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Crystal structures of sulfonamide protected bicyclic guanidines: (*S*)-8-{[(*tert*-butyldimethylsilyl)oxy]-methyl}-1-[(2,2,4,6,7-pentamethyl-2,3-dihydro-benzofuran-5-yl)sulfonyl]-1,3,4,6,7,8-hexahydro-2*H*-pyrimido[1,2-*a*]pyrimidin-1-ium trifluoro-methanesulfonate and (*S*)-8-(iodomethyl)-1-tosyl-1,3,4,6,7,8-hexahydro-2*H*-pyrimido[1,2-*a*]-pyrimidin-1-ium iodide

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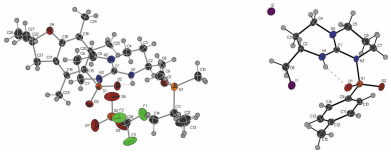
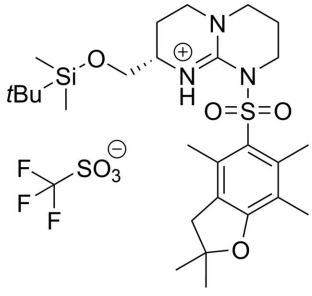
Two compounds, (*S*)-8-{[(*tert*-butyldimethylsilyl)oxy]methyl}-1-[(2,2,4,6,7-pentamethyl-2,3-dihydrobenzofuran-5-yl)sulfonyl]-1,3,4,6,7,8-hexahydro-2*H*-pyrimido[1,2-*a*]pyrimidin-1-ium trifluoromethanesulfonate, $C_{27}H_{46}N_3O_4SSi^+\cdot CF_3O_3S^-$, (**1**) and (*S*)-8-(iodomethyl)-1-tosyl-1,3,4,6,7,8-hexahydro-2*H*-pyrimido[1,2-*a*]pyrimidin-1-ium iodide, $C_{15}H_{21}IN_3O_2S^+\cdot I^-$, (**2**), have been synthesized and characterized. They are bicyclic guanidinium salts and were synthesized from *N*-(*tert*-butoxycarbonyl)-L-methionine (Boc-L-Met-OH). The guanidine is protected by a 2,2,4,6,7-pentamethyldihydrobenzofuran-5-sulfonyl (Pbf, **1**) or a tosyl (**2**) group. In the crystals of both compounds, the guanidinium group is almost planar and the N–H forms an intramolecular hydrogen bond in a six-membered ring to the oxygen atom of the sulfonamide protecting group.

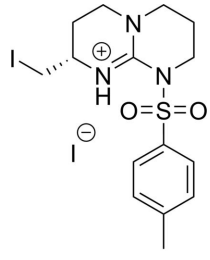
1. Chemical context

Cyclic guanidines have been observed as a structural motif in alkaloid natural products and been extensively explored as organocatalysts, ligands and receptors, among other applications (Chou *et al.*, 2019; Dong *et al.*, 2018; Lemrová & Soural, 2015; Selig, 2013; Fu & Tan, 2011; Coles, 2009; Leow & Tan, 2009; Best *et al.*, 2003). We have prepared sulfonamide-protected bicyclic guanidine derivatives **1** and **2** with a view towards incorporating the bicyclic guanidine moiety into synthetic peptides as torsionally constrained mimics of arginine.





1



2

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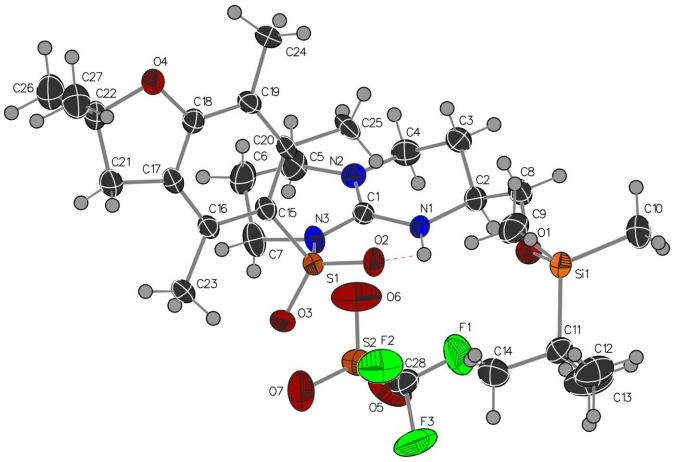

Figure 1
The molecular structure of **1** with ellipsoids at the 50% probability level.

2. Structural commentary

The molecular structures of **1** and **2** are shown in Figs. 1 and 2, respectively. The guanidine group is protonated in both cases and the central carbon (C1) adopts an essentially planar geometry with N—C—N bond angles close to 120°. The C1—N1 bond lengths of 1.323 (4) and 1.328 (6) Å (in **1** and **2**, respectively) are slightly shorter than the C1—N3 bond lengths of 1.380 (4) and 1.400 (7) Å. The sulfonamide group adopts a conformation that allows the formation of an intramolecular NH···OS hydrogen bond (Figs. 1 and 2, Tables 1 and 2). A second less optimal intramolecular N—H···O contact to the oxygen of the siloxy group is observed in compound **1**. The alkyl substituent on the six-membered ring lies in an equatorial-like conformation where the chiral centre originates from the starting material (Boc-L-Met-OH) used in the synthesis.

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O1	0.88	2.36	2.712 (4)	104
N1—H1···O2	0.88	2.07	2.700 (4)	127
C3—H3B···O7^{i}	0.99	2.36	3.229 (5)	146
C5—H5B···F1^{ii}	0.99	2.43	3.239 (6)	139
C24—H24A···O4	0.98	2.42	2.877 (4)	108
C25—H25B···O2	0.98	2.21	3.022 (5)	139
C7—H7A···O5^{iii}	0.99	2.60	3.433 (5)	142
C24—H24B···O6^{ii}	0.98	2.53	3.462 (5)	159

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

3. Supramolecular features

Compound **1** packs with the guanidinium groups and triflate counter-ions arranged in layers perpendicular to the c axis (Fig. 3). These are interleaved with layers composed of the *tert*-butyldimethylsilyl and 2,2,4,6,7-pentamethyldihydrobenzofuran-5-sulfonyl (Pbf) protecting groups. Each triflate anion is surrounded by four cations, forming interactions with the C—H groups of the guanidinium as depicted in Fig. 4. Additionally, weak C–H···F and C—H···O interactions also consolidate the structure (Table 1).

The molecular packing of **2** is illustrated in Fig. 5. The tosyl groups of adjacent molecules pack against each other placing the centroids of the aromatic rings 5.301 (4) Å apart. The methyl group forms a C—H···π interaction such that the methyl carbon C15 lies 3.597 (9) Å from the centroid of the adjacent aromatic ring. The other face of the ring forms an intermolecular interaction with the iodine atom I1 that lies

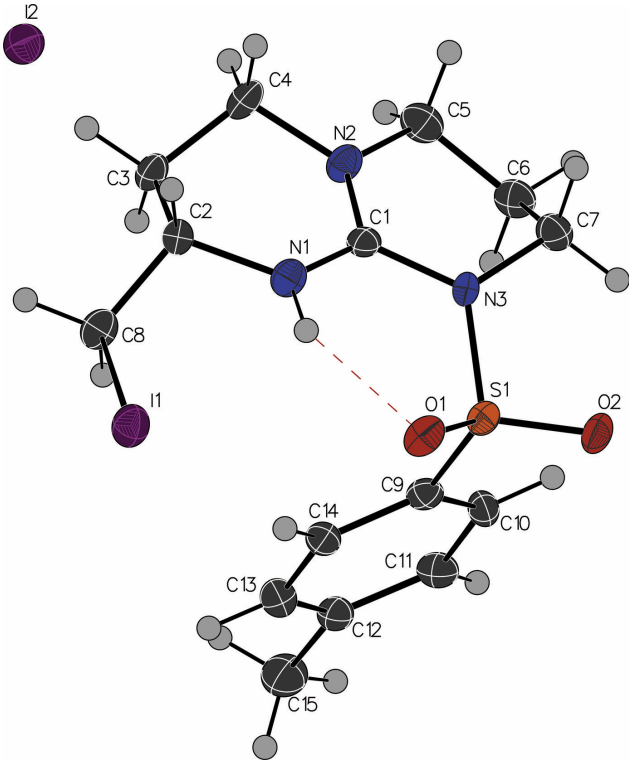

Figure 2
The molecular structure of **2** with ellipsoids at the 50% probability level.

