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Crystal structure of a mixed solvated form of amoxapine acetate

Rajni M. Bhardwaj, Vishal Raval, Iain D. H. Oswald and Alastair J. Florence*

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Strathclyde Institute of Pharmacy and Biomedical Sciences, University of Strathclyde, 161 Cathedral Street, Glasgow G4 0RE, Scotland. *Correspondence e-mail: alastair.florence@strath.ac.uk

The mixed solvated salt 4-(2-chlorodibenzo[b,f][1,4]oxazepin-11-yl)piperazin-1-ium acetate–acetic acid–cyclohexane (2/2/1), C17H17ClN3O+C2H3O2−C2H4O2−0.5C6H12, crystallizes with one molecule of protonated amoxapine (AXPN), an acetate anion and a molecule of acetic acid together with half a molecule of cyclohexane. In the centrosymmetric crystal, both enantiomers of the protonated AXPN molecule stack alternatively along [001]. Acetate anions connect the AXPN cations through N—H⋯O hydrogen bonding in the [010] direction, creating a sheet lying parallel to (100). The acetic acid molecules are linked to the acetate anions via O—H⋯O hydrogen bonds within the sheets. Within the sheets there are also a number of C—H⋯O hydrogen bonds present. The cyclohexane solvent molecules occupy the space between the sheets.

1. Chemical context

2-Chloro-11-(piperazin-1-yl)dibenzo[b,f][1,4]oxazepine (Amoxapine, AXPN) is a benzodiazepine derivative and exhibits anti-depressant properties (Greenbla & Osterber, 1968) with one reported crystal structure (CSD refcode: AMOXAP; Cosulich & Lovell, 1977). AXPN acetate acetic acid cyclohexane was obtained as a part of a wider investigation that couples experimental crystallization techniques with computational methods in order to obtain a better understanding of the factors underpinning the solid-state structure and diversity of structurally related compounds, i.e. olanzapine, clozapine, loxapine and AXPN (Bhardwaj & Florence, 2013; Bhardwaj, Johnston et al., 2013; Bhardwaj, Price et al., 2013). The sample of AXPN acetate acetic acid cyclohexane was isolated during an experimental physical form screen of AXPN. The sample was identified as a novel form using multi-sample foil transmission X-ray powder diffraction analysis (Florence et al., 2003). A suitable sample for single crystal X-ray diffraction analysis was obtained from slow evaporation of a saturated solution of AXPN in a 1:1 molar ratio of acetic acid and cyclohexane at room temperature.
2. Structural commentary

The title compound crystallizes with one molecule of protonated AXPN and an acetate anion each with a molecule of acetic acid and a half molecule of cyclohexane (which lies across a center of inversion) as solvent of crystallization in the asymmetric unit (Fig. 1). The dioxaazepine ring of AXPN exists in a puckered conformation between the planes of the benzene rings [the benzene rings fused to the central ring make a dihedral angle of 58.63° (6)], and the piperezine ring adopts a chair conformation, as observed in the AXPN free base (CSD refcode: AMOXAP; Cosulich and Lovell, 1977) and structurally related analogues (Bhardwaj & Florence, 2013; Bhardwaj, Johnston et al., 2013; Bhardwaj, Price et al., 2013).

3. Supramolecular features

In the crystal, opposite enantiomers of protonated AXPN molecules stack along the c-axis direction. Each protonated

![Image](image_url)

Figure 1
A view of the molecular structure of the asymmetric unit of the title molecular salt, showing the atom labelling. Displacement ellipsoids are drawn at the 50% probability level.

![Image](image_url)

Figure 2
The crystal packing of the title molecular salt, viewed down the b axis. The cyclohexane molecules are shown as a blue space-fill model. Hydrogen bonds are shown as green lines (see Table 1 for details; atom colour code: C, N, O, Cl and H are blue, violet, red, green and black, respectively. H atoms not involved in hydrogen bonding have been omitted for clarity).

