# organic compounds

# [1-(1-Adamantylamino)ethylidene]-oxonium methanesulfonate

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In the title salt, $C_{12}H_{20}NO^+ \cdot CH_3SO_3^-$, the [1-(1-adamantyl-amino)ethylidene]oxonium cations and methanesulfonate anions are linked into chains along the *a* axis *via* O—H···O and N—H···O hydrogen bonds. All non-H atoms of the acetamido group are essentially planar, with a maximum deviation of 0.0085 (12) Å. In comparison with related structures, the carbonyl C=O bond is slightly elongated [1.249 (2) Å], whereas the amide C—N bond is shortened [1.292 (2) Å].

## Related literature

For previously published structures of *N*-(1-adamantyl)-acetamide, see: Pröhl *et al.* (1997); Kashino *et al.* (1998); Mizoguchi *et al.* (1997). For the preparation of *N*-(1-adamantyl)acetamide, see: Bach *et al.* (1979, 1980); Gerzon *et al.* (1963); Stetter *et al.* (1959, 1960). For the biological activity of related adamantane derivatives, see: Davies *et al.* (1964); Aldrich *et al.* (1971).
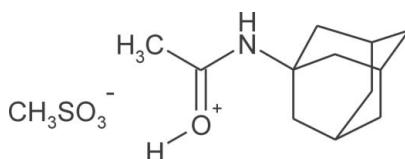


## Experimental

### Crystal data

| | |
|---|---|
| $C_{12}H_{20}NO^+ \cdot CH_3SO_3^-$ | $b$ = 11.2625 (6) Å |
| $M_r$ = 289.38 | $c$ = 19.0037 (10) Å |
| Orthorhombic, *Pbca* | $V$ = 2779.1 (3) Å$^3$ |
| $a$ = 12.9848 (7) Å | $Z$ = 8 |
| | Mo $K\alpha$ radiation |
| | $\mu$ = 0.24 mm$^{-1}$ |

| | |
|---|---|
| | $T$ = 120 K |
| | 0.40 × 0.40 × 0.35 mm |

### Data collection

| | |
|---|---|
| Kuma KM-4 CCD diffractometer | 19453 measured reflections |
| Absorption correction: multi-scan (*Xcalibur*; Oxford Diffraction, 2006) | 2454 independent reflections |
| | 2000 reflections with $I > 2\sigma(I)$ |
| $T_{min}$ = 0.824, $T_{max}$ = 0.914 | $R_{int}$ = 0.017 |

### Refinement

| | |
|---|---|
| $R[F^2 > 2\sigma(F^2)]$ = 0.034 | H atoms treated by a mixture of independent and constrained refinement |
| $wR(F^2)$ = 0.102 | |
| $S$ = 1.09 | |
| 2454 reflections | $\Delta\rho_{max}$ = 0.30 e Å$^{-3}$ |
| 180 parameters | $\Delta\rho_{min}$ = −0.47 e Å$^{-3}$ |

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| N1—H1···O3$^i$ | 0.87 (2) | 1.98 (2) | 2.838 (2) | 170.1 (19) |
| O1—H2···O2 | 0.98 (2) | 1.49 (2) | 2.4632 (18) | 173 (2) |

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

Data collection: *Xcalibur* (Oxford Diffraction, 2006); cell refinement: *Xcalibur* (Oxford Diffraction, 2006); data reduction: *Xcalibur* (Oxford Diffraction, 2006); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

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# supporting information

## [1-(1-Adamantylamino)ethylidene]oxonium methanesulfonate

### Robert Vícha, Marek Nečas, Zuzana Kozubková and Milan Potáček

### S1. Comment

Since 1964, 1-aminoadamantane and related compounds have seen extensive examination due to their antiviral activity (Davies *et al.*, 1964; Aldrich *et al.*, 1971). The consecution of adamantane bromination, reaction with acetonitrile and final hydrolysis of *N*-(1-adamantyl)acetamide provides a viable synthetic method for 1-aminoadamantane production. The synthesis of *N*-(1-adamantyl)acetamide *via* nucleophilic substitution from varied bridgehead-substituted derivatives was previously described. For this purpose 1-iodoadamantane (Bach *et al.*, 1980), 1-bromoadamantane (Stetter *et al.*, 1960), 1-chloroadamantane (Gerzon *et al.*, 1963), 1-alkoxyadamantane (Bach *et al.*, 1979; Bach *et al.*, 1980) or adamantan-1-ol (Stetter *et al.*, 1959) were used as starting material. The title salt was prepared by replacement of a good-leaving group in 1-adamantyl methanesulfonate with acetonitrile.