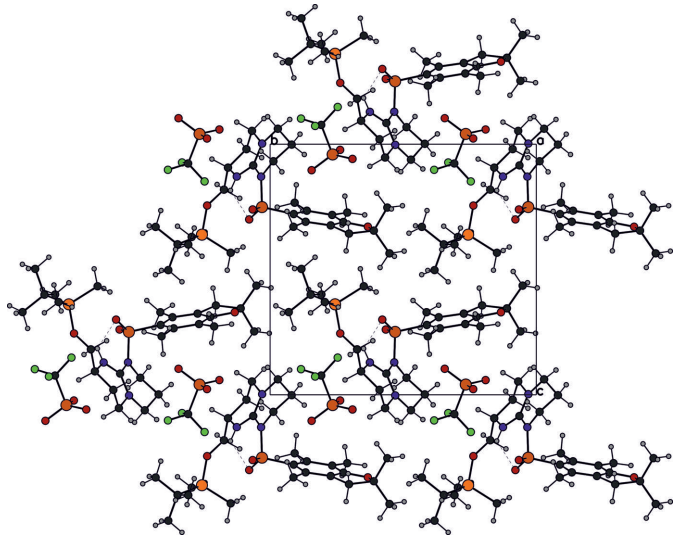

Figure 3
View of the packing of **1** along the a axis.

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···I1	0.88	2.87	3.3359 (4)	115
N1—H1···O1	0.88	2.00	2.737 (5)	141
C7—H7*A*···O2	0.99	2.29	2.807 (8)	112
C14—H14···O1	0.95	2.57	2.934 (8)	103
C6—H6*B*···O1^{i}	0.99	2.58	3.425 (8)	143
C7—H7*A*···O2^{ii}	0.99	2.56	3.391 (8)	141
C10—H10···O2^{ii}	0.95	2.49	3.441 (7)	174

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

3.600 (3) Å from the ring centroid. Weak C—H···O interactions also help to hold the structure together (Table 2).

4. Database survey

A search of the Cambridge Structural Database for sulfonyl guanidines revealed four related compounds. *N*,3-Diisopropyl-4-mesityl-1-[(4-methylphenyl)sulfonyl]imidazolidin-2-iminium tribromo(methanol)zinc(II) methanol solvate (CSD refcode: FOFJIV; Craig II *et al.*, 2014) is a monocyclic guanidine bearing a tosyl group. This structure displays the same intramolecular SO—HN hydrogen bond that is observed in **1** and **2**. 3,4,6,7,8,9-Hexahydro-2*H*-pyrimido[1,2-*a*]pyrimidin-1-ium-1-sulfinate (CSD refcode: SOWPOM; Adenot *et al.*, 2019) is a bicyclic guanidine–sulfur dioxide adduct that features a similar hydrogen bond. The guanidinium N to sulfonamide O distances in **1** and **2** are significantly shorter at 2.700 (4) and 2.737 (5) Å than the corresponding distances in related compounds that lack this intramolecular interaction. A neutral bicyclic tosylguanidine reported (CSD Refcode: WEWGAK) by Watanabe *et al.* (2023) places the sulfonamide oxygen

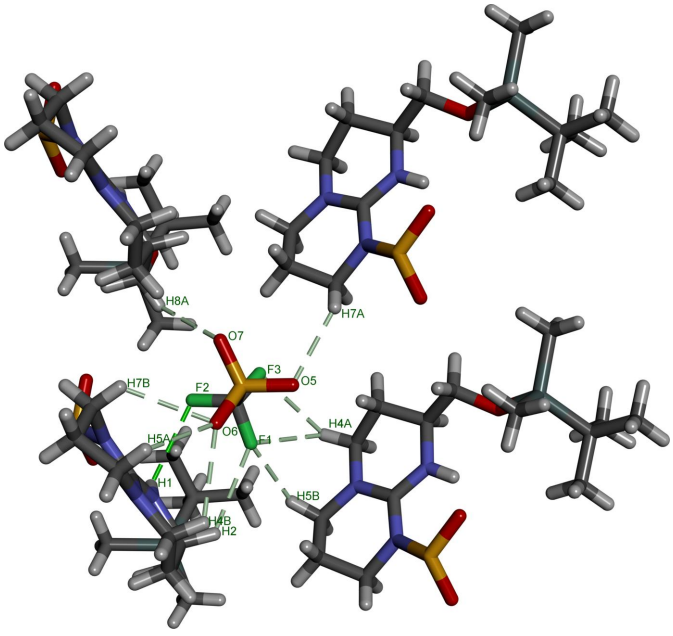

Figure 4
View of the packing of **1** around the trifluoromethanesulfonate anion. The pentamethyl-2,3-dihydrobenzofuran groups have been hidden for clarity.

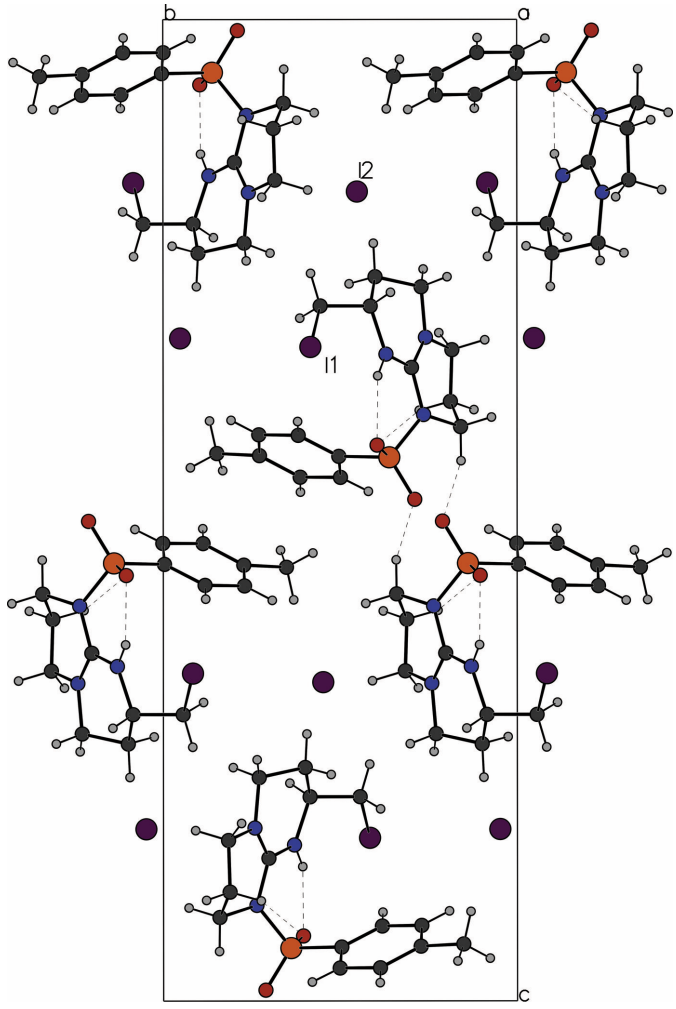

Figure 5
View of the packing of **2** along the *a* axis.

3.173 (5) Å from the guanidine nitrogen. The hydrogen bond is also absent in *N*-(1,3-dibenzyl-1,3,4,4a,5,7a-hexahydro-2*H*-cyclopenta[*d*]pyrimidin-2-ylidene)-2,2,5,7,8-pentamethylchromane-6-sulfonamide (CSD refcode: SIMSIS; Aranha Potter *et al.*, 2013) as the guanidinium group is fully alkylated resulting in an N—O distance of 2.926 (3) Å.

5. Synthesis and crystallization

Compounds **1** and **2** were synthesized from Boc-L-Met-OH according to Figs. 6 and 7, respectively. Full synthetic procedures have been reported elsewhere (Alaboosh, 2017; Hill, 2012). Single crystals of **1** were grown by vapour diffusion from an EtOH/H_2O solution. Single crystals of **2** were grown by vapour diffusion from a MeCN/Et_2O solution.

