Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.0568$
$wR(F^2) = 0.1148$
$S = 1.132$
2824 reflections
398 parameters
H atoms refined as riding model (except for H1)
$w = 1/[\sigma^2(F_o^2) + (0.0580P)^2 + 0.7204P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.004$
$\Delta\rho_{\max} = 0.211$ e Å^{-3}
$\Delta\rho_{\min} = -0.176$ e Å^{-3}
Extinction correction: *SHELXL*93 (Sheldrick, 1993)
Extinction coefficient: 0.0047 (12)
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

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

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

	x	y	z	$U_{\rm eq}$
Si1	0.1246 (2)	0.78695 (15)	0.20606 (11)	0.0546 (7)
O1	−0.0369 (6)	0.7720 (4)	0.0152 (2)	0.065 (2)
O2	0.1262 (5)	0.6910 (3)	0.1658 (2)	0.0525 (14)
O3	0.1054 (7)	0.4455 (4)	0.0132 (3)	0.100 (3)
O4	0.2803 (6)	0.4099 (3)	0.0725 (2)	0.063 (2)
N1	0.0914 (7)	0.7677 (5)	0.0154 (3)	0.055 (2)
N2	0.1858 (7)	0.5468 (4)	0.0802 (3)	0.059 (2)
C1	0.1579 (10)	0.6998 (7)	0.0374 (4)	0.057 (2)
C2	0.0907 (8)	0.6208 (5)	0.0685 (4)	0.049 (2)
C3	0.0258 (8)	0.6509 (5)	0.1274 (4)	0.052 (2)
C4	−0.0431 (11)	0.5714 (7)	0.1594 (5)	0.075 (3)
C5	0.1614 (9)	0.8486 (6)	−0.0107 (4)	0.067 (3)
C12	0.1836 (9)	0.4668 (6)	0.0505 (4)	0.059 (3)
C13	0.3025 (10)	0.3178 (6)	0.0456 (4)	0.065 (3)
C14	0.3316 (21)	0.3272 (12)	−0.0206 (5)	0.140 (6)
C15	0.4256 (9)	0.2841 (6)	0.0796 (5)	0.083 (3)
C16	0.1822 (12)	0.2566 (8)	0.0570 (9)	0.122 (5)
C17	0.1880 (9)	0.7518 (6)	0.2820 (4)	0.060 (2)
C18	0.3295 (10)	0.7140 (9)	0.2736 (5)	0.094 (3)
C19	0.1073 (14)	0.6734 (7)	0.3090 (5)	0.092 (3)
C20	0.1950 (13)	0.8346 (8)	0.3262 (4)	0.093 (3)
C6	0.1243 (8)	0.8702 (6)	−0.0730 (4)	0.052 (2)
C7	0.0612 (9)	0.9496 (7)	−0.0878 (6)	0.074 (3)
C8	0.0371 (11)	0.9726 (8)	−0.1475 (7)	0.090 (3)
C9	0.0711 (11)	0.9128 (10)	−0.1930 (6)	0.087 (3)
C10	0.1299 (11)	0.8309 (9)	−0.1781 (5)	0.086 (3)
C11	0.1576 (9)	0.8087 (6)	−0.1174 (5)	0.068 (3)
C21	0.2363 (9)	0.8712 (6)	0.1683 (4)	0.056 (2)
C22	0.3434 (9)	0.8423 (7)	0.1335 (4)	0.063 (2)
C23	0.4251 (10)	0.9048 (9)	0.1038 (5)	0.078 (3)
C24	0.4053 (12)	0.9988 (9)	0.1094 (5)	0.089 (3)
C25	0.3016 (13)	1.0286 (8)	0.1455 (5)	0.096 (4)
C26	0.2204 (11)	0.9667 (6)	0.1746 (4)	0.074 (3)
C27	−0.0437 (8)	0.8425 (6)	0.2073 (5)	0.058 (2)
C28	−0.0941 (9)	0.8828 (6)	0.1557 (5)	0.065 (3)
C29	−0.2168 (11)	0.9262 (6)	0.1530 (6)	0.079 (3)
C30	−0.2891 (11)	0.9334 (7)	0.2036 (7)	0.090 (3)
C31	−0.2455 (12)	0.8969 (8)	0.2558 (7)	0.100 (4)
C32	−0.1233 (11)	0.8504 (7)	0.2581 (5)	0.078 (3)