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

| D—H · · ·A | D—H | H · · ·A | D · · ·A | D—H · · ·A |
|------------|------|---------|---------|-----------|
| N3—H1N3···O1S | 0.91 (2) | 1.86 (2) | 2.7664 (13) | 175 (2) |
| O3S—H1S1···O2S* | 0.94 (2) | 1.61 (2) | 2.5375 (13) | 171 (2) |
| N3—H2N3···O1S* | 0.94 (2) | 1.82 (2) | 2.7292 (14) | 162 (1) |
| Cl5—H1S1···O3S* | 0.96 | 2.42 | 3.3778 (18) | 172 |
| Cl4—H1A···O1S* | 0.97 | 2.59 | 3.2448 (15) | 125 |
| Cl7—H17A···O4S* | 0.97 | 2.32 | 3.2314 (15) | 155 |

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

![Image](image_url)

Table 2
Experimental details.

| Crystal data | Chemical formula | C17H13ClN3O4·C2H5O2·C2H5O2·0.5C6H12 |
|--------------|------------------|--------------------------------------|
| Mw           |                  | 475.96                               |
| Crystal system, space group | Monoclinic, P21/c |
| Temperature (K) | 150 |
| a, b, c (Å) | 21.0726 (12), 6.0393 (3), 18.6087 (10) |
| β (°) | 92.096 (2) |
| V (Å³) | 2366.6 (2) |
| β     | 4 |
| Radiation type | Mo Kα |
| μ (mm⁻¹) | 0.20 |
| Crystal size (mm) | 0.55 × 0.22 × 0.11 |

| Data collection | Bruker APEXII CCD |
|----------------|------------------|
| Diffractometer | Multi-scan (SADABS; Bruker, 2007) |
| Absorption correction | Bruker APEXII CCD |
| Tmin, Tmax | 0.047, 0.745 |
| No. of measured, independent and observed [I > 2σ(I)] reflections | 18828, 4860, 4177 |
| Rint | 0.018 |
| (sin θ/λ)max (Å⁻¹) | 0.626 |

| Refinement | Bruker SHELX97 and SHELXL97 (Sheldrick, 2008), Mercury (Macrae et al., 2008), ORTEP-3 for Windows (Farrugia, 2012), enCIFer (Allen et al., 2004) and publCIF (Westrip, 2010) |
|-------------|----------------------------------|
| R[F² > 2σ(F²)] | 0.030, 0.082, 1.03 |
| No. of reflections | 4860 |
| No. of parameters | 312 |
| H-atom treatment | H atoms treated by a mixture of independent and constrained refinement |
| Δρmax, Δρmin (e Å⁻³) | 0.28, −0.22 |

Computer programs: APEX2 and SAINT (Bruker, 2007), SHELXS97 and SHELXL97 (Sheldrick, 2008), Mercury (Macrae et al., 2008), ORTEP-3 for Windows (Farrugia, 2012), enCIFer (Allen et al., 2004) and publCIF (Westrip, 2010).
AXPN molecule forms two N—H⋯O hydrogen bonds with two acetate anions, which connect it to an adjacent protonated AXPN molecule along the $b$ axis, creating a sheet-like structure parallel to (100); see Fig. 2 and Table 1. The acetic acid molecules act as hydrogen-bond donors to acetate anions and are present between the protonated AXPN molecules along the $c$-axis direction. There are also C—H⋯O hydrogen bonds present within the sheets (Table 1). These sheets stack along the $a$ axis and the cyclohexane molecules occupy the space between the sheets (Fig. 2).

4. Synthesis and crystallization

Rod-shaped crystals were grown from a saturated solution of AXPN in a 1:1 molar ratio of acetic acid and cyclohexane by isothermal solvent evaporation at 298 K.

5. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. The N- and O-bound H atoms were located in a difference Fourier map and freely refined. The C-bound H atoms were placed in calculated positions and refined as riding atoms: C—H = 0.95–0.99 Å with $U_{iso}(H) = 1.5U_{eq}(C)$ for methyl H atoms and $= 1.2U_{eq}(C)$ for other H atoms.

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Computing details

Data collection: APEX2 (Bruker, 2007); cell refinement: SAINT (Bruker, 2007); data reduction: SAINT (Bruker, 2007); program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: Mercury (Macrae et al., 2008), ORTEP-3 for Windows (Farrugia, 2012); software used to prepare material for publication: enCIFer (Allen et al., 2004), publCIF (Westrip, 2010).