Spectroscopic data for compound **1**:

^{1}H NMR (400 MHz, $CDCl_3$) δ 3.92–3.79 (*m*, 2H, SO_2NCH_2), 3.62–3.60 (*m*, 1H, OCHH), 3.33–3.28 (*m*, 1H, NCH), 3.12–2.99 (*m*, 4H, $NCHCH_2CH_2$, $SO_2NCH_2CH_2CH_2$), 2.83–2.80 (*m*, 1H, OCHH), 3.00 (*s*, 2H, furan-CH_2), 2.52 (*s*, 3H, $ArCH_3$), 2.48 (*s*, 3H, $ArCH_3$), 2.13 (*s*, 3H, $ArCH_3$), 2.06–1.93 (*m*, 3H, $SO_2NCH_2CH_2$, $NCHCH_2$), 1.46 [*s*, 6H, $(CH_3)_2$], 1.37–

Table 3
Experimental details.

	1	**2**
Crystal data		
Chemical formula	$C_{27}H_{46}N_3O_4SSi^+\cdot CF_3O_3S^-$	$C_{15}H_{21}IN_3O_2S^+\cdot I^-$
M_r	685.89	561.21
Crystal system, space group	Monoclinic, $P2_1$	Orthorhombic, $P2_12_12_1$
Temperature (K)	150	150
a, b, c (Å)	8.5784 (3), 14.4797 (5), 13.6961 (5)	6.6117 (2), 10.1482 (2), 28.1444 (9)
α, β, γ (°)	90, 96.052 (4), 90	90, 90, 90
V (Å^3)	1691.75 (10)	1888.40 (9)
Z	2	4
Radiation type	Cu $K\alpha$	Mo $K\alpha$
μ (mm^{-1})	2.32	3.45
Crystal size (mm)	0.33 × 0.06 × 0.04	0.41 × 0.20 × 0.20
Data collection		
Diffractometer	Agilent SuperNova, Dual, Cu at zero, Atlas	Nonius KappaCCD
Absorption correction	Gaussian (*CrysAlis PRO*; Agilent,2014)	Empirical (using intensity measurements) (*DENZO*/*SCALEPACK*; Otwinowski & Minor, 1997)
T_{min}, T_{max}	0.943, 0.988	0.339, 0.545
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	10136, 5593, 5350	4087, 4087, 3858
R_{int}	0.027	0.030
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.623	0.648
Refinement		
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.037, 0.097, 1.02	0.031, 0.072, 1.04
No. of reflections	5593	4087
No. of parameters	408	207
No. of restraints	1	0
H-atom treatment	H-atom parameters constrained	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.63, −0.48	0.77, −0.79
Absolute structure	Refined as an inversion twin	Flack x determined using 1475 quotients $[(I^+)-(I[(I^+)-(I^-)]/[(I^+)+(I^-)]^{-})]/[(I^+)+(I^-)]$ (Parsons et al., 2013)
Absolute structure parameter	0.39 (2)	−0.002 (19)

Computer programs: *CrysAlis PRO* (Agilent, 2014), *COLLECT* (Bruker, 2004), *HKL DENZO* and *SCALEPACK* (Otwinowski & Minor 1997), *SHELXS* (Sheldrick, 2008), *SHELXL2018/1* (Sheldrick, 2015), *OLEX2* (Dolomanov *et al.*, 2009) and *PLATON* (Spek, 2020).

1.29 (*m*, 1H, NCHCHH), 0.85 (*s*, 9H, *t*-Bu), 0.01 [*s*, 6H, Si(CH$_3$)$_2$]. **^{13}C NMR (100 MHz, CDCl$_3$) δ** 159.1 (ArC), 143.2 (ArC), 137.7 (ArC), 132.7 (ArC), 124.3 (ArC), 117.1 (ArC), 86.4 (C(CH$_3$)$_2$), 60.3 (CH$_2$), 50.3 (CH), 48.3 (CH$_2$), 47.7 (CH$_2$), 43.2 (CH$_2$), 42.1 (CH$_2$), 40.3 (CH$_2$), 28.5 (CH$_3$), 28.5 (CH$_3$), 27.7 (CH$_2$), 25.9 (*t*-Bu), 23.3 (CH$_2$), 19.1 (CH$_3$), 18.1 [C(CH$_3$)$_3$], 17.2 (CH$_3$), 12.5 (CH$_3$), −5.3 [Si(CH$_3$)$_2$]. **HRMS-ES+ (*m*/*z*):** calculated for $C_{27}H_{46}N_3O_4SSi$ $[M + H]^+$: 536.2973, found 536.2999.

Spectroscopic data for compound **2**:

^{1}H NMR (400 MHz, CD$_3$OD) δ 7.99 (*d*, 2H, $^3J_{HH}$ = 8.4 Hz, ArCH), 7.56 (*d*, 2H, $^3J_{HH}$ = 8.4 Hz, ArCH), 4.03–3.96 (*m*, 1H, SO$_2$NCH*H*), 3.93–3.85 (*m*, 2H, SO$_2$NCH*H*, NC*H*), 3.60–3.38 (*m*, 6H, ICH$_2$, SO$_2$NCH$_2$CH$_2$C*H*$_2$, NCHCH$_2$C*H*$_2$), 2.49 (*s*, 3H, ArCH$_3$), 2.24–2.17 (*m*, 1H, SO$_2$NCH$_2$C*H*$_2$), 2.11–2.02 (*m*, 1H, SO$_2$NCH$_2$C*H*$_2$), 1.92–1.83 (*m*, 1H, NCHC*H*$_2$), 1.81–1.73 (*m*, 1H, NCHC*H*$_2$). **^{13}C NMR (125 MHz, CD$_3$OD) δ** 150.9 (ArC), 148.7 (ArC), 135.0 (ArCH), 129.2 (ArCH), 52.7 (CH), 49.9 (CH$_2$), 47.9 (CH$_2$), 45.8 (CH$_2$), 26.3 (CH$_2$), 21.8 (CH$_2$) 21.8 (CH$_3$), 7.1 (CH$_2$I). **HRMS-ESI+ (*m*/*z*):** calculated for $C_{15}H_{21}IN_3O_2S$ $[M + H]^+$: 434.0394, found: 434.0404.

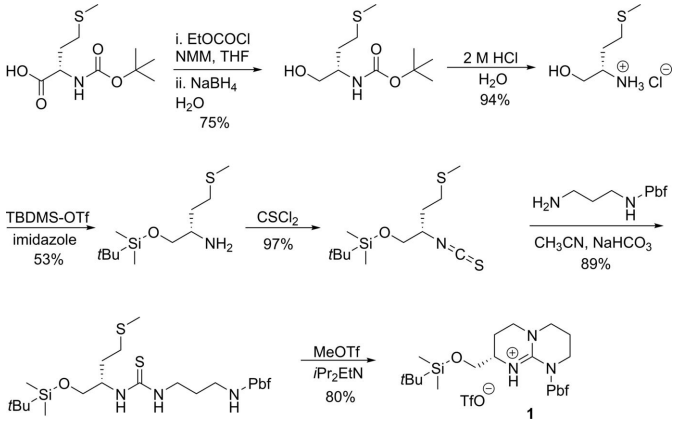

Figure 6
Synthetic scheme for **1**.

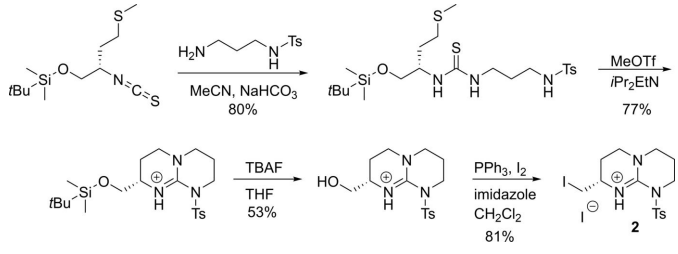

Figure 7
Synthetic scheme for **2**.

6. Refinement

Crystallographic data for **1** were collected on an Agilent SuperNova Dual Atlas diffractometer with a mirror monochromator using Cu $K\alpha$ (λ = 1.5418 Å) radiation, equipped with an Oxford cryosystems cooling apparatus. Crystallographic data for **2** were collected at 150 K on a Nonius Kappa CCD diffractometer using graphite monochromated Mo $K\alpha$ radiation (λ = 0.71073 Å) equipped with an Oxford Cryosystems cooling apparatus.

