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(1*RS*,4*RS*)-1-Methoxyspiro[bicyclo[2.2.2]oct-5-ene-2,2′-[1′,3′]dithiolane]. Corrigendum

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In the paper by Gültekin, Adams & Hökelek [*Acta Cryst.* (2003), E**59**, o926–o928], the placement of H atoms bonded to C3, C4, C10 and C11 is wrong. C10═C11 is a double bond, but there are two H atoms on each C atom instead of one. On the other hand, C3—C4 is a single bond and there is one H atom on each C atom instead of two. The structure has now been rerefined with the correct assignment of H atoms and the structure is shown in Fig. 1.

Experimental

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.058$
$wR(F^2) = 0.144$
$S = 1.02$
1983 reflections
133 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0805P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.30$ e Å^{-3}
$\Delta\rho_{min} = -0.22$ e Å^{-3}

Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.007 Å
R factor = 0.058
wR factor = 0.144
Data-to-parameter ratio = 14.9

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Table 1
Selected geometric parameters (Å, °).

S1—C7	1.802 (5)	C2—C1	1.558 (5)
S1—C2	1.823 (4)	C2—C6	1.566 (5)
S2—C8	1.774 (7)	C4—C3	1.511 (7)
S2—C2	1.844 (4)	C4—C5	1.529 (7)
O1—C1	1.416 (4)	C5—C6	1.536 (8)
O1—C9	1.420 (5)	C7—C8	1.467 (9)
C1—C10	1.506 (6)	C11—C10	1.332 (7)
C1—C3	1.516 (6)	C11—C5	1.502 (7)
C7—S1—C2	97.3 (2)	C1—C2—S2	110.7 (3)
C8—S2—C2	99.3 (2)	C6—C2—S2	110.4 (3)
C1—O1—C9	116.2 (3)	S1—C2—S2	106.1 (2)
O1—C1—C10	114.9 (3)	C4—C3—C1	112.1 (3)
O1—C1—C3	112.7 (3)	C4—C5—C6	106.0 (4)
O1—C1—C2	106.6 (3)	C8—C7—S1	109.5 (4)
C3—C1—C2	107.1 (3)	C7—C8—S2	114.0 (4)
C1—C2—S1	113.8 (2)	C11—C10—C1	114.7 (4)
C6—C2—S1	108.4 (3)	C10—C11—C5	114.0 (4)
C7—S1—C2—S2	30.0 (3)	C9—O1—C1—C3	−70.9 (5)
C2—S1—C7—C8	−37.9 (5)	S1—C2—C1—O1	56.8 (4)
C2—S2—C8—C7	−9.2 (5)	S2—C2—C1—O1	−62.5 (3)
C8—S2—C2—S1	−15.6 (3)	S1—C7—C8—S2	31.2 (6)

Atoms H10 and H11 were located in a difference map and refined isotropically [C—H = 0.96 (2)–0.99 (2) Å]. The other H atoms were positioned geometrically, with C—H = 0.96, 0.97 and 0.98 Å for methyl, methylene and methine H atoms, respectively, and constrained to ride on their parent atoms, with U_{iso}(H) = xU_{eq}(carrier atom), where x = 1.5 for methyl H atoms and x = 1.2 for all others.

Data collection: *XSCANS* (Siemens, 1996); cell refinement: *XSCANS*; data reduction: *SHELXTL* (Bruker, 1997); program(s)

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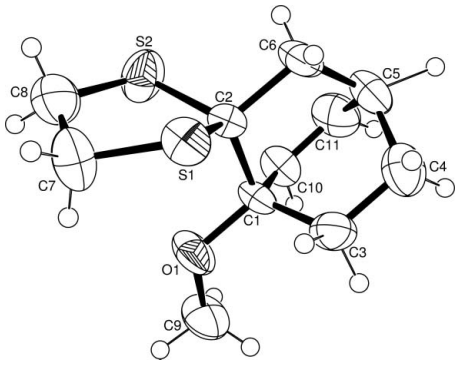

