perchlorate monohydrate (Małuszyńska, Hoser & Kałuski, 1979) whereas in anhydrous (+)-lupanine perchlorate (Skrzypczak-Jankun, Hoser, Kałuski & Perkowska, 1980) or 13-hydroxylupanine (Kałuski *et al.*, 1977), an inversion of the N16 configuration and chair conformation of ring *C* has been found.

The introduction of a methyl group into the lupanine skeleton in the 17β-orientation stabilizes the 'transoidal' arrangement of both N atoms in the case of 17β-methylsparteine perchlorate (Skrzypczak-Jankun, Małuszyńska, Perkowska & Kałuski, 1980). On the other hand, IR spectra of 17β-methylsparteine and 17β-methyllupanine in $CDCl_3$ in the region 2300–2000 cm^{-1} show that the equatorial methyl groups only partly block the access of the solvent molecules to the N16 atom whereas the 17β-isopropyl groups block this access completely. This effect, which prevents the configurational/conformational changes, has been called an anchor effect (Boczoń, 1989). Here we confirm these anchor effects for bulkier 17β-isopropyl substituents which produce even greater deformations

Bond lengths and angles for (1) and (2) are in agreement with those found in other sparteinium and lupaninium cations (Borowiak & Wolska, 1996, and references therein); the mean C—C length for sp^3 C atoms is 1.522 (5) for (1) and 1.524 (3) Å for (2).

In compound (1) the *A/B* ring junction is *trans* [torsion angles C2—N1—C6—C5 −55.8 (9), C7—C6—N1—C10 46.3 (8)°], as is the *C/D* junction [C9—C11—N16—C17 −51.3 (7), C12—C11—N16—C15 56.7 (7)°] (Fig. 1). The enantiomorph is that of the naturally occurring sparteine derivatives, both for (1) and (2) (Okuda, Kataoka & Tsuda, 1965; Klyne *et al.*, 1974) with C7 and C9 *S*. Rings *A*, *B* and *D* show almost no deviations from the ideal chair conformations. The largest distortions from the ideal form have been found for the *C* ring which adopts a distorted boat conformation. Asymmetry parameters (Duax & Norton, 1975) are as high as ΔC_s^8 = 10.7 (6) and $\Delta C_s^{9,11}$ = 18.1 (6)°. The isopropyl substituent is equato-

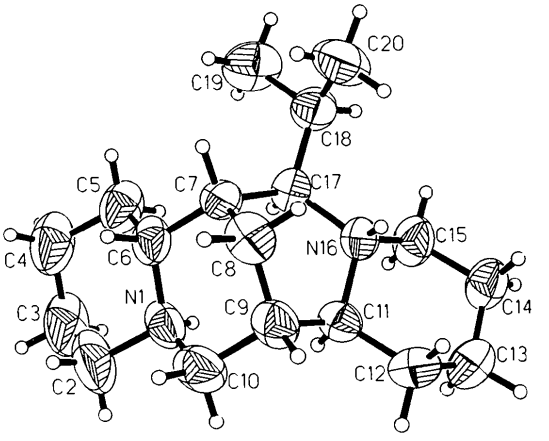


Fig. 1. View of the dication of (1) with displacement ellipsoids drawn at the 50% probability level.

rial [torsion angles C11—N16—C17—C18 +170.6 (5), C8—C7—C17—C18 −109.7 (6)°]. The latter, a rather small value, is the result of a large strain in the molecule caused by the bulky isopropyl group. This strain is also evidenced by the asymmetry in the values of the valence angles which characterize the inclination of one quinolizidine skeleton with respect to the other [C6—C7—C17 116.2 (5), C10—C9—C11 111.1 (6)°]. Each of the quarternary N atoms is hydrogen-bonded to one of the perchlorate anions. No other short contacts in the crystal packing have been found.