Crystal data, data collection and structure refinement details are summarized in Table 3. The structures were solved using direct methods with *SHELXS* (Sheldrick, 2008) and refined with *SHELXL* (Sheldrick, 2015). All non-hydrogen atoms were refined anisotropically, while the hydrogen atoms were inserted in idealized positions with U_{iso} set at 1.2 or 1.5 times the U_{eq} of the parent atom. The absolute structures were determined based on the anomalous dispersion effects in the diffraction data. The Flack parameter for **1** indicated possible racemic twinning, which was confirmed by TWIN/BASF refinement to give x = 0.39. The value for **2** is consistent with an untwinned structure.

Images were produced using *Olex2-1.5* (Dolomanov *et al.*, 2009) and *Discovery Studio Visualizer* (v21.1.0.20298; BIOVIA, 2024). Hydrogen bonds and other intermolecular contacts were identified with *PLATON* (Spek, 2020) and *Discovery Studio Visualizer*.

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supporting information

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Crystal structures of sulfonamide protected bicyclic guanidines: (*S*)-8-{[(*tert*-butyldimethylsilyl)oxy]methyl}-1-[(2,2,4,6,7-pentamethyl-2,3-dihydrobenzofuran-5-yl)sulfonyl]-1,3,4,6,7,8-hexahydro-2*H*-pyrimido[1,2-*a*]pyrimidin-1-ium trifluoromethanesulfonate and (*S*)-8-(iodomethyl)-1-tosyl-1,3,4,6,7,8-hexahydro-2*H*-pyrimido[1,2-*a*]pyrimidin-1-ium iodide

Jamal M. H. Alaboosh, Steven P. Hill, Benson M. Kariuki and James E. Redman

Computing details

(*S*)-8-{[(*tert*-Butyldimethylsilyl)oxy]methyl}-1-[(2,2,4,6,7-pentamethyl-2,3-dihydrobenzofuran-5-yl)sulfonyl]-1,3,4,6,7,8-hexahydro-2*H*-pyrimido[1,2-*a*]pyrimidin-1-ium trifluoromethanesulfonate (1)

Crystal data

$C_{27}H_{46}N_3O_4SSi^+\cdot CF_3O_3S^-$
M_r = 685.89
Monoclinic, $P2_1$
a = 8.5784 (3) Å
b = 14.4797 (5) Å
c = 13.6961 (5) Å
β = 96.052 (4)°
V = 1691.75 (10) Å^3
Z = 2
$F(000)$ = 728
D_x = 1.346 Mg m^{-3}
Cu $K\alpha$ radiation, λ = 1.54184 Å
Cell parameters from 3508 reflections
θ = 4.4–73.8°
μ = 2.32 mm^{-1}
T = 150 K
Block, colourless
0.33 × 0.06 × 0.04 mm

Data collection

Agilent SuperNova, Dual, Cu at zero, Atlas diffractometer
ω scans
Absorption correction: gaussian (CrysAlisPro; Agilent,2014)
T_{min} = 0.943, T_{max} = 0.988
10136 measured reflections
5593 independent reflections
5350 reflections with $I > 2\sigma(I)$
R_{int} = 0.027
θ_{max} = 73.8°, θ_{min} = 4.5°
h = −10→10
k = −17→17
l = −16→16

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.097
S = 1.02
5593 reflections
408 parameters
1 restraint
Primary atom site location: structure-invariant direct methods
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0537P)^2 + 0.493P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$

$\Delta\rho_{max}$ = 0.63 e Å^{-3}
$\Delta\rho_{min}$ = −0.47 e Å^{-3}

Absolute structure: Refined as an inversion twin
Absolute structure parameter: 0.39 (2)

Special details

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

Refinement. Refined as a 2-component inversion twin.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.6033 (3)	0.0629 (2)	0.0845 (2)	0.0240 (6)
C2	0.3643 (4)	0.1575 (2)	0.0936 (2)	0.0284 (7)
H2	0.373994	0.218073	0.059559	0.034*
C3	0.2893 (4)	0.0884 (3)	0.0195 (3)	0.0332 (7)
H3A	0.262817	0.030939	0.053364	0.040*
H3B	0.191646	0.114320	−0.014690	0.040*
C4	0.4044 (4)	0.0677 (3)	−0.0534 (3)	0.0350 (7)
H4A	0.357854	0.022887	−0.102633	0.042*
H4B	0.428321	0.125130	−0.088146	0.042*
C5	0.6408 (5)	−0.0298 (3)	−0.0615 (3)	0.0458 (10)
H5A	0.689976	0.008522	−0.109703	0.055*
H5B	0.569935	−0.074534	−0.098413	0.055*
C6	0.7667 (5)	−0.0816 (3)	0.0015 (4)	0.0502 (10)
H6A	0.718912	−0.129145	0.040960	0.060*
H6B	0.838164	−0.112741	−0.040288	0.060*
C7	0.8560 (4)	−0.0128 (3)	0.0679 (3)	0.0403 (8)
H7A	0.939829	−0.044524	0.110425	0.048*
H7B	0.905340	0.033850	0.028138	0.048*
C8	0.2761 (4)	0.1728 (3)	0.1813 (3)	0.0333 (7)
H8A	0.170991	0.198660	0.160565	0.040*
H8B	0.262903	0.113620	0.215726	0.040*
C9	0.3773 (6)	0.1475 (3)	0.4330 (3)	0.0469 (9)
H9A	0.298018	0.101375	0.410458	0.070*
H9B	0.371689	0.160347	0.502847	0.070*
H9C	0.481673	0.123899	0.423649	0.070*
C10	0.1355 (5)	0.2941 (3)	0.3702 (4)	0.0518 (11)
H10A	0.112109	0.348206	0.328246	0.078*
H10B	0.123141	0.310132	0.438453	0.078*
H10C	0.063191	0.243892	0.348736	0.078*
C11	0.4901 (4)	0.3470 (3)	0.3994 (3)	0.0329 (7)
C12	0.4700 (7)	0.3821 (4)	0.5020 (4)	0.0589 (12)
H12A	0.484443	0.330792	0.548814	0.088*
H12B	0.364584	0.407852	0.502918	0.088*
H12C	0.548150	0.430070	0.520430	0.088*
C13	0.4753 (8)	0.4294 (4)	0.3298 (4)	0.0747 (18)
H13A	0.369261	0.455009	0.327393	0.112*