In the structure of title salt (Fig. 1), the *O*-protonated *N*-(1-adamantyl)acetamide and methansulfonate are linked alternately into chains parallel to the *a* axis *via* O1–H2···O2 and N1–H1···O3 hydrogen bonds (Table 1, Fig. 2). All non-hydrogen atoms of the acetamido group (C11, C12, N1, O1) and C1 lie in plane with the maximum deviation from the best plane being 0.0085 (12) Å for atom N1. The distance of the H1 and H2 from the best plane (C1, C11, C12, N1, O1) is 0.004 (19) and 0.11 (2) Å respectively. In comparison with previously published structures of *N*-(1-adamantyl)acetamide (Pröhl *et al.*, 1997; Kashino *et al.*, 1998 and Mizoguchi *et al.*, 1997), the length of N1–C11 is slightly shorter, being 1.292 (2) Å [published 1.323 (5)–1.345 (2) Å] and C11–O1 is slightly longer at 1.294 (2) Å [published 1.230 (5)–1.237 (4) Å]. This may be attributed to enhanced electron withdrawing effect of the protonated O1.

### S2. Experimental

1-Adamantyl methanesulfonate (500 mg, 2.17 mmol) was stirred in 20 ml of dry acetonitrile at room temperature for 1 h. After this period, the solution was allowed to stand at room temperature for several days and growth of crystals was observed. The solid was filtered off with suction and mother liquor was evaporated to obtain a second crop of title compound as a colourless powder. The combined yield of the title salt was 583 mg (93%). ^{1}H NMR spectra were similar to those obtained for equimolar mixture of separately prepared *N*-(1-adamantyl)acetamide and methanesulfonic acid.

### S3. Refinement

H atoms were found in difference Fourier maps. Those attached to N and O were refined while those attached to C were placed in idealized positions with constrained distances of 0.98 Å (RCH$_3$) and 0.99 Å (R$_2$CH$_2$). U$_{iso}$(H) values were set to either 1.2U$_{eq}$ or 1.5U$_{eq}$ (RCH$_3$, OH) of the attached atom.
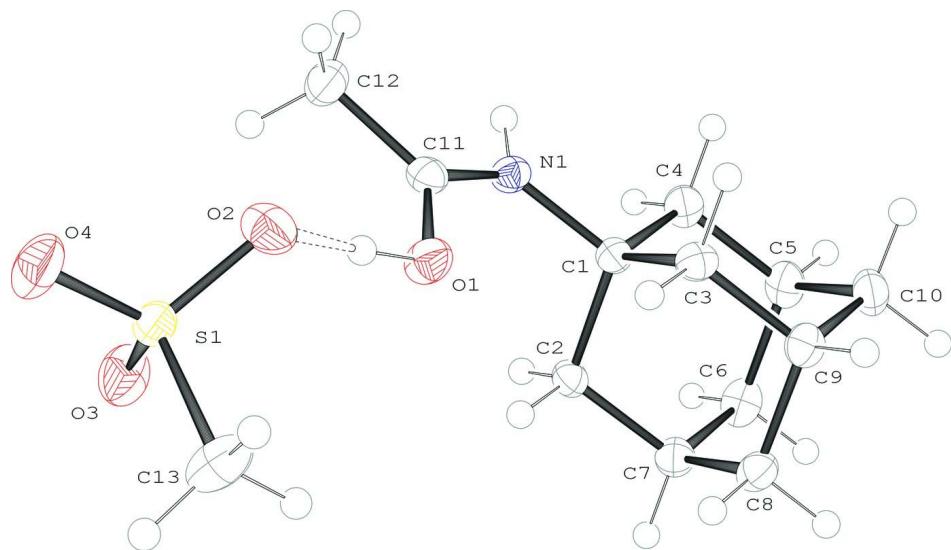
**Figure 1**

