Acta Crystallographica Section E
Structure Reports
Online

ISSN 1600-5368

2-Methylsulfanyl-1*H*-perimidin-3-ium iodide

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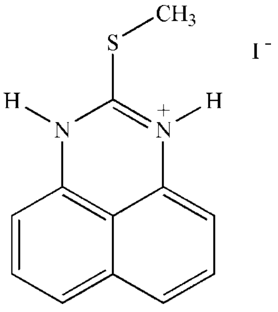
Received 19 July 2012; accepted 26 July 2012

Key indicators: single-crystal X-ray study; *T* = 298 K; mean σ(C–C) = 0.007 Å; *R* factor = 0.044; *wR* factor = 0.094; data-to-parameter ratio = 21.5.

In the structure of the title salt $C_{12}H_{11}N_2S^+\cdot I^-$, the methylsulfanyl group of the cation is nearly coplanar with the perimidine rings, as indicated by the C—S—C—N torsion angles of 2.9 (5) and −177.2 (3)°, respectively. The (S)C—N bond lengths in the heterocyclic ring are approximately equal [1.325 (5) and 1.326 (6) Å] suggesting a degree of delocalization. In the crystal, cations and anions are linked *via* two discrete N—H···I hydrogen bonds, forming chains along the *b* axis.

Related literature

For synthetic details and applications, see: Liu & Chen (1984); Herbert *et al.* (1987). For the NMR spectra, see Woodgate *et al.* (1988). For related structures, see: Molčanov *et al.* (2012); Wang (2012); Tiritiris & Kantlehner (2012).



Experimental

Crystal data

$C_{12}H_{11}N_2S^+\cdot I^-$
M_r = 342.19
Monoclinic, $P2_1/c$
a = 7.0107 (14) Å
b = 8.8968 (18) Å
c = 19.520 (4) Å
β = 95.90 (3)°
V = 1211.1 (4) Å^3
Z = 4
Mo *K*α radiation
μ = 2.79 mm^{-1}
T = 298 K
0.20 × 0.17 × 0.10 mm

Data collection

Stoe IPDS 2T diffractometer
Absorption correction: numerical (*X-RED32*; Stoe & Cie, 2005)
T_{min} = 0.605, T_{max} = 0.768
8695 measured reflections
3266 independent reflections
2151 reflections with $I > 2\sigma(I)$
R_{int} = 0.053

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.094
S = 1.11
3266 reflections
152 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.98 e Å^{-3}
$\Delta\rho_{min}$ = −0.47 e Å^{-3}

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2···I1^{i}	0.81 (2)	2.72 (3)	3.500 (4)	161 (5)
N1—H1···I1	0.85 (6)	2.98 (6)	3.813 (4)	169 (5)

Symmetry code: (i) *x*, *y* + 1, *z*.

Data collection: *X-AREA* (Stoe & Cie, 2005); cell refinement: *X-AREA*; data reduction: *X-RED32* (Stoe & Cie, 2005); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

The author thanks the Islamic Azad University-Falavarjan Branch for financial support. He also wishes to thank Professor Mehdi Bakavoli, Ferdowsi University of Mashhad, and Dr Behrouz Notash, Shahid Beheshti University, for their helpful assistance.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SJ5260).

References

Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
Herbert, J. M., Woodgate, P. D. & Denny, W. A. (1987). *J. Med. Chem.* **30**, 2081–2086.
Liu, K.-C. & Chen, H.-H. (1984). *J. Heterocycl. Chem.* **21**, 911–912.
Molčanov, K., Stolić, I., Kojić-Prodić, B., Kovačević, G. & Bajić, M. (2012). *Acta Cryst.* E**68**, o1360.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Stoe & Cie (2005). *X-AREA* and *X-RED32*. Stoe & Cie, Darmstadt, Germany.
Tiritiris, I. & Kantlehner, W. (2012). *Acta Cryst.* E**68**, o1812.
Wang, Y. (2012). *Acta Cryst.* E**68**, o1619.
Woodgate, P. D., Herbert, J. M. & Denny, W. A. (1988). *Mag. Res. Chem.* **26**, 191–196.

supporting information

Acta Cryst. (2012). E**68**, o2605 [doi:10.1107/S1600536812033697]

2-Methylsulfanyl-1*H*-perimidin-3-ium iodide

Mohammad Hassan Ghorbani

S1. Comment

The title compound is used in the synthesis of some potentially active antitumor agents (Herbert *et al.*, 1987) and heterocyclic compounds (Liu & Chen, 1984). So far, the structure of this compound and its neutral form has been studied using ^{13}C and ^{1}H NMR spectroscopy (Woodgate *et al.*, 1988). Herein, the crystal structure of this salt is investigated using X-ray crystallography.