H13B	0.495358	0.409358	0.263920	0.112*
H13C	0.551844	0.476737	0.353329	0.112*
C14	0.6544 (6)	0.3066 (4)	0.4006 (5)	0.0684 (15)
H14A	0.732249	0.354354	0.420425	0.103*
H14B	0.670084	0.284193	0.334761	0.103*
H14C	0.666527	0.255118	0.447235	0.103*
C15	0.8052 (4)	−0.0894 (2)	0.2787 (2)	0.0250 (6)
C16	0.9498 (3)	−0.1338 (2)	0.3094 (2)	0.0266 (6)
C17	0.9419 (4)	−0.2285 (3)	0.3241 (2)	0.0299 (7)
C18	0.8017 (4)	−0.2756 (2)	0.3106 (3)	0.0293 (7)
C19	0.6591 (4)	−0.2337 (2)	0.2825 (2)	0.0274 (6)
C20	0.6615 (3)	−0.1383 (2)	0.2653 (2)	0.0251 (6)
C21	1.0691 (4)	−0.2967 (3)	0.3563 (4)	0.0446 (9)
H21A	1.111684	−0.285926	0.425402	0.053*
H21B	1.155647	−0.293256	0.314050	0.053*
C22	0.9847 (4)	−0.3900 (3)	0.3449 (3)	0.0383 (8)
C23	1.1083 (4)	−0.0882 (3)	0.3296 (3)	0.0348 (7)
H23A	1.184820	−0.133406	0.358473	0.052*
H23B	1.101159	−0.036815	0.375526	0.052*
H23C	1.141792	−0.064792	0.268038	0.052*
C24	0.5107 (4)	−0.2903 (3)	0.2726 (3)	0.0340 (7)
H24A	0.536982	−0.355774	0.282412	0.051*
H24B	0.455493	−0.281562	0.206879	0.051*
H24C	0.443114	−0.270473	0.322062	0.051*
C25	0.5054 (4)	−0.0929 (3)	0.2334 (3)	0.0334 (7)
H25A	0.481814	−0.098784	0.162068	0.050*
H25B	0.510525	−0.027348	0.251311	0.050*
H25C	0.422930	−0.123127	0.266095	0.050*
C26	1.0218 (5)	−0.4408 (3)	0.2530 (3)	0.0518 (10)
H26A	0.958062	−0.497013	0.244839	0.078*
H26B	1.133183	−0.457475	0.259351	0.078*
H26C	0.998221	−0.400819	0.195675	0.078*
C27	1.0088 (5)	−0.4488 (3)	0.4357 (3)	0.0486 (10)
H27A	0.981648	−0.413149	0.492346	0.073*
H27B	1.118750	−0.468025	0.446673	0.073*
H27C	0.941545	−0.503578	0.427334	0.073*
C28	0.8065 (5)	0.3074 (3)	0.0768 (4)	0.0524 (11)
N1	0.5220 (3)	0.12491 (19)	0.1289 (2)	0.0263 (5)
H1	0.566303	0.148499	0.184230	0.032*
N2	0.5494 (3)	0.0295 (2)	−0.00255 (19)	0.0291 (5)
N3	0.7461 (3)	0.0332 (2)	0.1294 (2)	0.0300 (6)
O1	0.3655 (3)	0.23600 (18)	0.24468 (18)	0.0356 (6)
O2	0.6885 (3)	0.07815 (18)	0.30011 (17)	0.0330 (5)
O3	0.9590 (3)	0.06521 (18)	0.2604 (2)	0.0377 (6)
O4	0.8158 (3)	−0.36761 (18)	0.3294 (2)	0.0407 (6)
O5	0.8248 (4)	0.3436 (3)	−0.1041 (3)	0.0778 (13)
O6	0.7409 (5)	0.1900 (3)	−0.0587 (3)	0.0731 (11)
O7	1.0094 (4)	0.2368 (3)	−0.0229 (3)	0.0648 (10)

Si1	0.33991 (10)	0.25617 (7)	0.36096 (7)	0.0305 (2)
S1	0.80381 (8)	0.03078 (5)	0.25091 (5)	0.02638 (16)
S2	0.84970 (11)	0.26548 (7)	−0.04097 (7)	0.0423 (2)
F1	0.6552 (4)	0.3303 (3)	0.0731 (4)	0.1025 (16)
F2	0.8335 (4)	0.2457 (2)	0.1485 (2)	0.0695 (8)
F3	0.8890 (5)	0.3835 (2)	0.1023 (3)	0.0914 (13)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0235 (14)	0.0214 (14)	0.0286 (14)	−0.0013 (11)	0.0101 (11)	0.0019 (11)
C2	0.0252 (15)	0.0261 (16)	0.0332 (16)	0.0028 (12)	−0.0006 (12)	0.0008 (13)
C3	0.0277 (16)	0.0345 (18)	0.0357 (17)	0.0004 (14)	−0.0040 (13)	−0.0014 (14)
C4	0.0370 (18)	0.0371 (18)	0.0301 (16)	−0.0033 (15)	−0.0007 (13)	−0.0007 (14)
C5	0.041 (2)	0.059 (3)	0.039 (2)	0.0010 (19)	0.0118 (16)	−0.0197 (18)
C6	0.050 (2)	0.044 (2)	0.059 (3)	0.0054 (19)	0.022 (2)	−0.012 (2)
C7	0.0326 (18)	0.053 (2)	0.0379 (18)	0.0119 (17)	0.0142 (14)	0.0007 (17)
C8	0.0246 (15)	0.0392 (18)	0.0353 (17)	0.0030 (14)	−0.0002 (13)	−0.0051 (15)
C9	0.057 (2)	0.0314 (19)	0.053 (2)	0.0008 (17)	0.0131 (19)	0.0044 (17)
C10	0.038 (2)	0.052 (3)	0.067 (3)	0.0096 (19)	0.0119 (19)	−0.014 (2)
C11	0.0388 (18)	0.0284 (17)	0.0310 (16)	0.0004 (14)	0.0012 (13)	0.0007 (14)
C12	0.082 (3)	0.052 (3)	0.044 (2)	−0.016 (2)	0.014 (2)	−0.014 (2)
C13	0.105 (5)	0.052 (3)	0.060 (3)	−0.026 (3)	−0.026 (3)	0.024 (2)
C14	0.040 (2)	0.064 (3)	0.100 (4)	−0.004 (2)	−0.001 (2)	−0.035 (3)
C15	0.0213 (14)	0.0279 (15)	0.0264 (14)	0.0009 (12)	0.0058 (11)	0.0007 (12)
C16	0.0192 (14)	0.0317 (17)	0.0291 (15)	0.0006 (12)	0.0039 (11)	−0.0043 (13)
C17	0.0204 (14)	0.0327 (17)	0.0367 (16)	0.0035 (13)	0.0046 (12)	−0.0001 (14)
C18	0.0268 (16)	0.0246 (15)	0.0370 (17)	0.0025 (12)	0.0063 (13)	0.0020 (13)
C19	0.0215 (14)	0.0315 (16)	0.0298 (14)	−0.0033 (13)	0.0056 (11)	−0.0024 (13)
C20	0.0164 (13)	0.0314 (16)	0.0281 (14)	0.0012 (12)	0.0049 (11)	0.0000 (12)
C21	0.0267 (17)	0.0324 (19)	0.074 (3)	0.0048 (15)	0.0039 (17)	0.0027 (18)
C22	0.0313 (17)	0.0317 (18)	0.053 (2)	0.0074 (14)	0.0080 (15)	0.0009 (16)
C23	0.0190 (15)	0.0342 (18)	0.050 (2)	−0.0001 (13)	−0.0020 (13)	−0.0014 (15)
C24	0.0251 (16)	0.0357 (19)	0.0411 (18)	−0.0060 (14)	0.0034 (13)	−0.0011 (15)
C25	0.0152 (13)	0.0419 (19)	0.0429 (19)	−0.0020 (13)	0.0028 (12)	0.0093 (16)
C26	0.047 (2)	0.054 (3)	0.056 (2)	0.0102 (19)	0.0099 (19)	−0.004 (2)
C27	0.045 (2)	0.049 (3)	0.052 (2)	0.0091 (18)	0.0093 (17)	0.0053 (19)
C28	0.050 (2)	0.0266 (18)	0.085 (3)	−0.0061 (18)	0.029 (2)	−0.004 (2)
N1	0.0231 (13)	0.0224 (13)	0.0328 (13)	0.0037 (10)	0.0005 (10)	−0.0051 (10)
N2	0.0317 (13)	0.0289 (13)	0.0275 (12)	−0.0013 (12)	0.0065 (10)	−0.0021 (12)
N3	0.0235 (12)	0.0345 (14)	0.0334 (13)	0.0062 (12)	0.0087 (10)	0.0008 (13)
O1	0.0331 (12)	0.0372 (14)	0.0368 (13)	−0.0004 (10)	0.0046 (10)	−0.0081 (11)
O2	0.0307 (12)	0.0371 (13)	0.0309 (12)	0.0100 (10)	0.0022 (9)	−0.0083 (10)
O3	0.0244 (12)	0.0284 (12)	0.0587 (16)	−0.0032 (9)	−0.0027 (10)	0.0003 (11)
O4	0.0294 (13)	0.0270 (12)	0.0664 (18)	0.0031 (10)	0.0083 (12)	0.0015 (12)
O5	0.0482 (19)	0.088 (3)	0.094 (3)	−0.0116 (18)	−0.0053 (18)	0.059 (2)
O6	0.092 (3)	0.073 (2)	0.0518 (19)	−0.040 (2)	−0.0033 (18)	0.0070 (18)
O7	0.0530 (19)	0.069 (2)	0.073 (2)	0.0229 (17)	0.0087 (16)	−0.0075 (19)