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

O1—N1	1.285 (8)	C1—C2	1.480 (11)
O2—C3	1.431 (8)	C1—H1	0.86 (6)
N1—C1	1.271 (10)	C2—C3	1.510 (10)
N1—C5	1.467 (9)	C3—C4	1.503 (10)
C1—N1—O1	124.1 (8)	N1—C1—H1	125 (4)
C1—N1—C5	120.0 (7)	C2—C1—H1	113 (4)
O1—N1—C5	115.9 (7)	N2—C2—C1	109.9 (7)
N1—C1—C2	121.3 (8)	C1—C2—C3	112.0 (7)
C2—C1—N1—C5	175.0 (7)	H1—C1—N1—O1	−175 (5)

Data collection: *XSCANS* (Siemens, 1992). Cell refinement: *XSCANS*. Data reduction: *XSCANS*. Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *SHELXL*93. Software used to prepare material for publication: *SHELXL*93.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: NA1167). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Two Products of Ring Opening Reactions of an Arylthionitrooxirane

WILLIAM CLEGG,* JOANNA M. DUNBAR,
MARK R. J. ELSEGOOD, RICHARD F. W. JACKSON AND
NICHOLAS J. PALMER

Department of Chemistry, University of Newcastle upon Tyne, Newcastle upon Tyne NE1 7RU, England

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Abstract

Crystal structure determination of the products, *S*-(4-methylphenyl) (αR,4R)-α-bromo-2,2-dimethyl-1,3-dioxolane-4-thioacetate, $C_{14}H_{17}BrO_3S$, (2), and *S*-(4-methylphenyl) (αR,4R)-α-(benzyloxycarbonylamino)-2,2-dimethyl-1,3-dioxolane-4-thioacetate, $C_{22}H_{25}NO_5S$, (3), of the two reactions, including absolute-structure assignment, shows that they proceed stereospecifically with inversion of configuration. Common groups of the two structures have essentially the same bond lengths and angles, but different conformations, arising from weak hydrogen bonding and van der Waals interactions.

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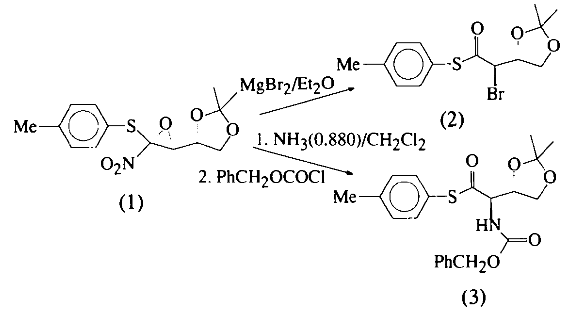
Comment

The title compounds, (2) and (3), were synthesized as part of an investigation into methods of synthesizing β-hydroxy-α-amino acid derivatives *via* oxirane intermediates (Jackson, Palmer, Wythes, Clegg & Elsegood, 1995; Ashwell, Jackson & Kirk, 1990). Both compounds were prepared from the arylthionitrooxirane (1), which has been structurally characterized by X-ray diffraction, establishing its *syn* configuration and enantiomeric purity (Jackson *et al.*, 1995). The results show that both ring-opening reactions proceed stereospecifically with inversion of configuration, and confirm the suitability of this type of reaction for the facile synthesis of diastereoisomerically pure, fully protected γ-hydroxythreonine derivatives such as (3).



Each structure is of an optically pure isomer, with the same absolute stereochemistry for both molecules. There are no major differences in the bond lengths and angles for the common portions of the molecular structures, the largest difference being in the C1—C2—C3 angle at the C atom bearing the two different substituents. The conformations about the S—C and C—C bonds in the C—S—C—C—C chain are quite different in the two structures, but the two dioxolane rings are both envelopes with O2 as the flap atom.