In (2), N16 has been protonated and the ring *C* conformation is that of the free base (Wolska, Borowiak & Boczoń, 1993) (Fig. 2). The *A/B* and *C/D* ring junctions are the same as in the free base, *i.e.* quasi-*trans* [C2—N1—C6—C5 −11.7 (6), C10—N1—C6—C7 +47.6 (5)°] and *trans* [C9—C11—N16—C17 −59.9 (5), C15—N16—C11—C12 +52.1 (5)°], respectively. As in the unsubstituted (±)-lupanine (Doucerain, Chiaroni & Riche, 1976), ring *A* adopts a conformation intermediate between sofa and half-chair. The deviations of ring *C* from an ideal boat conformation are high, with asymmetry parameters ΔC_s^8 = 17.6 (4) and $\Delta C_s^{9,11}$ = 13.0 (5)°. The isopropyl substituent in (2) is also equatorial with torsion angles C11—N16—C17—C18 +173.6 (4) and C8—C7—C17—C18 −109.7 (5)°. The asymmetry in the inclination of one quinolizidine skeleton with respect to the other is also very high [C6—C7—C17 117.3 (4), C10—C9—C11 108.3 (4)°]. Compound (2) crystallizes with one water molecule per asymmetric unit and in the crystal packing all proton donors are involved in intermolecular or interionic hydrogen bonds. The ribbons of hydrogen-bonded species extend along the *b* axis (Fig. 3).

In conclusion, it seems unlikely that there will be applications in asymmetric synthesis for 17β-substituted sparteine and lupanine because of their rigidity.

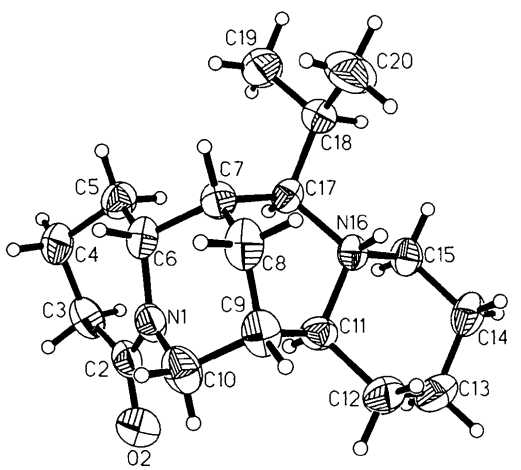


Fig. 2. View of the cation of (2) with displacement ellipsoids drawn at the 50% probability level.

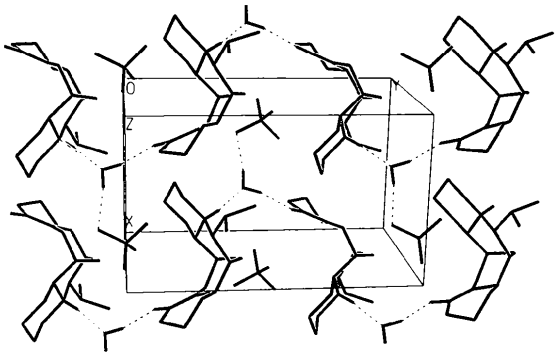


Fig. 3. A perspective drawing of the packing arrangement of (2) viewed along the *c* axis with dashed lines indicating hydrogen bonds.

Experimental

The details of the synthesis of (1) and (2) have been published (Boczoń, 1989). Crystals of (1) were grown with difficulty from methyl alcohol by slow evaporation, while those of (2) by vapour diffusion from an ethyl alcohol-isopropyl ether system.

Compound (1)

Crystal data

$C_{18}H_{34}N_2^{2+}.2ClO_4^-$
M_r = 477.37
Orthorhombic
$P2_12_12_1$
a = 10.362 (2) Å
b = 14.463 (2) Å
c = 15.209 (2) Å
V = 2279.3 (6) Å^3
Z = 4
D_x = 1.391 Mg m^{-3}
D_m not measured
Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 39 reflections
θ = 6.87–15.89°
μ = 0.331 mm^{-1}
T = 293 (2) K
Prism
0.25 × 0.25 × 0.2 mm
Colourless