Si1	0.0291 (4)	0.0282 (4)	0.0349 (4)	0.0042 (4)	0.0066 (3)	−0.0028 (4)
S1	0.0200 (3)	0.0256 (4)	0.0333 (4)	0.0017 (3)	0.0019 (3)	−0.0027 (3)
S2	0.0404 (5)	0.0408 (5)	0.0434 (5)	−0.0072 (4)	−0.0063 (3)	0.0111 (4)
F1	0.058 (2)	0.075 (2)	0.185 (5)	0.0069 (16)	0.063 (3)	0.003 (3)
F2	0.089 (2)	0.074 (2)	0.0463 (14)	−0.0234 (17)	0.0077 (13)	−0.0024 (14)
F3	0.106 (3)	0.0572 (19)	0.120 (3)	−0.0416 (19)	0.052 (2)	−0.047 (2)

Geometric parameters (Å, º)

C1—N1	1.323 (4)	C14—H14C	0.9800
C1—N2	1.324 (4)	C15—C20	1.416 (4)
C1—N3	1.380 (4)	C15—C16	1.421 (4)
C2—N1	1.466 (4)	C15—S1	1.781 (3)
C2—C8	1.503 (5)	C16—C17	1.388 (5)
C2—C3	1.519 (5)	C16—C23	1.511 (4)
C2—H2	1.0000	C17—C18	1.378 (5)
C3—C4	1.506 (5)	C17—C21	1.503 (5)
C3—H3A	0.9900	C18—O4	1.361 (4)
C3—H3B	0.9900	C18—C19	1.383 (5)
C4—N2	1.468 (5)	C19—C20	1.402 (5)
C4—H4A	0.9900	C19—C24	1.508 (4)
C4—H4B	0.9900	C20—C25	1.514 (4)
C5—N2	1.462 (4)	C21—C22	1.533 (6)
C5—C6	1.510 (7)	C21—H21A	0.9900
C5—H5A	0.9900	C21—H21B	0.9900
C5—H5B	0.9900	C22—O4	1.478 (4)
C6—C7	1.503 (6)	C22—C27	1.504 (6)
C6—H6A	0.9900	C22—C26	1.520 (6)
C6—H6B	0.9900	C23—H23A	0.9800
C7—N3	1.486 (4)	C23—H23B	0.9800
C7—H7A	0.9900	C23—H23C	0.9800
C7—H7B	0.9900	C24—H24A	0.9800
C8—O1	1.428 (4)	C24—H24B	0.9800
C8—H8A	0.9900	C24—H24C	0.9800
C8—H8B	0.9900	C25—H25A	0.9800
C9—Si1	1.867 (4)	C25—H25B	0.9800
C9—H9A	0.9800	C25—H25C	0.9800
C9—H9B	0.9800	C26—H26A	0.9800
C9—H9C	0.9800	C26—H26B	0.9800
C10—Si1	1.854 (4)	C26—H26C	0.9800
C10—H10A	0.9800	C27—H27A	0.9800
C10—H10B	0.9800	C27—H27B	0.9800
C10—H10C	0.9800	C27—H27C	0.9800
C11—C12	1.521 (6)	C28—F2	1.330 (6)
C11—C13	1.523 (6)	C28—F1	1.335 (6)
C11—C14	1.525 (6)	C28—F3	1.336 (5)
C11—Si1	1.878 (4)	C28—S2	1.798 (5)
C12—H12A	0.9800	N1—H1	0.8800

C12—H12B	0.9800	N3—S1	1.686 (3)
C12—H12C	0.9800	O1—Si1	1.656 (3)
C13—H13A	0.9800	O2—S1	1.429 (2)
C13—H13B	0.9800	O3—S1	1.415 (2)
C13—H13C	0.9800	O5—S2	1.426 (4)
C14—H14A	0.9800	O6—S2	1.441 (4)
C14—H14B	0.9800	O7—S2	1.428 (4)

N1—C1—N2	120.7 (3)	C18—C17—C21	108.1 (3)
N1—C1—N3	119.5 (3)	C16—C17—C21	130.3 (3)
N2—C1—N3	119.8 (3)	O4—C18—C17	113.6 (3)
N1—C2—C8	107.9 (3)	O4—C18—C19	122.7 (3)
N1—C2—C3	108.6 (3)	C17—C18—C19	123.7 (3)
C8—C2—C3	114.7 (3)	C18—C19—C20	116.7 (3)
N1—C2—H2	108.5	C18—C19—C24	120.0 (3)
C8—C2—H2	108.5	C20—C19—C24	123.2 (3)
C3—C2—H2	108.5	C19—C20—C15	119.9 (3)
C4—C3—C2	108.2 (3)	C19—C20—C25	116.6 (3)
C4—C3—H3A	110.1	C15—C20—C25	123.5 (3)
C2—C3—H3A	110.1	C17—C21—C22	103.2 (3)
C4—C3—H3B	110.1	C17—C21—H21A	111.1
C2—C3—H3B	110.1	C22—C21—H21A	111.1
H3A—C3—H3B	108.4	C17—C21—H21B	111.1
N2—C4—C3	110.0 (3)	C22—C21—H21B	111.1
N2—C4—H4A	109.7	H21A—C21—H21B	109.1
C3—C4—H4A	109.7	O4—C22—C27	106.8 (3)
N2—C4—H4B	109.7	O4—C22—C26	106.0 (3)
C3—C4—H4B	109.7	C27—C22—C26	112.8 (3)
H4A—C4—H4B	108.2	O4—C22—C21	105.4 (3)
N2—C5—C6	111.7 (3)	C27—C22—C21	113.1 (4)
N2—C5—H5A	109.3	C26—C22—C21	111.9 (4)
C6—C5—H5A	109.3	C16—C23—H23A	109.5
N2—C5—H5B	109.3	C16—C23—H23B	109.5
C6—C5—H5B	109.3	H23A—C23—H23B	109.5
H5A—C5—H5B	107.9	C16—C23—H23C	109.5
C7—C6—C5	107.7 (4)	H23A—C23—H23C	109.5
C7—C6—H6A	110.2	H23B—C23—H23C	109.5
C5—C6—H6A	110.2	C19—C24—H24A	109.5
C7—C6—H6B	110.2	C19—C24—H24B	109.5
C5—C6—H6B	110.2	H24A—C24—H24B	109.5
H6A—C6—H6B	108.5	C19—C24—H24C	109.5
N3—C7—C6	109.1 (3)	H24A—C24—H24C	109.5
N3—C7—H7A	109.9	H24B—C24—H24C	109.5
C6—C7—H7A	109.9	C20—C25—H25A	109.5
N3—C7—H7B	109.9	C20—C25—H25B	109.5
C6—C7—H7B	109.9	H25A—C25—H25B	109.5
H7A—C7—H7B	108.3	C20—C25—H25C	109.5
O1—C8—C2	107.3 (3)	H25A—C25—H25C	109.5