The N—H group in structure (3) is suitably oriented for hydrogen-bond formation with the C1=O1 carbonyl group in the neighbouring molecule, with N—H···O and C—O···H angles of 155 (6) and 128 (2)°, respectively; however, this hydrogen bond is not very strong, the O···H and O···N separations being 2.26 (6) and 2.989 (14) Å, respectively (these geometric parameters are based on a free refinement of the N—H H atom). With only translational symmetry present in the space group $P1$, these weak hydrogen bonds link the molecules to form chains parallel to the a axis. There are no particularly short intermolecular contacts in the structure of (2).

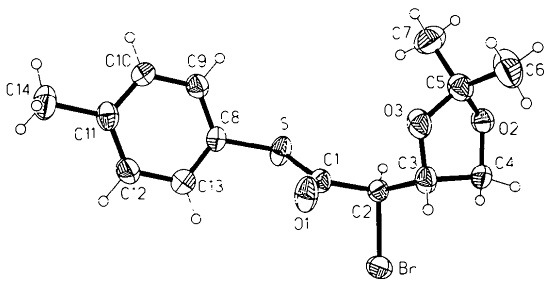


Fig. 1. Structure of compound (2) with 40% probability ellipsoids.

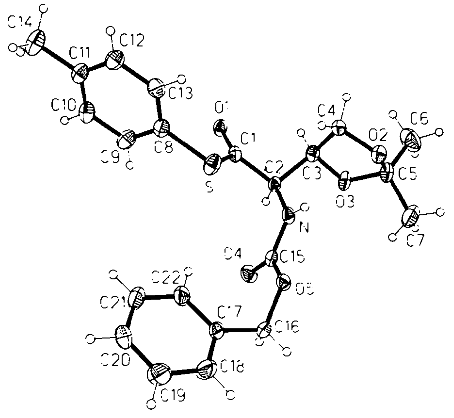


Fig. 2. Structure of compound (3) with 40% probability ellipsoids.

Experimental

The syntheses of the two title compounds have been reported (Jackson, Palmer, Wythes, Clegg & Elsegood, 1995). Compound (2) was recrystallized by slow cooling from petrol (40–60° fraction). Compound (3) was recrystallized by diffusion of pentane into a solution in ethyl acetate.

Compound (2)

Crystal data

$C_{14}H_{17}BrO_3S$
$M_r = 345.25$
Orthorhombic
$P2_12_12_1$
$a = 6.8923\,(11)$ Å
$b = 12.836\,(2)$ Å
$c = 17.387\,(3)$ Å
$V = 1538.2\,(4)$ Å^3
$Z = 4$
$D_x = 1.491$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 32 reflections
$\theta = 11.11$–$12.14°$
$\mu = 2.809$ mm^{-1}
$T = 240\,(2)$ K
Thick plate
$0.52 \times 0.48 \times 0.20$ mm
Colourless

Data collection

Stoe Siemens diffractometer
ω/θ scans with on-line profile fitting (Clegg, 1981)
Absorption correction: semi-empirical using azimuthal scans applied with *SHELXTL* (Sheldrick, 1990)
$T_{min} = 0.172$, $T_{max} = 0.328$
4066 measured reflections

2698 independent reflections
2119 observed reflections $[I > 2\sigma(I)]$
$R_{int} = 0.0476$
$\theta_{max} = 24.98°$
$h = -8 \rightarrow 8$
$k = -15 \rightarrow 15$
$l = -20 \rightarrow 20$
3 standard reflections frequency: 60 min intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.0372$
$wR(F^2) = 0.1059$
$S = 1.071$
2697 reflections
176 parameters
$w = 1/[\sigma^2(F_o^2) + (0.0620P)^2 + 0.2482P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.110$
$\Delta\rho_{\max} = 0.307$ e Å^{-3}
$\Delta\rho_{\min} = -0.500$ e Å^{-3}

Extinction correction: *SHELXL*93 (Sheldrick, 1993)
Extinction coefficient: 0.0027 (10)
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)
Absolute configuration: $x = -0.001$ (13) (Flack, 1983)