Data collection

KM-4 four-circle diffractometer
$\omega/2\theta$ scans
Absorption correction: none
2299 measured reflections
2299 independent reflections
1331 observed reflections [$I > 2\sigma(I)$]
θ_{max} = 25.06°
$h = 0 \rightarrow 12$
$k = 0 \rightarrow 17$
$l = 0 \rightarrow 18$
2 standard reflections monitored every 100 reflections
intensity decay: 1.5%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.0653
$wR(F^2)$ = 0.1837
S = 1.069
1966 reflections
279 parameters
H atoms: see text
$w = 1/[\sigma^2(F_o^2) + (0.1254P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.402
$\Delta\rho_{max}$ = 0.614 e Å^{-3}
$\Delta\rho_{min}$ = −0.219 e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)
Absolute configuration: Flack (1983)
Flack parameter = 0.17 (17)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å²) for (1)*

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

	x	y	z	U_{eq}
Cl1	−0.0059 (2)	−0.37715 (15)	0.39793 (11)	0.0747 (6)
O1	0.1083 (7)	−0.3912 (8)	0.3517 (5)	0.155 (4)
O2	0.0106 (7)	−0.3912 (4)	0.4900 (3)	0.097 (2)
O3	−0.1025 (7)	−0.4396 (4)	0.3665 (4)	0.097 (2)
O4	−0.0493 (7)	−0.2834 (4)	0.3827 (4)	0.108 (2)
Cl2	0.0961 (2)	0.14548 (12)	0.65992 (10)	0.0729 (6)
O5	0.0193 (9)	0.1264 (5)	0.5858 (4)	0.133 (3)
O6	0.0832 (11)	0.0802 (7)	0.7245 (6)	0.166 (4)
O7	0.2185 (11)	0.1434 (11)	0.6267 (7)	0.212 (6)
O8	0.0601 (16)	0.2299 (6)	0.6874 (6)	0.211 (6)
N1	0.1841 (6)	0.1004 (5)	0.4195 (4)	0.069 (2)
C2	0.2661 (12)	0.1848 (7)	0.4161 (7)	0.117 (4)
C3	0.1907 (13)	0.2713 (6)	0.4256 (7)	0.118 (4)
C4	0.0833 (13)	0.2780 (6)	0.3580 (6)	0.112 (3)
C5	0.0000 (11)	0.1923 (5)	0.3626 (5)	0.086 (2)
C6	0.0801 (8)	0.1044 (5)	0.3515 (4)	0.067 (2)
C7	0.0034 (7)	0.0154 (4)	0.3443 (3)	0.0540 (14)
C8	0.0926 (7)	−0.0666 (4)	0.3293 (4)	0.063 (2)
C9	0.1778 (6)	−0.0717 (5)	0.4103 (5)	0.065 (2)
C10	0.2626 (7)	0.0156 (6)	0.4160 (6)	0.081 (2)
C11	0.0993 (7)	−0.0854 (4)	0.4951 (4)	0.058 (2)
C12	0.1357 (7)	−0.1702 (5)	0.5457 (5)	0.076 (2)
C13	0.0582 (9)	−0.1808 (7)	0.6312 (5)	0.092 (3)
C14	−0.0816 (8)	−0.1799 (5)	0.6126 (5)	0.073 (2)
C15	−0.1186 (8)	−0.0953 (5)	0.5604 (5)	0.073 (2)
N16	−0.0442 (5)	−0.0871 (4)	0.4757 (4)	0.0513 (13)
C17	−0.0912 (6)	−0.0055 (4)	0.4211 (4)	0.0526 (14)
C18	−0.2310 (7)	−0.0194 (5)	0.3911 (5)	0.068 (2)
C19	−0.2891 (9)	0.0684 (5)	0.3533 (7)	0.091 (3)
C20	−0.2491 (9)	−0.0971 (5)	0.3262 (8)	0.102 (3)

Table 2. *Selected geometric parameters (Å, °) for (1)*

N1—C10	1.473 (11)	N16—C11	1.517 (9)
N1—C6	1.494 (10)	N16—C15	1.506 (8)
N1—C2	1.489 (10)	N16—C17	1.523 (8)
C10—N1—C6	113.9 (6)	C15—N16—C11	109.7 (5)
C10—N1—C2	111.5 (7)	C15—N16—C17	111.3 (5)
C6—N1—C2	110.8 (6)	C11—N16—C17	114.0 (5)

Table 3. *Hydrogen-bonding geometry (Å, °) for (1)*

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H11···O5	0.81 (6)	2.26 (6)	3.07 (1)	176 (5)
N16—H161···O4	0.80 (6)	2.38 (6)	3.173 (9)	174 (5)