In the structure of the 2-methylsulfanylperimidinium cation, the methylsulfanyl group is nearly coplanar with perimidine rings [the torsion angles N1—C2—S—C1 and N2—C2—S—C1 are 2.9 (5)° and -177.2 (3)°, respectively]. Because of conjugation between the lone pair electrons of the S atom and the amidinum moiety (HN—C=NH$^+$) in the cation, the C2—S bond length [1.730 (5) Å] is shorter than C1—S [1.789 (5) Å]. Also, like other amidinium cations (Molčanov *et al.*, 2012; Wang, 2012; Tiritiris & Kantlehner, 2012), the C—N bond lengths in the cation are approximately equal [the bond lengths of C2—N1 and C2—N2 are 1.325 (5) Å and 1.325 (6) Å, respectively].

In the crystal lattice, the cations and anions are linked together *via* two different N—H···I hydrogen bonds, in which every iodide anion act as a bridge between two 2-methylsulfanylperimidinium cations.

S2. Experimental

The title salt was prepared by a literature method (Liu & Chen, 1984; Herbert *et al.*, 1987). Suitable single crystals for X-ray analysis were obtained from ethanol solution at room temperature.

S3. Refinement

All hydrogen atoms bound to carbon were positioned geometrically with C—H distances = 0.93–0.96 Å and included in a riding model approximation with $U_{\rm iso}$(H) = 1.2 or 1.5$U_{\rm eq}$(C). The N–H hydrogen atoms were located in a difference Fourier map and refined freely.

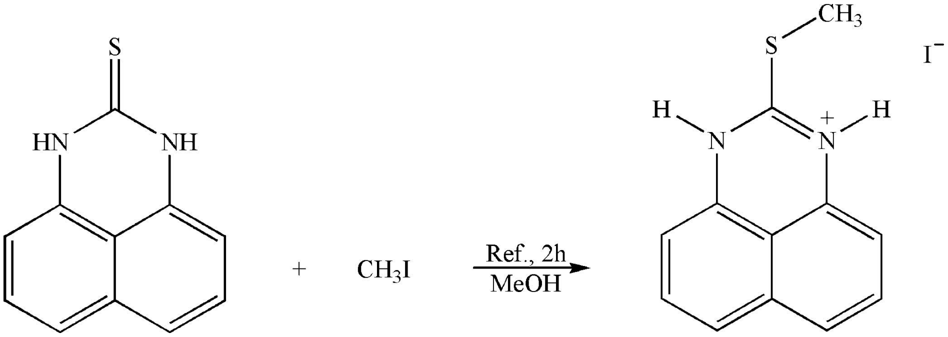


Figure 1

Preparation of the title compound from the reaction of perimidine-2-thione with methyl iodide under reflux conditions.