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

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

	x	y	z	U_{eq}
Br	1.04505 (8)	0.61449 (4)	0.23332 (3)	0.0586 (2)
S	0.5406 (2)	0.51613 (9)	0.28630 (7)	0.0530 (3)
O1	0.8270 (6)	0.5135 (3)	0.3883 (2)	0.0604 (10)
O2	0.7538 (5)	0.8892 (3)	0.3352 (2)	0.0479 (8)
O3	0.7570 (6)	0.7488 (2)	0.4152 (2)	0.0528 (9)
C1	0.7510 (7)	0.5566 (3)	0.3347 (3)	0.0381 (10)
C2	0.8291 (6)	0.6575 (3)	0.2991 (3)	0.0380 (10)
C3	0.9055 (7)	0.7327 (3)	0.3598 (3)	0.0470 (12)
C4	0.9385 (7)	0.8423 (3)	0.3262 (3)	0.0476 (11)
C5	0.6781 (9)	0.8513 (4)	0.4063 (3)	0.0538 (13)
C6	0.7424 (13)	0.9181 (5)	0.4730 (4)	0.084 (2)
C7	0.4610 (10)	0.8426 (5)	0.3982 (4)	0.079 (2)
C8	0.4658 (7)	0.4029 (3)	0.3377 (2)	0.0385 (9)
C9	0.2843 (8)	0.4036 (4)	0.3706 (3)	0.0457 (12)
C10	0.2141 (8)	0.3144 (4)	0.4064 (3)	0.0478 (12)
C11	0.3262 (8)	0.2251 (3)	0.4106 (3)	0.0460 (12)
C12	0.5064 (8)	0.2253 (3)	0.3771 (3)	0.0515 (13)
C13	0.5781 (8)	0.3143 (3)	0.3399 (3)	0.0508 (13)
C14	0.2463 (10)	0.1283 (4)	0.4500 (3)	0.067 (2)

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

Br—C2	1.956 (5)	O3—C5	1.432 (6)
S—C1	1.755 (5)	C1—C2	1.533 (6)
S—C8	1.783 (4)	C2—C3	1.525 (6)
O1—C1	1.203 (6)	C3—C4	1.540 (6)
O2—C4	1.417 (6)	C5—C7	1.507 (9)
O2—C5	1.427 (6)	C5—C6	1.509 (9)
O3—C3	1.420 (6)		
C1—S—C8	103.9 (2)	O3—C3—C4	103.3 (4)
C4—O2—C5	106.2 (4)	C2—C3—C4	111.5 (4)
C3—O3—C5	109.5 (4)	O2—C4—C3	102.4 (4)
O1—C1—C2	123.3 (4)	O2—C5—O3	105.5 (4)
O1—C1—S	126.5 (3)	O2—C5—C7	108.0 (5)
C2—C1—S	110.2 (3)	O3—C5—C7	108.6 (5)
C3—C2—C1	112.0 (4)	O2—C5—C6	111.4 (5)
C3—C2—Br	108.7 (3)	O3—C5—C6	109.1 (5)
C1—C2—Br	105.4 (3)	C7—C5—C6	113.9 (6)
O3—C3—C2	108.2 (4)		
C8—S—C1—O1	2.6 (5)	C5—O2—C4—C3	36.5 (5)
C8—S—C1—C2	−177.5 (3)	O3—C3—C4—O2	−28.8 (5)
O1—C1—C2—C3	−39.1 (6)	C2—C3—C4—O2	87.2 (5)
S—C1—C2—C3	141.0 (3)	C4—O2—C5—O3	−30.8 (5)
C5—O3—C3—C2	−107.6 (4)	C3—O3—C5—O2	11.3 (5)
C5—O3—C3—C4	10.7 (5)	C1—S—C8—C9	120.7 (4)
C1—C2—C3—O3	−53.6 (5)	C1—S—C8—C13	−64.3 (4)
C1—C2—C3—C4	−166.6 (4)		

Compound (3)

Crystal data

$C_{22}H_{25}NO_5S$
$M_r = 415.49$
Triclinic
$P1$
$a = 5.584$ (2) Å
$b = 9.042$ (3) Å
$c = 10.902$ (3) Å
$\alpha = 77.06$ (3)°
$\beta = 78.75$ (2)°
$\gamma = 87.98$ (3)°
$V = 526.1$ (3) Å^3
$Z = 1$
$D_x = 1.311$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 35 reflections
$\theta = 10.15$–12.50°
$\mu = 0.187$ mm^{-1}
$T = 160$ (2) K
Needle
0.44 × 0.14 × 0.08 mm
Colourless