Compound (2)

Crystal data

$C_{18}H_{31}N_2O^+.ClO_4^-.H_2O$
M_r = 408.91
Monoclinic
$P2_1$
a = 8.262 (2) Å
b = 13.774 (4) Å
c = 8.915 (2) Å
β = 92.49 (2)°
V = 1013.6 (4) Å³
Z = 2
D_x = 1.340 Mg m^{-3}
D_m not measured
Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 50 reflections
θ = 3.23–13.5°
μ = 0.225 mm^{-1}
T = 293 (2) K
Prism
0.6 × 0.4 × 0.3 mm
Colourless

Data collection

KM-4 four-circle diffractometer
$\omega/2\theta$ scans
Absorption correction: none
2006 measured reflections
1883 independent reflections
1447 observed reflections [$I > 2\sigma(I)$]
R_{int} = 0.0176
θ_{max} = 25.06°
$h = -9 \rightarrow 9$
$k = 0 \rightarrow 16$
$l = 0 \rightarrow 10$
2 standard reflections monitored every 100 reflections
intensity decay: 2%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.0357
$wR(F^2)$ = 0.1371
S = 1.125
1879 reflections
256 parameters
H atoms: see text
$w = 1/[\sigma^2(F_o^2) + (0.0625P)^2 + 0.4799P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.024
$\Delta\rho_{max}$ = 0.326 e Å^{-3}
$\Delta\rho_{min}$ = −0.368 e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)
Absolute configuration: Flack (1983)
Flack parameter = 0.01 (13)

Table 4. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å²) for (2)*

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

	x	y	z	U_{eq}
N1	1.1567 (5)	0.2387 (3)	0.6824 (4)	0.0413 (10)
C2	1.2186 (5)	0.1493 (4)	0.7098 (6)	0.0408 (11)
O2	1.2667 (5)	0.0998 (3)	0.6049 (4)	0.0563 (10)
C3	1.2334 (6)	0.1121 (4)	0.8674 (6)	0.0504 (13)
C4	1.2177 (7)	0.1902 (5)	0.9854 (6)	0.0526 (14)
C5	1.0684 (6)	0.2507 (4)	0.9440 (5)	0.0454 (12)
C6	1.0906 (5)	0.3039 (4)	0.7963 (5)	0.0402 (11)
C7	0.9346 (6)	0.3559 (3)	0.7362 (6)	0.0377 (11)
C8	0.9684 (7)	0.4057 (4)	0.5879 (6)	0.0481 (13)
C9	1.0111 (6)	0.3261 (4)	0.4785 (6)	0.0475 (13)
C10	1.1693 (6)	0.2804 (5)	0.5332 (7)	0.0523 (14)
C11	0.8816 (6)	0.2465 (4)	0.4678 (5)	0.0372 (11)
C12	0.8370 (7)	0.2203 (5)	0.3060 (6)	0.0546 (15)
C13	0.7065 (7)	0.1428 (5)	0.2923 (6)	0.0556 (14)
C14	0.5605 (7)	0.1778 (4)	0.3716 (6)	0.0486 (13)
C15	0.6037 (6)	0.2008 (4)	0.5330 (5)	0.0407 (11)
N16	0.7336 (4)	0.2762 (3)	0.5508 (4)	0.0317 (8)
C17	0.7796 (5)	0.2942 (3)	0.7168 (5)	0.0325 (9)
C18	0.6361 (6)	0.3354 (4)	0.7992 (6)	0.0453 (12)
C19	0.6665 (8)	0.3288 (5)	0.9690 (6)	0.0581 (15)
C20	0.5902 (9)	0.4393 (5)	0.7547 (8)	0.076 (2)
Cl1	0.76868 (15)	1.0	0.8502 (2)	0.0517 (4)
O11	0.8545 (6)	1.0311 (4)	0.7245 (6)	0.087 (2)
O12	0.6454 (6)	1.0698 (4)	0.8739 (6)	0.092 (2)
O13	0.6997 (7)	0.9075 (4)	0.8160 (7)	0.093 (2)
O14	0.8745 (6)	0.9938 (5)	0.9786 (6)	0.093 (2)
O1W	1.4020 (7)	−0.0766 (4)	0.6280 (6)	0.079 (2)