*ORTEP* of the asymmetric unit with atoms represented as 50% probability ellipsoids. Hydrogen bonding is indicated by dashed lines.
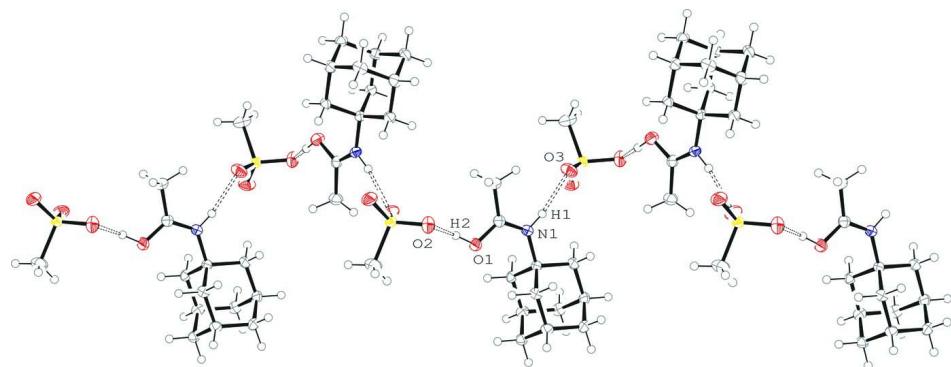


**Figure 2**

Hydrogen bonded chains of alternating protonated *N*-(1-adamantyl)acetamide and methansulfonate parallel to the *a* axis.

## [1-(1-Adamantylamino)ethylidene]oxonium methanesulfonate

*Crystal data*
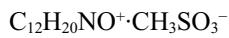
| | |
|---|---|
| $C_{12}H_{20}NO^+ \cdot CH_3SO_3^-$ | $D_x = 1.383$ Mg m$^{-3}$ |
| $M_r = 289.38$ | Melting point: 445 K |
| Orthorhombic, *Pbca* | Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å |
| Hall symbol: -P 2ac 2ab | Cell parameters from 2454 reflections |
| $a = 12.9848$ (7) Å | $\theta = 3.1–25.0°$ |
| $b = 11.2625$ (6) Å | $\mu = 0.24$ mm$^{-1}$ |
| $c = 19.0037$ (10) Å | $T = 120$ K |
| $V = 2779.1$ (3) Å$^3$ | Block, colourless |
| $Z = 8$ | $0.40 \times 0.40 \times 0.35$ mm |
| $F(000) = 1248$ | |

*Data collection*

| | |
|---|---|
| Kuma KM-4 CCD diffractometer | 19453 measured reflections |
| Radiation source: fine-focus sealed tube | 2454 independent reflections |
| Graphite monochromator | 2000 reflections with $I > 2\sigma(I)$ |
| Detector resolution: 0.06 pixels mm$^{-1}$ | $R_{int} = 0.017$ |
| $\omega$ scans | $\theta_{max} = 25.0°$, $\theta_{min} = 3.1°$ |
| Absorption correction: multi-scan (Xcalibur; Oxford Diffraction, 2006) | $h = -15 \rightarrow 14$ |
| | $k = -13 \rightarrow 13$ |
| $T_{min} = 0.824$, $T_{max} = 0.914$ | $l = -21 \rightarrow 22$ |

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | Secondary atom site location: difference Fourier map |
| Least-squares matrix: full | |
| $R[F^2 > 2\sigma(F^2)] = 0.034$ | Hydrogen site location: inferred from neighbouring sites |
| $wR(F^2) = 0.102$ | |
| $S = 1.09$ | H atoms treated by a mixture of independent and constrained refinement |
| 2454 reflections | |
| 180 parameters | $w = 1/[\sigma^2(F_o^2) + (0.054P)^2 + 1.529P]$ |
| 0 restraints | where $P = (F_o^2 + 2F_c^2)/3$ |
| Primary atom site location: structure-invariant direct methods | $(\Delta/\sigma)_{max} = 0.001$ |
| | $\Delta\rho_{max} = 0.30$ 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 $F^2 > 2\sigma(F^2)$ 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$^2$)*