Data collection

Stoe Siemens diffractometer
ω/θ scans with on-line profile fitting (Clegg, 1981)
Absorption correction: none
1606 measured reflections
1606 independent reflections

1492 observed reflections $[I > 2\sigma(I)]$
$\theta_{\max} = 22.53°$
$h = -5 \rightarrow 6$
$k = -9 \rightarrow 9$
$l = -8 \rightarrow 11$
5 standard reflections
frequency: 60 min
intensity decay: 6%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.0431$
$wR(F^2) = 0.1246$
$S = 1.121$
1606 reflections
268 parameters
$w = 1/[\sigma^2(F_o^2) + (0.0647P)^2 + 0.3010P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$

$\Delta\rho_{\max} = 0.226$ e Å^{-3}
$\Delta\rho_{\min} = -0.240$ 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: $x = -0.03$ (14) (Flack, 1983)

Table 3. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2) *for* (3)

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

	x	y	z	U_{eq}
S	0.1784 (2)	0.96466 (15)	0.03797 (12)	0.0370 (4)
O1	−0.2412 (6)	0.8254 (4)	0.1579 (3)	0.0294 (9)
O2	0.3720 (6)	0.5569 (4)	0.3988 (4)	0.0329 (9)
O3	0.0338 (6)	0.6623 (4)	0.4958 (3)	0.0334 (9)
O4	0.1314 (6)	1.1347 (4)	0.3364 (4)	0.0351 (9)
O5	0.5351 (6)	1.0932 (4)	0.2724 (4)	0.0293 (8)
N	0.2700 (8)	0.9094 (5)	0.2909 (4)	0.0259 (10)
C1	−0.0458 (9)	0.8688 (6)	0.1688 (5)	0.0264 (12)
C2	0.0328 (8)	0.8483 (6)	0.2982 (5)	0.0256 (11)
C3	0.0082 (9)	0.6776 (6)	0.3648 (5)	0.0286 (12)
C4	0.2104 (9)	0.5755 (6)	0.3110 (6)	0.0318 (12)
C5	0.2241 (10)	0.5564 (6)	0.5202 (5)	0.0338 (13)
C6	0.1112 (16)	0.4008 (8)	0.5857 (8)	0.066 (2)
C7	0.3751 (12)	0.6178 (9)	0.5997 (7)	0.054 (2)
C8	0.0178 (9)	0.9875 (6)	−0.0911 (5)	0.0309 (13)
C9	−0.1812 (10)	1.0839 (7)	−0.0956 (6)	0.0389 (14)
C10	−0.2989 (10)	1.1047 (7)	−0.2000 (6)	0.0393 (14)

C11	−0.2214 (10)	1.0318 (7)	−0.2976 (5)	0.0341 (13)
C12	−0.0221 (10)	0.9369 (7)	−0.2927 (6)	0.0369 (14)
C13	0.0969 (10)	0.9147 (6)	−0.1894 (6)	0.0346 (13)
C14	−0.3474 (11)	1.0573 (9)	−0.4128 (6)	0.050 (2)
C15	0.2966 (8)	1.0527 (6)	0.3031 (5)	0.0249 (12)
C16	0.5894 (9)	1.2495 (6)	0.2720 (5)	0.0304 (12)
C17	0.5988 (9)	1.3531 (6)	0.1416 (6)	0.0310 (13)
C18	0.7802 (12)	1.4614 (8)	0.0971 (7)	0.050 (2)
C19	0.7933 (15)	1.5640 (9)	−0.0208 (7)	0.064 (2)
C20	0.6258 (13)	1.5558 (7)	−0.0965 (7)	0.050 (2)
C21	0.4426 (12)	1.4465 (8)	−0.0515 (7)	0.050 (2)
C22	0.4281 (11)	1.3468 (7)	0.0665 (7)	0.043 (2)

Table 4. *Selected geometric parameters* (Å, °) *for* (3)