O1—C8—H8A	110.3	H25B—C25—H25C	109.5
C2—C8—H8A	110.3	C22—C26—H26A	109.5
O1—C8—H8B	110.3	C22—C26—H26B	109.5
C2—C8—H8B	110.3	H26A—C26—H26B	109.5
H8A—C8—H8B	108.5	C22—C26—H26C	109.5
Si1—C9—H9A	109.5	H26A—C26—H26C	109.5
Si1—C9—H9B	109.5	H26B—C26—H26C	109.5
H9A—C9—H9B	109.5	C22—C27—H27A	109.5
Si1—C9—H9C	109.5	C22—C27—H27B	109.5
H9A—C9—H9C	109.5	H27A—C27—H27B	109.5
H9B—C9—H9C	109.5	C22—C27—H27C	109.5
Si1—C10—H10A	109.5	H27A—C27—H27C	109.5
Si1—C10—H10B	109.5	H27B—C27—H27C	109.5
H10A—C10—H10B	109.5	F2—C28—F1	106.7 (4)
Si1—C10—H10C	109.5	F2—C28—F3	108.3 (5)
H10A—C10—H10C	109.5	F1—C28—F3	106.9 (4)
H10B—C10—H10C	109.5	F2—C28—S2	113.6 (3)
C12—C11—C13	107.9 (4)	F1—C28—S2	110.0 (4)
C12—C11—C14	108.2 (4)	F3—C28—S2	111.1 (3)
C13—C11—C14	108.9 (5)	C1—N1—C2	125.4 (3)
C12—C11—Si1	110.6 (3)	C1—N1—H1	117.3
C13—C11—Si1	111.3 (3)	C2—N1—H1	117.3
C14—C11—Si1	109.9 (3)	C1—N2—C5	123.7 (3)
C11—C12—H12A	109.5	C1—N2—C4	119.1 (3)
C11—C12—H12B	109.5	C5—N2—C4	115.9 (3)
H12A—C12—H12B	109.5	C1—N3—C7	118.3 (3)
C11—C12—H12C	109.5	C1—N3—S1	126.9 (2)
H12A—C12—H12C	109.5	C7—N3—S1	114.3 (2)
H12B—C12—H12C	109.5	C8—O1—Si1	125.5 (2)
C11—C13—H13A	109.5	C18—O4—C22	108.0 (3)
C11—C13—H13B	109.5	O1—Si1—C10	109.86 (19)
H13A—C13—H13B	109.5	O1—Si1—C9	109.10 (17)
C11—C13—H13C	109.5	C10—Si1—C9	108.9 (2)
H13A—C13—H13C	109.5	O1—Si1—C11	103.67 (15)
H13B—C13—H13C	109.5	C10—Si1—C11	113.55 (19)
C11—C14—H14A	109.5	C9—Si1—C11	111.59 (19)
C11—C14—H14B	109.5	O3—S1—O2	118.63 (16)
H14A—C14—H14B	109.5	O3—S1—N3	104.99 (15)
C11—C14—H14C	109.5	O2—S1—N3	108.02 (14)
H14A—C14—H14C	109.5	O3—S1—C15	109.86 (15)
H14B—C14—H14C	109.5	O2—S1—C15	110.96 (15)
C20—C15—C16	122.2 (3)	N3—S1—C15	103.06 (15)
C20—C15—S1	118.3 (2)	O5—S2—O7	114.6 (2)
C16—C15—S1	119.4 (2)	O5—S2—O6	116.5 (2)
C17—C16—C15	115.8 (3)	O7—S2—O6	113.6 (3)
C17—C16—C23	117.5 (3)	O5—S2—C28	104.1 (3)
C15—C16—C23	126.7 (3)	O7—S2—C28	103.2 (2)
C18—C17—C16	121.6 (3)	O6—S2—C28	102.5 (2)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O1	0.88	2.36	2.712 (4)	104
N1—H1···O2	0.88	2.07	2.700 (4)	127
C3—H3*B*···O7^{i}	0.99	2.36	3.229 (5)	146
C5—H5*B*···F1^{ii}	0.99	2.43	3.239 (6)	139
C24—H24*A*···O4	0.98	2.42	2.877 (4)	108
C25—H25*B*···O2	0.98	2.21	3.022 (5)	139
C7—H7*A*···O5^{iii}	0.99	2.60	3.433 (5)	142
C24—H24*B*···O6^{ii}	0.98	2.53	3.462 (5)	159

Symmetry codes: (i) *x*−1, *y*, *z*; (ii) −*x*+1, *y*−1/2, −*z*; (iii) −*x*+2, *y*−1/2, −*z*.

(*S*)-8-(Iodomethyl)-1-4-(methylbenzenesulfonyl)-1,3,4,6,7,8-hexahydro-2*H*-pyrimido[1,2-*a*]pyrimidin-1-ium iodide (2)

Crystal data

$C_{15}H_{21}IN_3O_2S^+\cdot I^-$
M_r = 561.21
Orthorhombic, $P2_12_12_1$
a = 6.6117 (2) Å
b = 10.1482 (2) Å
c = 28.1444 (9) Å
V = 1888.40 (9) Å^3
Z = 4
F(000) = 1080
D_x = 1.974 Mg m^{-3}
Mo *Kα* radiation, *λ* = 0.71073 Å
Cell parameters from 3858 reflections
θ = 3.0–27.4°
μ = 3.45 mm^{-1}
T = 150 K
Needle, colourless
0.41 × 0.20 × 0.20 mm

Data collection

Nonius KappaCCD diffractometer
CCD scans
Absorption correction: empirical (using intensity measurements) Denzo/Scalepack
T_{min} = 0.339, T_{max} = 0.545
4087 measured reflections
4087 independent reflections
3858 reflections with $I > 2\sigma(I)$
R_{int} = 0.030
θ_{max} = 27.4°, θ_{min} = 2.1°
h = −8→8
k = −13→13
l = −36→36

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.031
$wR(F^2)$ = 0.072
S = 1.04
4087 reflections
207 parameters
0 restraints
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0251P)^2 + 5.6778P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.77 e Å^{-3}
$\Delta\rho_{min}$ = −0.79 e Å^{-3}
Extinction correction: SHELXL-2018/1 (Sheldrick 2018), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0068 (4)
Absolute structure: Flack x determined using 1475 quotients [(I+)-(I-)]/[(I+)+(I-)] (Parsons, Flack and Wagner, Acta Cryst. B69 (2013) 249-259).
Absolute structure parameter: −0.002 (19)

Special details

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

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

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.6440 (9)	0.2979 (6)	0.3545 (2)	0.0176 (12)
C2	0.8412 (10)	0.4145 (6)	0.2919 (2)	0.0200 (12)
H2	0.949093	0.357300	0.277960	0.024*
C3	0.6515 (10)	0.4008 (6)	0.2620 (2)	0.0224 (13)
H3A	0.557522	0.474061	0.269212	0.027*
H3B	0.687244	0.405578	0.227891	0.027*
C4	0.5492 (11)	0.2707 (7)	0.2723 (2)	0.0263 (15)
H4A	0.421253	0.264838	0.254181	0.032*
H4B	0.637911	0.197383	0.262082	0.032*
C5	0.3238 (11)	0.1838 (6)	0.3364 (2)	0.0272 (13)
H5A	0.340605	0.090934	0.326324	0.033*
H5B	0.206408	0.220687	0.319050	0.033*
C6	0.2819 (9)	0.1881 (7)	0.3890 (2)	0.0245 (14)
H6A	0.178509	0.121590	0.397449	0.029*
H6B	0.230543	0.276178	0.398078	0.029*
C7	0.4773 (10)	0.1594 (6)	0.4148 (2)	0.0231 (14)
H7A	0.453776	0.158156	0.449487	0.028*
H7B	0.530122	0.072116	0.405091	0.028*
C8	0.9149 (9)	0.5559 (7)	0.2918 (2)	0.0227 (13)
H8A	0.806441	0.613563	0.304233	0.027*
H8B	0.942861	0.582957	0.258602	0.027*
C9	0.5690 (9)	0.5036 (6)	0.4450 (2)	0.0187 (13)
C10	0.3777 (9)	0.4989 (6)	0.4664 (2)	0.0195 (13)
H10	0.329194	0.420139	0.480654	0.023*
C11	0.2610 (10)	0.6127 (7)	0.4662 (2)	0.0241 (14)
H11	0.132677	0.611967	0.481372	0.029*
C12	0.3273 (11)	0.7277 (6)	0.4442 (2)	0.0219 (12)
C13	0.5197 (10)	0.7297 (7)	0.4230 (3)	0.0259 (15)
H13	0.567125	0.807997	0.408247	0.031*
C14	0.6406 (10)	0.6182 (6)	0.4236 (2)	0.0223 (14)
H14	0.771147	0.619926	0.409492	0.027*
C15	0.1948 (13)	0.8482 (7)	0.4420 (3)	0.0351 (17)
H15A	0.068847	0.831523	0.459393	0.053*
H15B	0.163584	0.868658	0.408810	0.053*
H15C	0.265650	0.922976	0.456504	0.053*
I1	1.18349 (7)	0.58374 (4)	0.33364 (2)	0.02686 (13)
I2	0.17276 (7)	0.45181 (4)	0.17516 (2)	0.02671 (13)
N1	0.801213	0.370167	0.340930	0.0209 (10)
H1	0.888533	0.393510	0.362975	0.025*