| | $x$ | $y$ | $z$ | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| C1 | −0.09044 (13) | 0.28700 (14) | 0.34406 (9) | 0.0181 (4) |
| C2 | −0.00380 (13) | 0.22857 (15) | 0.30176 (9) | 0.0195 (4) |
| H2A | 0.0621 | 0.2698 | 0.3111 | 0.023* |
| H2B | 0.0038 | 0.1445 | 0.3162 | 0.023* |
| C3 | −0.10319 (14) | 0.41701 (15) | 0.32215 (9) | 0.0201 (4) |
| H3A | −0.1594 | 0.4541 | 0.3496 | 0.024* |
| H3B | −0.0388 | 0.4613 | 0.3317 | 0.024* |
| C4 | −0.19133 (14) | 0.22087 (16) | 0.33012 (9) | 0.0229 (4) |
| H4A | −0.1846 | 0.1370 | 0.3451 | 0.027* |
| H4B | −0.2477 | 0.2576 | 0.3576 | 0.027* |
| C5 | −0.21663 (15) | 0.22635 (17) | 0.25177 (10) | 0.0251 (4) |
| H5 | −0.2824 | 0.1828 | 0.2427 | 0.030* |
| C6 | −0.13002 (15) | 0.16913 (16) | 0.20928 (10) | 0.0262 (4) |
| H6A | −0.1467 | 0.1724 | 0.1585 | 0.031* |
| H6B | −0.1225 | 0.0847 | 0.2230 | 0.031* |

| | | | | |
|---|---|---|---|---|
| C7 | −0.02922 (14) | 0.23530 (16) | 0.22322 (10) | 0.0225 (4) |
| H7 | 0.0276 | 0.1979 | 0.1954 | 0.027* |
| C8 | −0.04113 (14) | 0.36523 (15) | 0.20149 (9) | 0.0224 (4) |
| H8A | −0.0567 | 0.3702 | 0.1506 | 0.027* |
| H8B | 0.0240 | 0.4084 | 0.2103 | 0.027* |
| C9 | −0.12838 (13) | 0.42199 (15) | 0.24371 (9) | 0.0213 (4) |
| H9 | −0.1362 | 0.5068 | 0.2291 | 0.026* |
| C10 | −0.22900 (14) | 0.35595 (17) | 0.22953 (10) | 0.0256 (4) |
| H10A | −0.2461 | 0.3603 | 0.1788 | 0.031* |
| H10B | −0.2858 | 0.3932 | 0.2564 | 0.031* |
| C11 | 0.00322 (13) | 0.32640 (15) | 0.45588 (9) | 0.0211 (4) |
| C12 | 0.01287 (15) | 0.30548 (18) | 0.53284 (10) | 0.0271 (4) |
| H12A | 0.0762 | 0.2609 | 0.5424 | 0.041* |
| H12B | −0.0467 | 0.2599 | 0.5495 | 0.041* |
| H12C | 0.0155 | 0.3819 | 0.5575 | 0.041* |
| N1 | −0.06903 (11) | 0.27539 (13) | 0.42026 (8) | 0.0195 (3) |
| H1 | −0.1102 (15) | 0.2308 (18) | 0.4446 (11) | 0.023* |
| O1 | 0.06855 (10) | 0.39456 (12) | 0.42407 (6) | 0.0268 (3) |
| H2 | 0.1158 (17) | 0.436 (2) | 0.4560 (12) | 0.040* |
| S1 | 0.29158 (4) | 0.49446 (4) | 0.50332 (2) | 0.02087 (16) |
| O2 | 0.17962 (12) | 0.51334 (12) | 0.50208 (7) | 0.0302 (3) |
| O3 | 0.31512 (12) | 0.37147 (12) | 0.48779 (7) | 0.0342 (4) |
| O4 | 0.33796 (11) | 0.54034 (13) | 0.56618 (7) | 0.0357 (4) |
| C13 | 0.33885 (17) | 0.57840 (19) | 0.43323 (10) | 0.0356 (5) |
| H13A | 0.3191 | 0.6617 | 0.4396 | 0.053* |
| H13B | 0.3099 | 0.5485 | 0.3890 | 0.053* |
| H13C | 0.4141 | 0.5721 | 0.4318 | 0.053* |