4-(2-Chlorodibenzo[b,f][1,4]oxazepin-11-yl)piperazin-1-ium acetate–acetic acid–cyclohexane (2/2/1)

Crystal data

C₁₇H₁₇ClN₃O⁺·C₂H₃O₂⁻·C₂H₄O₂·0.5C₆H₁₂  

F(000) = 1008  

Dₐ = 1.336 Mg m⁻³  

Mo Ka radiation, λ = 0.71073 Å  

Cell parameters from 9940 reflections  

θ = 2.9–26.4°  

µ = 0.20 mm⁻¹  

T = 150 K  

Rod, colourless  

0.55 × 0.22 × 0.11 mm

Data collection

Bruker APEXII CCD  

diffractometer  

Radiation source: fine-focus sealed tube  

Graphite monochromator  

φ and o scans  

Absorption correction: multi-scan  

(SADABS; Bruker, 2007)  

Tmin = 0.647, Tmax = 0.745

Refinement

Refinement on F²  

Least-squares matrix: full  

R[F² > 2σ(F²)] = 0.030  

wR(F²) = 0.082  

S = 1.03  

4860 reflections  

312 parameters  

0 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/[σ²(Fo²) + (0.0383P)² + 1.089P]  

where P = (Fo² + 2Fc²)/3

(Δ/σ)max = 0.001  

Δρmax = 0.28 e Å⁻³  

Δρmin = −0.22 e Å⁻³
**Special details**

**Geometry.** All s.u.'s (except the s.u. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell s.u.'s are taken into account individually in the estimation of s.u.'s in distances, angles and torsion angles; correlations between s.u.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell s.u.'s is used for estimating s.u.'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 ($\AA^2$)**

| Atom | $x$  | $y$  | $z$  | $U_{iso}/U_{eq}$ |
|------|------|------|------|------------------|
| H2N3 | 0.0503 (7) | -0.014 (3) | 0.3171 (8) | 0.029 (4)* |
| H1N3 | 0.0584 (7) | 0.167 (3) | 0.2658 (9) | 0.025 (4)* |
| H1S  | 0.1043 (10) | 0.564 (4) | 0.5635 (12) | 0.064 (6)* |
| Cl   | 0.276361 (16) | 0.39554 (6) | 0.594267 (17) | 0.02772 (10) |
| O1   | 0.30157 (4) | 0.90957 (15) | 0.32837 (5) | 0.0225 (2) |
| O2S  | 0.07221 (4) | 0.3016 (16) | 0.13430 (5) | 0.0259 (2) |
| O1S  | 0.01425 (4) | 0.28471 (16) | 0.18632 (5) | 0.0234 (2) |
| N3   | 0.07793 (5) | 0.09975 (18) | 0.30430 (6) | 0.0161 (2) |
| N2   | 0.19325 (5) | 0.34168 (17) | 0.31916 (5) | 0.0160 (2) |
| N1   | 0.27645 (5) | 0.48819 (18) | 0.25813 (6) | 0.0196 (2) |
| C4   | 0.33285 (6) | 0.6088 (2) | 0.25098 (7) | 0.0198 (3) |
| C6   | 0.25997 (5) | 0.4584 (2) | 0.45099 (6) | 0.0177 (2) |
| H6   | 0.2404 | 0.3204 | 0.4501 | 0.021* |
| C2S  | 0.03703 (5) | 0.1974 (2) | 0.13128 (6) | 0.0175 (2) |
| C5   | 0.24619 (5) | 0.4789 (2) | 0.31684 (6) | 0.0170 (2) |
| C2   | 0.29573 (5) | 0.7852 (2) | 0.39086 (7) | 0.0183 (3) |
| C8   | 0.31125 (6) | 0.7556 (2) | 0.51865 (7) | 0.0228 (3) |
| H8   | 0.3256 | 0.8140 | 0.5625 | 0.027* |
| C15  | 0.13972 (6) | 0.0041 (2) | 0.28291 (6) | 0.0167 (2) |
| H15A | 0.1326 | -0.1006 | 0.2439 | 0.020* |
| H15B | 0.1596 | -0.0741 | 0.3233 | 0.020* |
| C1   | 0.26655 (5) | 0.5783 (2) | 0.38728 (7) | 0.0168 (2) |
| C17  | 0.08763 (5) | 0.2614 (2) | 0.36432 (6) | 0.0169 (2) |
| H17A | 0.1044 | 0.1855 | 0.4068 | 0.020* |
| H17B | 0.0473 | 0.3278 | 0.3757 | 0.020* |
| C7   | 0.28300 (6) | 0.5483 (2) | 0.51529 (7) | 0.0200 (3) |
| C9   | 0.31772 (6) | 0.8743 (2) | 0.45570 (7) | 0.0219 (3) |
| H9   | 0.3368 | 1.0133 | 0.4569 | 0.026* |
| C16  | 0.13360 (5) | 0.4404 (2) | 0.34257 (7) | 0.0164 (2) |
| H16A | 0.1145 | 0.5276 | 0.3037 | 0.020* |
| H16B | 0.1425 | 0.5384 | 0.3830 | 0.020* |
| C10  | 0.39883 (6) | 0.9372 (2) | 0.26691 (7) | 0.0264 (3) |
| H10  | 0.4059 | 1.0745 | 0.2884 | 0.032* |
| C3   | 0.34532 (6) | 0.8156 (2) | 0.28257 (7) | 0.0208 (3) |
| C13  | 0.37702 (6) | 0.5278 (2) | 0.20305 (7) | 0.0234 (3) |
Atomic displacement parameters ($\AA^2$)