N2	0.5063 (7)	0.2585 (5)	0.3237 (2)	0.0218 (11)
N3	0.6254 (8)	0.2639 (5)	0.40248 (18)	0.0176 (11)
O1	0.9203 (6)	0.3955 (5)	0.43363 (16)	0.0234 (10)
O2	0.6798 (7)	0.2891 (4)	0.48886 (14)	0.0233 (9)
S1	0.7169 (2)	0.36092 (16)	0.44604 (6)	0.0184 (3)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.021 (3)	0.015 (3)	0.017 (3)	0.003 (2)	0.000 (2)	−0.002 (2)
C2	0.023 (3)	0.020 (3)	0.017 (3)	−0.001 (3)	0.001 (2)	0.002 (2)
C3	0.030 (3)	0.024 (3)	0.014 (3)	0.002 (3)	−0.003 (3)	0.002 (2)
C4	0.033 (4)	0.033 (4)	0.013 (3)	0.000 (3)	−0.001 (3)	0.000 (3)
C5	0.024 (3)	0.025 (3)	0.032 (4)	−0.004 (3)	−0.002 (3)	−0.001 (3)
C6	0.019 (3)	0.025 (3)	0.029 (4)	−0.004 (3)	0.001 (3)	−0.002 (3)
C7	0.023 (3)	0.021 (3)	0.025 (3)	−0.004 (3)	0.002 (3)	0.000 (3)
C8	0.021 (3)	0.028 (4)	0.020 (3)	0.002 (3)	−0.005 (2)	0.002 (3)
C9	0.017 (3)	0.022 (3)	0.018 (3)	−0.003 (2)	−0.002 (2)	−0.003 (2)
C10	0.021 (3)	0.018 (3)	0.020 (3)	−0.003 (2)	0.000 (2)	0.003 (2)
C11	0.023 (3)	0.023 (3)	0.026 (3)	0.000 (2)	0.001 (2)	−0.004 (3)
C12	0.027 (3)	0.019 (3)	0.020 (3)	0.002 (3)	−0.001 (3)	−0.001 (2)
C13	0.031 (4)	0.022 (3)	0.025 (4)	−0.002 (3)	0.003 (3)	0.001 (3)
C14	0.025 (4)	0.022 (3)	0.020 (3)	−0.008 (3)	0.000 (2)	−0.001 (2)
C15	0.045 (4)	0.029 (4)	0.032 (4)	0.011 (4)	0.001 (4)	−0.002 (3)
I1	0.0253 (2)	0.0316 (2)	0.0236 (2)	−0.00409 (18)	0.00070 (18)	0.00262 (17)
I2	0.0248 (2)	0.0301 (2)	0.0253 (2)	−0.00338 (18)	0.00167 (18)	−0.00130 (17)
N1	0.019 (2)	0.025 (3)	0.019 (2)	0.001 (2)	−0.001 (2)	0.001 (2)
N2	0.020 (3)	0.028 (3)	0.017 (3)	−0.002 (2)	−0.003 (2)	0.001 (2)
N3	0.022 (3)	0.018 (3)	0.013 (2)	−0.002 (2)	0.0020 (19)	0.003 (2)
O1	0.015 (2)	0.036 (3)	0.020 (2)	−0.0010 (19)	0.0012 (17)	−0.002 (2)
O2	0.029 (2)	0.027 (2)	0.0144 (19)	0.002 (2)	−0.001 (2)	0.0046 (17)
S1	0.0193 (8)	0.0210 (7)	0.0149 (7)	0.0010 (6)	−0.0015 (5)	0.0006 (6)

Geometric parameters (Å, º)

C1—N2	1.318 (8)	C8—I1	2.150 (6)
C1—N1	1.328 (6)	C8—H8A	0.9900
C1—N3	1.400 (7)	C8—H8B	0.9900
C2—N1	1.475 (6)	C9—C14	1.394 (9)
C2—C8	1.516 (9)	C9—C10	1.402 (8)
C2—C3	1.517 (9)	C9—S1	1.747 (6)
C2—H2	1.0000	C10—C11	1.389 (9)
C3—C4	1.511 (10)	C10—H10	0.9500
C3—H3A	0.9900	C11—C12	1.391 (9)
C3—H3B	0.9900	C11—H11	0.9500
C4—N2	1.479 (8)	C12—C13	1.405 (10)
C4—H4A	0.9900	C12—C15	1.506 (9)
C4—H4B	0.9900	C13—C14	1.386 (10)

C5—N2	1.469 (8)	C13—H13	0.9500
C5—C6	1.507 (9)	C14—H14	0.9500
C5—H5A	0.9900	C15—H15A	0.9800
C5—H5B	0.9900	C15—H15B	0.9800
C6—C7	1.510 (9)	C15—H15C	0.9800
C6—H6A	0.9900	N1—H1	0.8800
C6—H6B	0.9900	N3—S1	1.685 (5)
C7—N3	1.484 (8)	O1—S1	1.433 (4)
C7—H7A	0.9900	O2—S1	1.429 (4)
C7—H7B	0.9900		

N2—C1—N1	121.3 (5)	C2—C8—H8B	109.0
N2—C1—N3	119.9 (6)	I1—C8—H8B	109.0
N1—C1—N3	118.8 (5)	H8A—C8—H8B	107.8
N1—C2—C8	110.4 (5)	C14—C9—C10	121.4 (6)
N1—C2—C3	110.1 (5)	C14—C9—S1	120.6 (5)
C8—C2—C3	110.5 (5)	C10—C9—S1	118.0 (5)
N1—C2—H2	108.6	C11—C10—C9	118.1 (6)
C8—C2—H2	108.6	C11—C10—H10	120.9
C3—C2—H2	108.6	C9—C10—H10	120.9
C4—C3—C2	110.1 (5)	C10—C11—C12	121.6 (6)
C4—C3—H3A	109.6	C10—C11—H11	119.2
C2—C3—H3A	109.6	C12—C11—H11	119.2
C4—C3—H3B	109.6	C11—C12—C13	119.1 (6)
C2—C3—H3B	109.6	C11—C12—C15	121.1 (7)
H3A—C3—H3B	108.2	C13—C12—C15	119.8 (6)
N2—C4—C3	110.3 (5)	C14—C13—C12	120.4 (6)
N2—C4—H4A	109.6	C14—C13—H13	119.8
C3—C4—H4A	109.6	C12—C13—H13	119.8
N2—C4—H4B	109.6	C13—C14—C9	119.4 (6)
C3—C4—H4B	109.6	C13—C14—H14	120.3
H4A—C4—H4B	108.1	C9—C14—H14	120.3
N2—C5—C6	112.0 (5)	C12—C15—H15A	109.5
N2—C5—H5A	109.2	C12—C15—H15B	109.5
C6—C5—H5A	109.2	H15A—C15—H15B	109.5
N2—C5—H5B	109.2	C12—C15—H15C	109.5
C6—C5—H5B	109.2	H15A—C15—H15C	109.5
H5A—C5—H5B	107.9	H15B—C15—H15C	109.5
C5—C6—C7	108.0 (5)	C1—N1—C2	125.2 (4)
C5—C6—H6A	110.1	C1—N1—H1	117.4
C7—C6—H6A	110.1	C2—N1—H1	117.4
C5—C6—H6B	110.1	C1—N2—C5	124.4 (6)
C7—C6—H6B	110.1	C1—N2—C4	119.0 (5)
H6A—C6—H6B	108.4	C5—N2—C4	116.0 (5)
N3—C7—C6	108.3 (5)	C1—N3—C7	117.3 (5)
N3—C7—H7A	110.0	C1—N3—S1	121.8 (4)
C6—C7—H7A	110.0	C7—N3—S1	119.0 (4)
N3—C7—H7B	110.0	O2—S1—O1	119.4 (3)

C6—C7—H7B	110.0	O2—S1—N3	104.7 (3)
H7A—C7—H7B	108.4	O1—S1—N3	107.6 (3)
C2—C8—I1	112.8 (4)	O2—S1—C9	109.9 (3)
C2—C8—H8A	109.0	O1—S1—C9	108.6 (3)
I1—C8—H8A	109.0	N3—S1—C9	105.7 (3)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···I1	0.88	2.87	3.3359 (4)	115
N1—H1···O1	0.88	2.00	2.737 (5)	141
C7—H7*A*···O2	0.99	2.29	2.807 (8)	112
C14—H14···O1	0.95	2.57	2.934 (8)	103
C6—H6*B*···O1^{i}	0.99	2.58	3.425 (8)	143
C7—H7*A*···O2^{ii}	0.99	2.56	3.391 (8)	141
C10—H10···O2^{ii}	0.95	2.49	3.441 (7)	174

Symmetry codes: (i) $x-1, y, z$; (ii) $x-1/2, -y+1/2, -z+1$.