*Atomic displacement parameters (Å$^2$)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| C1 | 0.0179 (9) | 0.0183 (8) | 0.0179 (9) | 0.0002 (7) | 0.0006 (7) | 0.0014 (7) |
| C2 | 0.0182 (9) | 0.0204 (9) | 0.0201 (9) | 0.0034 (7) | −0.0005 (7) | −0.0017 (7) |
| C3 | 0.0235 (9) | 0.0163 (8) | 0.0206 (9) | 0.0009 (7) | 0.0028 (7) | −0.0011 (7) |
| C4 | 0.0213 (9) | 0.0212 (9) | 0.0261 (10) | −0.0034 (7) | −0.0001 (7) | 0.0048 (7) |
| C5 | 0.0213 (9) | 0.0250 (10) | 0.0289 (10) | −0.0074 (8) | −0.0056 (7) | 0.0029 (8) |
| C6 | 0.0341 (11) | 0.0185 (9) | 0.0259 (10) | −0.0019 (8) | −0.0061 (8) | −0.0019 (7) |
| C7 | 0.0250 (9) | 0.0230 (9) | 0.0195 (9) | 0.0043 (8) | −0.0002 (7) | −0.0037 (7) |
| C8 | 0.0238 (9) | 0.0260 (9) | 0.0176 (9) | −0.0027 (8) | −0.0004 (7) | 0.0002 (7) |
| C9 | 0.0254 (9) | 0.0150 (8) | 0.0236 (9) | 0.0005 (7) | −0.0008 (7) | 0.0027 (7) |
| C10 | 0.0209 (9) | 0.0281 (10) | 0.0277 (10) | 0.0022 (8) | −0.0044 (8) | 0.0042 (8) |
| C11 | 0.0207 (9) | 0.0209 (9) | 0.0216 (9) | 0.0041 (7) | 0.0029 (7) | −0.0015 (7) |
| C12 | 0.0311 (11) | 0.0299 (10) | 0.0202 (10) | 0.0010 (8) | −0.0021 (8) | 0.0001 (8) |
| N1 | 0.0214 (8) | 0.0187 (7) | 0.0183 (8) | −0.0001 (6) | 0.0022 (6) | 0.0015 (6) |
| O1 | 0.0257 (7) | 0.0330 (7) | 0.0216 (7) | −0.0080 (6) | −0.0009 (5) | −0.0008 (6) |
| S1 | 0.0221 (3) | 0.0229 (3) | 0.0175 (3) | −0.00163 (17) | −0.00125 (16) | 0.00108 (17) |
| O2 | 0.0240 (7) | 0.0328 (8) | 0.0337 (8) | −0.0003 (6) | 0.0006 (5) | −0.0114 (6) |
| O3 | 0.0433 (9) | 0.0270 (8) | 0.0323 (8) | 0.0092 (6) | −0.0058 (6) | 0.0028 (6) |

| | | | | | |
|---|---|---|---|---|---|
| O4 | 0.0406 (8) | 0.0440 (8) | 0.0226 (7) | −0.0109 (7) | −0.0093 (6) | 0.0014 (6) |
| C13 | 0.0489 (13) | 0.0353 (11) | 0.0227 (10) | −0.0117 (10) | 0.0030 (9) | 0.0021 (8) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| C1—N1 | 1.480 (2) | C8—H8A | 0.9900 |
| C1—C4 | 1.530 (2) | C8—H8B | 0.9900 |
| C1—C3 | 1.531 (2) | C9—C10 | 1.527 (2) |
| C1—C2 | 1.531 (2) | C9—H9 | 1.0000 |
| C2—C7 | 1.530 (3) | C10—H10A | 0.9900 |
| C2—H2A | 0.9900 | C10—H10B | 0.9900 |
| C2—H2B | 0.9900 | C11—N1 | 1.292 (2) |
| C3—C9 | 1.527 (2) | C11—O1 | 1.294 (2) |
| C3—H3A | 0.9900 | C11—C12 | 1.487 (3) |
| C3—H3B | 0.9900 | C12—H12A | 0.9800 |
| C4—C5 | 1.526 (3) | C12—H12B | 0.9800 |
| C4—H4A | 0.9900 | C12—H12C | 0.9800 |
| C4—H4B | 0.9900 | N1—H1 | 0.87 (2) |
| C5—C6 | 1.527 (3) | O1—H2 | 0.98 (2) |
| C5—C10 | 1.528 (2) | S1—O4 | 1.4343 (14) |
| C5—H5 | 1.0000 | S1—O3 | 1.4489 (14) |
| C6—C7 | 1.529 (3) | S1—O2 | 1.4695 (15) |
| C6—H6A | 0.9900 | S1—C13 | 1.7448 (19) |
| C6—H6B | 0.9900 | C13—H13A | 0.9800 |
| C7—C8 | 1.528 (2) | C13—H13B | 0.9800 |
| C7—H7 | 1.0000 | C13—H13C | 0.9800 |
| C8—C9 | 1.528 (2) | | |
| | | | |
| N1—C1—C4 | 106.69 (13) | C7—C8—H8A | 109.8 |
| N1—C1—C3 | 111.76 (14) | C9—C8—H8A | 109.8 |
| C4—C1—C3 | 109.01 (14) | C7—C8—H8B | 109.8 |
| N1—C1—C2 | 109.73 (14) | C9—C8—H8B | 109.8 |
| C4—C1—C2 | 109.20 (14) | H8A—C8—H8B | 108.2 |
| C3—C1—C2 | 110.34 (14) | C3—C9—C10 | 109.73 (14) |
| C7—C2—C1 | 109.40 (14) | C3—C9—C8 | 109.77 (14) |
| C7—C2—H2A | 109.8 | C10—C9—C8 | 109.74 (14) |
| C1—C2—H2A | 109.8 | C3—C9—H9 | 109.2 |
| C7—C2—H2B | 109.8 | C10—C9—H9 | 109.2 |
| C1—C2—H2B | 109.8 | C8—C9—H9 | 109.2 |
| H2A—C2—H2B | 108.2 | C9—C10—C5 | 109.05 (14) |
| C9—C3—C1 | 108.89 (14) | C9—C10—H10A | 109.9 |
| C9—C3—H3A | 109.9 | C5—C10—H10A | 109.9 |
| C1—C3—H3A | 109.9 | C9—C10—H10B | 109.9 |
| C9—C3—H3B | 109.9 | C5—C10—H10B | 109.9 |
| C1—C3—H3B | 109.9 | H10A—C10—H10B | 108.3 |
| H3A—C3—H3B | 108.3 | N1—C11—O1 | 119.67 (16) |
| C5—C4—C1 | 109.48 (14) | N1—C11—C12 | 120.42 (16) |
| C5—C4—H4A | 109.8 | O1—C11—C12 | 119.90 (16) |