Table 5. *Selected geometric parameters (Å, °) for (2)*

N1—C2	1.351 (7)	N16—C11	1.513 (6)
N1—C10	1.457 (7)	N16—C15	1.497 (6)
N1—C6	1.478 (6)	N16—C17	1.532 (5)
C2—O2	1.236 (6)		
C2—N1—C10	118.9 (5)	C15—N16—C11	110.6 (4)
C2—N1—C6	125.3 (4)	C15—N16—C17	111.2 (3)
C10—N1—C6	115.5 (4)	C11—N16—C17	110.1 (3)

Table 6. *Hydrogen-bonding geometry (Å, °) for (2)*

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N16—H161···O1*W*^{i}	0.86 (5)	1.96 (5)	2.784 (6)	162 (4)
O1*W*—H1*W*···O13^{ii}	0.87 (7)	2.06 (7)	2.923 (7)	171 (6)
O1*W*—H1*W*···O2	0.93 (8)	1.78 (8)	2.680 (7)	161 (6)

Symmetry codes: (i) $2-x, \frac{1}{2}+y, 1-z$; (ii) $1+x, y-1, z$.

The rather poor crystal quality caused high e.s.d.'s in all parameters; reflections with net negative measured intensities were suppressed in the refinement. H atoms were refined with a riding model and with U_{iso}(H) set at 1.2 (1.5 for methyl groups) times U_{eq} of the bonded atom; the positions of the N—H and H_2O H atoms were freely refined.

For both compounds, data collection: *KM-4 Software* (Kuma Diffraction, 1992); cell refinement: *KM-4 Software*; data reduction: *KM-4 Software*; program(s) used to solve structures: *SHELXS86* (Sheldrick, 1990); program(s) used to refine structures: *SHELXL93* (Sheldrick, 1993); molecular graphics: *Stereochemical Workstation* (Siemens, 1989); software used to prepare material for publication: *SHELXL93*.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: CF1098). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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A Triphenylboroxin Derivative Possessing Two Intramolecular Chelates

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Abstract

Condensation of 2-formylbenzeneboronic acid and 1,1-dimethylhydrazine afforded the title compound, bis-(8-*B*-4)-1,3,5-tris{2-[(*N*,*N*-dimethylhydrazono)methyl]-phenyl)}boroxin, $C_{27}H_{33}B_3N_6O_3$, a tridehydro cyclic trimer of the expected simple benzaldehyde hydrazone and the first triphenylboroxin derivative found to possess two B chelating interactions. The double chelation induces molecular asymmetry but effects only a slight puckering in the central boroxin ring.

Comment

In an investigation parallel to that of our study of the physicochemical properties of 2,4,1-benzoxaza- and benzodiazaborines, we are undertaking a similar examination of their 2,3,1-isomeric counterparts in order to delineate the similarities and differences between these two classes of structurally related heterocycles. It has been known for quite some time that the condensation of 2-formylbenzeneboronic acid (1) with hydroxylamine and hydrazine-based carbonyl derivatizing reagents can be used to prepare bicyclic 2,3,1-benzoxaza- and benzodiazaborines, respectively (Tschampel & Snyder, 1964; Dewar & Dougherty, 1964). One potential member of this latter class of heterocycles is the previously unknown *N*,*N*-dimethylhydrazone, (2), a compound we view with some interest because in an internally chelated, zwitterionic structural form it would bear some resemblance to (4), a bis-methanol adduct of 1,2-dihydro-1-hydroxy-2,4,1-benzodiazaborine we examined recently by X-ray crystallographic and other means (Groziak, Ganguly & Robinson, 1994). We now relate that (2) obtained *via* the condensation of (1) and 1,1-dimethylhydrazine exists in tridehydro trimeric form as the substituted triphenylboroxin (3) in the solid state. Compound (2) can be obtained *via* gentle hydrolysis of (3) effected by simple dissolution in water at 298 K, but it deboronates in this medium at temperatures near 373 K. Evaporation of an aqueous solution of (2) results in the regeneration of (3).