Figure 1
A view of the molecular structure, with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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supporting information

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(1*RS*,4*RS*)-1-Methoxyspiro[bicyclo[2.2.2]oct-5-ene-2,2′-[1′,3′]dithiolane]. Corrigendum

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(1RS,4RS)-1-Methoxyspiro[bicyclo[2.2.2]oct-5-ene-2,2′-[1′,3′]dithiolane]

Crystal data

$C_{11}H_{16}OS_2$
$M_r = 228.36$
Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a = 6.748$ (4) Å
$b = 7.870$ (4) Å
$c = 11.474$ (7) Å
$\alpha = 99.23$ (4)°
$\beta = 103.00$ (5)°
$\gamma = 102.69$ (3)°
$V = 564.9$ (6) Å^3
$Z = 2$
$F(000) = 244$
$D_x = 1.342$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 12$–20°
$\mu = 0.44$ mm^{-1}
$T = 293$ K
Block, colorless
0.55 × 0.34 × 0.28 mm

Data collection

Siemens P4 diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
non–profiled ω scans
2493 measured reflections
1983 independent reflections
1040 reflections with $I > 2\sigma(I)$
$R_{int} = 0.037$
$\theta_{max} = 25.0°$, $\theta_{min} = 2.7°$
$h = -1 \rightarrow 8$
$k = -9 \rightarrow 9$
$l = -13 \rightarrow 13$
3 standard reflections every 100 reflections
intensity decay: 1%

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.058$
$wR(F^2) = 0.144$
$S = 1.02$
1983 reflections
133 parameters
6 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0805P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.30$ e Å^{-3}
$\Delta\rho_{min} = -0.22$ e Å^{-3}

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit *S* are based on F^2, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on *F*, and *R*- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
S1	0.14528 (18)	0.75241 (15)	0.10473 (10)	0.0530 (4)
S2	−0.0295 (2)	1.01482 (16)	0.22939 (14)	0.0668 (4)
O1	0.2275 (5)	0.8109 (5)	0.3843 (3)	0.0615 (9)
C1	0.0184 (6)	0.7119 (5)	0.3203 (3)	0.0403 (9)
C2	−0.0367 (6)	0.7759 (5)	0.1983 (3)	0.0389 (9)
C3	−0.0041 (7)	0.5130 (6)	0.2860 (4)	0.0540 (11)
C4	−0.2285 (8)	0.4089 (7)	0.2191 (5)	0.0670 (14)
C5	−0.3620 (7)	0.5409 (6)	0.1991 (4)	0.0589 (12)
C6	−0.2629 (7)	0.6603 (6)	0.1225 (4)	0.0571 (12)
C7	0.3242 (9)	0.9721 (7)	0.1652 (6)	0.0780 (16)
C8	0.2042 (9)	1.1024 (7)	0.1882 (6)	0.0796 (15)
C9	0.3010 (8)	0.7912 (8)	0.5062 (4)	0.0679 (14)
C10	−0.1471 (7)	0.7445 (6)	0.3834 (4)	0.0524 (11)
C11	−0.3444 (8)	0.6569 (8)	0.3204 (5)	0.0657 (14)
H3A	0.0876	0.4917	0.2343	0.065*
H3B	0.0409	0.4707	0.3599	0.065*
H4A	−0.2839	0.3288	0.2671	0.080*
H4B	−0.2326	0.3380	0.1408	0.080*
H5	−0.5095	0.4782	0.1566	0.071*
H6A	−0.3509	0.7379	0.0987	0.069*
H6B	−0.2542	0.5865	0.0484	0.069*
H7A	0.4240	0.9733	0.2410	0.094*
H7B	0.4027	1.0033	0.1071	0.094*
H8A	0.1669	1.1482	0.1150	0.096*
H8B	0.2940	1.2020	0.2535	0.096*
H9A	0.4438	0.8629	0.5409	0.102*
H9B	0.2957	0.6681	0.5055	0.102*
H9C	0.2132	0.8293	0.5546	0.102*
H10	−0.127 (8)	0.869 (4)	0.416 (5)	0.097 (19)*
H11	−0.438 (8)	0.613 (10)	0.371 (5)	0.14 (3)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0519 (7)	0.0612 (7)	0.0462 (6)	0.0142 (5)	0.0169 (5)	0.0086 (5)
S2	0.074	0.0470 (7)	0.0913 (10)	0.0230 (5)	0.0366 (6)	0.0178 (6)