S—C8	1.782 (6)	O5—C16	1.455 (7)
S—C1	1.783 (5)	N—C15	1.348 (7)
O1—C1	1.208 (6)	N—C2	1.434 (7)
O2—C5	1.416 (7)	N—H1	0.79 (6)
O2—C4	1.419 (7)	C1—C2	1.528 (7)
O3—C5	1.429 (7)	C2—C3	1.549 (7)
O3—C3	1.438 (7)	C3—C4	1.544 (7)
O4—C15	1.214 (6)	C5—C7	1.518 (9)
O5—C15	1.351 (6)	C5—C6	1.524 (9)
C8—S—C1	101.1 (2)	O3—C3—C4	104.0 (4)
C5—O2—C4	106.0 (4)	O3—C3—C2	108.0 (4)
C5—O3—C3	108.3 (4)	C4—C3—C2	115.1 (4)
C15—O5—C16	116.1 (4)	O2—C4—C3	103.4 (4)
C15—N—C2	120.9 (4)	O2—C5—O3	105.7 (4)
C2—N—H1	119 (4)	O2—C5—C7	108.3 (5)
C15—N—H1	120 (4)	O3—C5—C7	107.7 (5)
O1—C1—C2	122.9 (5)	O2—C5—C6	112.3 (5)
O1—C1—S	124.2 (4)	O3—C5—C6	109.1 (5)
C2—C1—S	112.9 (4)	C7—C5—C6	113.5 (6)
N—C2—C1	114.3 (4)	O4—C15—N	125.4 (5)
N—C2—C3	112.8 (4)	O4—C15—O5	124.4 (5)
C1—C2—C3	108.1 (4)	N—C15—O5	110.2 (4)
C8—S—C1—O1	5.4 (5)	C5—O2—C4—C3	33.4 (5)
C8—S—C1—C2	−173.8 (3)	O3—C3—C4—O2	−18.8 (5)
O1—C1—C2—C3	54.4 (6)	C2—C3—C4—O2	99.2 (5)
S—C1—C2—C3	−126.3 (4)	C4—O2—C5—O3	−36.0 (5)
C5—O3—C3—C4	−2.4 (5)	C3—O3—C5—O2	23.1 (5)
C5—O3—C3—C2	−125.2 (4)	C1—S—C8—C13	−114.9 (4)
C1—C2—C3—O3	−168.5 (4)	C1—S—C8—C9	67.8 (5)
C1—C2—C3—C4	75.8 (5)		

The measured data set for (2) consisted of an octant, a complete set of Friedel opposites and a further octant equivalent to the first, thus producing the overall index limits given. For (3), the measured data set consisted of a complete sphere of reflections to the specified angle limit.

Isotropic H atoms were refined with a riding model for both compounds, including a parameter for rotation of each methyl group about the C—C bond.

For both compounds, data collection: *DIF*4 (Stoe & Cie, 1990); cell refinement: *DIF*4; data reduction: local programs; program(s) used to solve structures: *SHELXTL* (Sheldrick, 1990, 1994); program(s) used to refine structures: *SHELXL*93 (Sheldrick, 1993); molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXL*93 and local programs.

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Lists of structure factors, anisotropic displacement parameters and H-atom coordinates have been deposited with the IUCr (Reference: BM1008). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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p-(4,6-Diamino-*s*-triazin-2-yl)aminophenylarsonic Acid Dihydrate

GORDON A. LEONARD AND WILLIAM N. HUNTER*

Department of Chemistry, University of Manchester, Manchester M13 9PL, England

BRADLEY J. BERGER† AND ALAN H. FAIRLAMB

Department of Medical Parasitology, London School of Hygiene and Tropical Medicine, Keppel Street, London WC1E 7HT, England

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Abstract

The structure of the title compound, $C_9H_{11}AsN_6O_3.2H_2O$, contains a planar organic moiety bonded to pentavalent arsenic. The lattice is stabilized by a combination of aromatic stacking interactions and by a hydrogen-bonding network involving water molecules of crystallization.

Comment

Melarsamine hydrochloride (trade name Cymelarsen) is a water-soluble trivalent arsenical drug which finds use in the treatment of trypanosomal infection in some animals (Zweygarth & Kaminsky, 1990). A study of the properties of the drug in aerobic aqueous solution indicates that the compound is unstable, with a complex mode of dissociation (Berger & Fairlamb, 1994). This analysis was undertaken to identify the structure of a

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† Present address: The Picower Institute for Medical Research, Manhasset, New York, NY 11030, USA.