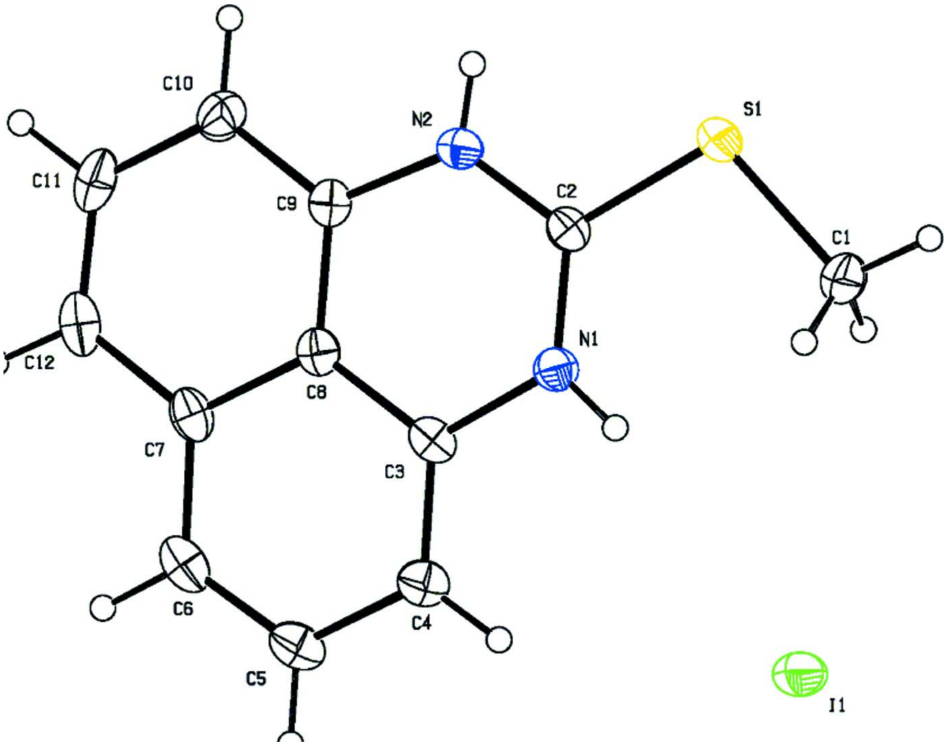


Figure 2

The molecular structure of the title compound, showing 50% probability displacement ellipsoids.

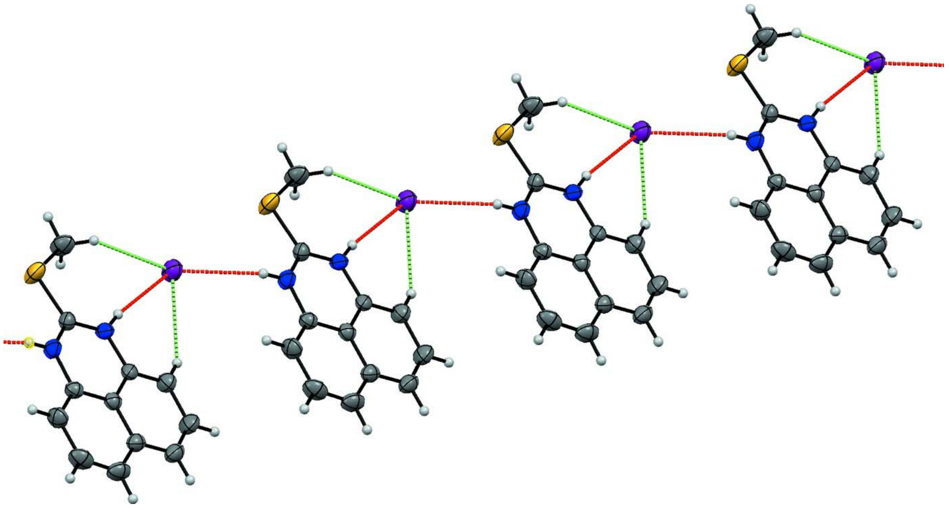


Figure 3

Chains of molecules along the *b* axis.

2-Methylsulfanyl-1*H*-perimidin-3-ium iodide

Crystal data

$C_{12}H_{11}N_2S^+\cdot I^-$

$M_r = 342.19$

Monoclinic, $P2_1/c$

Hall symbol: -P 2ybc

$a = 7.0107\ (14)$ Å

$b = 8.8968\ (18)$ Å

$c = 19.520\ (4)$ Å

$\beta = 95.90\ (3)°$

V = 1211.1 (4) Å^3
Z = 4
F(000) = 664
D_x = 1.877 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 3266 reflections
θ = 2.1–29.2°
μ = 2.79 mm^{-1}
T = 298 K
Plate, green
0.20 × 0.17 × 0.10 mm

Data collection

Stoe IPDS 2T diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 0.15 pixels mm^{-1}
rotation method scans
Absorption correction: numerical (*X-RED32*; Stoe & Cie, 2005)
T_{min} = 0.605, T_{max} = 0.768
8695 measured reflections
3266 independent reflections
2151 reflections with $I > 2\sigma(I)$
R_{int} = 0.053
θ_{max} = 29.2°, θ_{min} = 2.1°
$h = -9 \rightarrow 9$
$k = -12 \rightarrow 10$
$l = -26 \rightarrow 23$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.094
S = 1.11
3266 reflections
152 parameters
1 restraint
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
$w = 1/[\sigma^2(F_o^2) + (0.0351P)^2 + 0.0203P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.98 e Å^{-3}
$\Delta\rho_{min}$ = −0.47 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 (Å^2)

	x	y	z	U_{iso}*/U_{eq}
I1	0.23717 (5)	0.01191 (3)	0.335840 (18)	0.04724 (12)
S1	0.3337 (2)	0.57350 (14)	0.29413 (7)	0.0475 (3)
N1	0.2710 (5)	0.4113 (4)	0.4069 (2)	0.0308 (8)
N2	0.3149 (6)	0.6660 (4)	0.4194 (2)	0.0341 (9)
C1	0.2992 (8)	0.3909 (6)	0.2564 (3)	0.0481 (13)
H1A	0.3861	0.3211	0.2806	0.072*
H1B	0.3232	0.3950	0.2089	0.072*
H1C	0.1696	0.3588	0.2595	0.072*
C2	0.3031 (6)	0.5445 (5)	0.3799 (2)	0.0304 (9)