O1	0.0422 (18)	0.084 (2)	0.0387 (15)	−0.0088 (15)	−0.0036 (13)	0.0145 (15)
C1	0.0234 (18)	0.053 (2)	0.0344 (19)	0.0040 (15)	−0.0018 (15)	0.0045 (16)
C2	0.032 (2)	0.0438 (19)	0.0374 (18)	0.0113 (16)	0.0040 (15)	0.0055 (16)
C3	0.044 (3)	0.066 (3)	0.055 (2)	0.023 (2)	0.009 (2)	0.020 (2)
C4	0.072 (3)	0.054 (3)	0.060 (3)	0.002 (2)	0.012 (2)	−0.002 (2)
C5	0.035 (2)	0.070 (3)	0.053 (2)	−0.0014 (19)	−0.0009 (19)	0.001 (2)
C6	0.038 (2)	0.056 (2)	0.056 (3)	0.0066 (19)	−0.016 (2)	0.003 (2)
C7	0.061 (3)	0.068 (3)	0.095 (4)	−0.004 (3)	0.029 (3)	0.008 (3)
C8	0.071	0.058 (3)	0.094 (4)	0.009 (2)	0.000 (3)	0.014 (3)
C9	0.058 (3)	0.093 (4)	0.035 (2)	0.000 (3)	−0.004 (2)	0.015 (2)
C10	0.044 (2)	0.053 (2)	0.054 (2)	0.0088 (19)	0.012 (2)	0.002 (2)
C11	0.045 (3)	0.089 (4)	0.068 (3)	0.021 (2)	0.022 (2)	0.018 (3)

Geometric parameters (Å, º)

S1—C7	1.802 (5)	C5—C6	1.536 (8)
S1—C2	1.823 (4)	C5—H5	0.9800
S2—C8	1.774 (7)	C6—H6A	0.9700
S2—C2	1.844 (4)	C6—H6B	0.9700
O1—C1	1.416 (4)	C7—C8	1.467 (9)
O1—C9	1.420 (5)	C7—H7A	0.9700
C1—C10	1.506 (6)	C7—H7B	0.9700
C1—C3	1.516 (6)	C8—H8A	0.9700
C2—C1	1.558 (5)	C8—H8B	0.9700
C2—C6	1.566 (5)	C9—H9A	0.9600
C3—H3A	0.9700	C9—H9B	0.9600
C3—H3B	0.9700	C9—H9C	0.9600
C4—C3	1.511 (7)	C10—H10	0.96 (2)
C4—C5	1.529 (7)	C11—C10	1.332 (7)
C4—H4A	0.9700	C11—C5	1.502 (7)
C4—H4B	0.9700	C11—H11	0.99 (2)
C7—S1—C2	97.3 (2)	C6—C5—H5	111.1
C8—S2—C2	99.3 (2)	C5—C6—C2	110.2 (4)
C1—O1—C9	116.2 (3)	C5—C6—H6A	109.6
O1—C1—C10	114.9 (3)	C2—C6—H6A	109.6
O1—C1—C3	112.7 (3)	C5—C6—H6B	109.6
C10—C1—C3	109.7 (3)	C2—C6—H6B	109.6
O1—C1—C2	106.6 (3)	H6A—C6—H6B	108.1
C10—C1—C2	105.2 (3)	C8—C7—S1	109.5 (4)
C3—C1—C2	107.1 (3)	C8—C7—H7A	109.8
C1—C2—C6	107.5 (3)	S1—C7—H7A	109.8
C1—C2—S1	113.8 (2)	C8—C7—H7B	109.8
C6—C2—S1	108.4 (3)	S1—C7—H7B	109.8
C1—C2—S2	110.7 (3)	H7A—C7—H7B	108.2
C6—C2—S2	110.4 (3)	C7—C8—S2	114.0 (4)
S1—C2—S2	106.1 (2)	C7—C8—H8A	108.8
C4—C3—C1	112.1 (3)	S2—C8—H8A	108.8