| | | | |
|---|---|---|---|
| C1—C4—H4A | 109.8 | C11—C12—H12A | 109.5 |
| C5—C4—H4B | 109.8 | C11—C12—H12B | 109.5 |
| C1—C4—H4B | 109.8 | H12A—C12—H12B | 109.5 |
| H4A—C4—H4B | 108.2 | C11—C12—H12C | 109.5 |
| C4—C5—C6 | 109.89 (15) | H12A—C12—H12C | 109.5 |
| C4—C5—C10 | 109.34 (15) | H12B—C12—H12C | 109.5 |
| C6—C5—C10 | 109.53 (15) | C11—N1—C1 | 127.58 (15) |
| C4—C5—H5 | 109.4 | C11—N1—H1 | 115.2 (14) |
| C6—C5—H5 | 109.4 | C1—N1—H1 | 117.2 (14) |
| C10—C5—H5 | 109.4 | C11—O1—H2 | 113.8 (13) |
| C5—C6—C7 | 109.45 (14) | O4—S1—O3 | 115.18 (9) |
| C5—C6—H6A | 109.8 | O4—S1—O2 | 112.12 (8) |
| C7—C6—H6A | 109.8 | O3—S1—O2 | 110.11 (8) |
| C5—C6—H6B | 109.8 | O4—S1—C13 | 107.03 (9) |
| C7—C6—H6B | 109.8 | O3—S1—C13 | 106.76 (9) |
| H6A—C6—H6B | 108.2 | O2—S1—C13 | 104.92 (10) |
| C8—C7—C6 | 109.47 (15) | S1—C13—H13A | 109.5 |
| C8—C7—C2 | 109.44 (14) | S1—C13—H13B | 109.5 |
| C6—C7—C2 | 109.23 (15) | H13A—C13—H13B | 109.5 |
| C8—C7—H7 | 109.6 | S1—C13—H13C | 109.5 |
| C6—C7—H7 | 109.6 | H13A—C13—H13C | 109.5 |
| C2—C7—H7 | 109.6 | H13B—C13—H13C | 109.5 |
| C7—C8—C9 | 109.49 (14) | | |

*Hydrogen-bond geometry (Å, º)*

| *D*—H⋯*A* | *D*—H | H⋯*A* | *D*⋯*A* | *D*—H⋯*A* |
|---|---|---|---|---|
| N1—H1⋯O3[i] | 0.87 (2) | 1.98 (2) | 2.838 (2) | 170.1 (19) |
| O1—H2⋯O2 | 0.98 (2) | 1.49 (2) | 2.4632 (18) | 173 (2) |

Symmetry code: (i) *x*−1/2, −*y*+1/2, −*z*+1.