C3	0.2384 (6)	0.3919 (5)	0.4769 (2)	0.0295 (9)
C4	0.1976 (7)	0.2561 (5)	0.5032 (3)	0.0376 (11)
H4	0.1910	0.1707	0.4756	0.045*
C5	0.1651 (7)	0.2459 (6)	0.5733 (3)	0.0432 (12)
H5	0.1363	0.1531	0.5916	0.052*
C6	0.1753 (7)	0.3702 (6)	0.6146 (3)	0.0414 (12)
H6	0.1535	0.3607	0.6606	0.050*
C7	0.2184 (6)	0.5129 (5)	0.5885 (2)	0.0344 (9)
C8	0.2492 (5)	0.5236 (5)	0.5181 (2)	0.0295 (8)
C9	0.2892 (6)	0.6642 (5)	0.4898 (2)	0.0299 (9)
C10	0.3011 (7)	0.7923 (5)	0.5285 (3)	0.0406 (11)
H10	0.3295	0.8844	0.5095	0.049*
C11	0.2689 (7)	0.7801 (6)	0.5986 (3)	0.0451 (13)
H11	0.2756	0.8664	0.6257	0.054*
C12	0.2284 (7)	0.6463 (6)	0.6281 (3)	0.0434 (12)
H12	0.2074	0.6427	0.6744	0.052*
H1	0.274 (8)	0.328 (7)	0.387 (3)	0.052*
H2	0.327 (8)	0.746 (4)	0.401 (3)	0.052*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
I1	0.0704 (2)	0.02971 (16)	0.04296 (19)	−0.00194 (17)	0.01260 (14)	0.00215 (16)
S1	0.0825 (10)	0.0347 (6)	0.0260 (6)	−0.0092 (6)	0.0095 (6)	0.0024 (5)
N1	0.036 (2)	0.0292 (18)	0.029 (2)	−0.0009 (15)	0.0099 (16)	−0.0010 (16)
N2	0.046 (2)	0.0261 (18)	0.031 (2)	−0.0010 (16)	0.0056 (18)	0.0030 (15)
C1	0.074 (4)	0.037 (3)	0.034 (3)	−0.008 (2)	0.011 (3)	−0.008 (2)
C2	0.031 (2)	0.032 (2)	0.028 (2)	0.0019 (17)	0.0010 (18)	0.0031 (17)
C3	0.025 (2)	0.035 (2)	0.030 (2)	0.0053 (17)	0.0057 (18)	0.0056 (18)
C4	0.038 (3)	0.036 (2)	0.039 (3)	0.000 (2)	0.007 (2)	0.004 (2)
C5	0.047 (3)	0.044 (3)	0.040 (3)	−0.001 (2)	0.011 (2)	0.011 (2)
C6	0.038 (3)	0.056 (3)	0.030 (3)	0.005 (2)	0.006 (2)	0.009 (2)
C7	0.0287 (19)	0.047 (3)	0.027 (2)	0.006 (2)	0.0014 (16)	0.002 (2)
C8	0.0248 (18)	0.034 (2)	0.029 (2)	0.0038 (17)	0.0010 (16)	−0.0004 (19)
C9	0.027 (2)	0.033 (2)	0.029 (2)	0.0021 (17)	0.0024 (18)	−0.0025 (18)
C10	0.047 (3)	0.033 (2)	0.040 (3)	0.003 (2)	0.003 (2)	−0.005 (2)
C11	0.046 (3)	0.048 (3)	0.040 (3)	0.003 (2)	−0.002 (2)	−0.019 (2)
C12	0.041 (3)	0.062 (3)	0.026 (3)	0.004 (2)	0.001 (2)	−0.007 (2)

Geometric parameters (Å, º)

S1—C2	1.730 (5)	C4—H4	0.9300
S1—C1	1.789 (5)	C5—C6	1.366 (7)
N1—C2	1.325 (5)	C5—H5	0.9300
N1—C3	1.418 (6)	C6—C7	1.413 (7)
N1—H1	0.85 (6)	C6—H6	0.9300
N2—C2	1.325 (6)	C7—C12	1.415 (7)
N2—C9	1.404 (6)	C7—C8	1.415 (6)