C4—C3—H3A	109.2	C7—C8—H8B	108.8
C1—C3—H3A	109.2	S2—C8—H8B	108.8
C4—C3—H3B	109.2	H8A—C8—H8B	107.7
C1—C3—H3B	109.2	O1—C9—H9A	109.5
H3A—C3—H3B	107.9	O1—C9—H9B	109.5
C3—C4—C5	108.6 (4)	H9A—C9—H9B	109.5
C3—C4—H4A	110.0	O1—C9—H9C	109.5
C5—C4—H4A	110.0	H9A—C9—H9C	109.5
C3—C4—H4B	110.0	H9B—C9—H9C	109.5
C5—C4—H4B	110.0	C11—C10—C1	114.7 (4)
H4A—C4—H4B	108.3	C11—C10—H10	114 (3)
C11—C5—C4	109.5 (4)	C1—C10—H10	112 (4)
C11—C5—C6	108.0 (4)	C10—C11—C5	114.0 (4)
C4—C5—C6	106.0 (4)	C10—C11—H11	115 (4)
C11—C5—H5	111.1	C5—C11—H11	119 (4)
C4—C5—H5	111.1		

C7—S1—C2—C1	−91.9 (3)	S2—C2—C1—O1	−62.5 (3)
C7—S1—C2—C6	148.6 (3)	C6—C2—C1—C10	−60.7 (4)
C7—S1—C2—S2	30.0 (3)	S1—C2—C1—C10	179.3 (3)
C2—S1—C7—C8	−37.9 (5)	S2—C2—C1—C10	60.0 (3)
C2—S2—C8—C7	−9.2 (5)	C6—C2—C1—C3	56.0 (4)
C8—S2—C2—C1	108.2 (3)	S1—C2—C1—C3	−64.0 (4)
C8—S2—C2—C6	−132.9 (3)	S2—C2—C1—C3	176.7 (3)
C8—S2—C2—S1	−15.6 (3)	C1—C2—C6—C5	6.6 (4)
C9—O1—C1—C10	55.7 (5)	S1—C2—C6—C5	129.9 (3)
C9—O1—C1—C3	−70.9 (5)	S2—C2—C6—C5	−114.3 (4)
C9—O1—C1—C2	171.9 (4)	C5—C4—C3—C1	3.8 (6)
O1—C1—C3—C4	179.3 (4)	C3—C4—C5—C11	−56.4 (5)
C10—C1—C3—C4	50.0 (5)	C3—C4—C5—C6	59.8 (5)
C2—C1—C3—C4	−63.8 (5)	C11—C5—C6—C2	51.3 (5)
O1—C1—C10—C11	177.3 (4)	C4—C5—C6—C2	−65.9 (4)
C3—C1—C10—C11	−54.6 (5)	S1—C7—C8—S2	31.2 (6)
C2—C1—C10—C11	60.3 (5)	C5—C11—C10—C1	0.7 (7)
C6—C2—C1—O1	176.8 (3)	C10—C11—C5—C4	56.4 (6)
S1—C2—C1—O1	56.8 (4)	C10—C11—C5—C6	−58.6 (6)