N2—H2	0.811 (19)	C8—C9	1.408 (6)
C1—H1A	0.9600	C9—C10	1.366 (6)
C1—H1B	0.9600	C10—C11	1.414 (7)
C1—H1C	0.9600	C10—H10	0.9300
C3—C4	1.356 (6)	C11—C12	1.365 (8)
C3—C8	1.419 (6)	C11—H11	0.9300
C4—C5	1.412 (7)	C12—H12	0.9300

C2—S1—C1	103.8 (2)	C6—C5—H5	119.5
C2—N1—C3	122.9 (4)	C4—C5—H5	119.5
C2—N1—H1	126 (4)	C5—C6—C7	121.0 (4)
C3—N1—H1	111 (4)	C5—C6—H6	119.5
C2—N2—C9	123.6 (4)	C7—C6—H6	119.5
C2—N2—H2	118 (4)	C6—C7—C12	123.8 (4)
C9—N2—H2	119 (4)	C6—C7—C8	118.0 (4)
S1—C1—H1A	109.5	C12—C7—C8	118.1 (4)
S1—C1—H1B	109.5	C9—C8—C7	119.8 (4)
H1A—C1—H1B	109.5	C9—C8—C3	120.8 (4)
S1—C1—H1C	109.5	C7—C8—C3	119.4 (4)
H1A—C1—H1C	109.5	C10—C9—N2	121.7 (4)
H1B—C1—H1C	109.5	C10—C9—C8	121.8 (4)
N1—C2—N2	120.1 (4)	N2—C9—C8	116.5 (4)
N1—C2—S1	124.2 (3)	C9—C10—C11	117.7 (5)
N2—C2—S1	115.8 (3)	C9—C10—H10	121.1
C4—C3—N1	122.4 (4)	C11—C10—H10	121.1
C4—C3—C8	121.4 (4)	C12—C11—C10	122.4 (5)
N1—C3—C8	116.2 (4)	C12—C11—H11	118.8
C3—C4—C5	119.1 (5)	C10—C11—H11	118.8
C3—C4—H4	120.4	C11—C12—C7	120.1 (5)
C5—C4—H4	120.4	C11—C12—H12	119.9
C6—C5—C4	121.0 (5)	C7—C12—H12	119.9

C3—N1—C2—N2	3.3 (7)	C12—C7—C8—C3	−179.5 (4)
C3—N1—C2—S1	−177.2 (3)	C4—C3—C8—C9	−179.2 (4)
C9—N2—C2—N1	−2.1 (7)	N1—C3—C8—C9	0.3 (6)
C9—N2—C2—S1	178.4 (3)	C4—C3—C8—C7	0.6 (6)
C1—S1—C2—N1	2.9 (5)	N1—C3—C8—C7	−179.9 (4)
C1—S1—C2—N2	−177.7 (4)	C2—N2—C9—C10	−179.5 (4)
C2—N1—C3—C4	177.1 (4)	C2—N2—C9—C8	0.0 (6)
C2—N1—C3—C8	−2.4 (6)	C7—C8—C9—C10	0.5 (6)
N1—C3—C4—C5	−179.4 (4)	C3—C8—C9—C10	−179.7 (4)
C8—C3—C4—C5	0.0 (7)	C7—C8—C9—N2	−179.0 (4)
C3—C4—C5—C6	−0.4 (8)	C3—C8—C9—N2	0.8 (6)
C4—C5—C6—C7	0.1 (8)	N2—C9—C10—C11	178.6 (5)
C5—C6—C7—C12	179.1 (5)	C8—C9—C10—C11	−0.9 (7)
C5—C6—C7—C8	0.5 (7)	C9—C10—C11—C12	0.5 (8)
C6—C7—C8—C9	178.9 (4)	C10—C11—C12—C7	0.3 (8)
C12—C7—C8—C9	0.3 (6)	C6—C7—C12—C11	−179.3 (5)

C6—C7—C8—C3	−0.8 (6)	C8—C7—C12—C11	−0.7 (7)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2···I1^{i}	0.81 (2)	2.72 (3)	3.500 (4)	161 (5)
N1—H1···I1	0.85 (6)	2.98 (6)	3.813 (4)	169 (5)

Symmetry code: (i) *x*, *y*+1, *z*.