|   | $U^{11}$     | $U^{22}$     | $U^{33}$     | $U^{12}$     | $U^{13}$     | $U^{23}$     |
|---|--------------|--------------|--------------|--------------|--------------|--------------|
| Cl| 0.02984 (17) | 0.0361 (2)   | 0.01704 (16) | 0.00076 (14) | −0.00169 (12) | 0.00358 (14) |
| O1| 0.0221 (4)   | 0.0177 (4)   | 0.0275 (5)   | −0.0006 (4)  | −0.0010 (4)  | 0.0056 (4)   |
| O2S| 0.0311 (5)  | 0.0282 (5)   | 0.0183 (5)   | 0.0132 (4)   | 0.0001 (4)   | 0.0000 (4)   |
| O1S| 0.0258 (5)  | 0.0263 (5)   | 0.0179 (4)   | 0.0112 (4)   | 0.0001 (4)   | 0.0005 (4)   |
| N3| 0.0174 (5)   | 0.0156 (5)   | 0.0151 (5)   | −0.0003 (4)  | −0.0012 (4)  | 0.0029 (4)   |
| N2| 0.0154 (5)   | 0.0161 (5)   | 0.0165 (5)   | −0.0018 (4)  | 0.0005 (4)   | −0.0018 (4)  |
| N1| 0.0178 (5)   | 0.0222 (5)   | 0.0186 (5)   | −0.0040 (4)  | −0.0010 (4)  | 0.0033 (4)   |
| C4| 0.0183 (6)   | 0.0232 (7)   | 0.0175 (6)   | −0.0036 (5)  | −0.0034 (5)  | 0.0071 (5)   |
| C6| 0.0149 (5)   | 0.0185 (6)   | 0.0197 (6)   | 0.0015 (5)   | −0.0005 (4)  | −0.0006 (5)  |
| C2S| 0.0154 (5)  | 0.0197 (6)   | 0.0174 (6)   | −0.0009 (5)  | −0.0012 (4)  | 0.0017 (5)   |
| C5| 0.0164 (5)   | 0.0151 (6)   | 0.0192 (6)   | −0.0001 (5)  | −0.0024 (4)  | 0.0025 (5)   |
| C2| 0.0146 (5)   | 0.0179 (6)   | 0.0223 (6)   | 0.0017 (5)   | −0.0010 (5)  | 0.0020 (5)   |
| C8| 0.0184 (6)   | 0.0274 (7)   | 0.0222 (6)   | 0.0019 (5)   | −0.0036 (5)  | −0.0073 (6)  |
| C15| 0.0201 (6)  | 0.0144 (6)   | 0.0155 (6)   | 0.0002 (5)   | −0.0011 (4)  | 0.0011 (5)   |
| C1| 0.0133 (5)   | 0.0175 (6)   | 0.0196 (6)   | 0.0010 (4)   | −0.0013 (4)  | −0.0007 (5)  |
| C17| 0.0171 (5)  | 0.0184 (6)   | 0.0154 (6)   | −0.0001 (5)  | 0.0010 (4)   | 0.0006 (5)   |
C7  0.0166 (6)  0.0259 (7)  0.0175 (6)  0.0040 (5)  −0.0005 (5)  0.0011 (5)  
C9  0.0164 (6)  0.0192 (6)  0.0298 (7)  −0.0009 (5)  −0.0021 (5)  −0.0047 (5)  
C16  0.0173 (6)  0.0141 (6)  0.0179 (6)  −0.0001 (4)  0.0003 (4)  −0.0003 (5)  
C10  0.0266 (7)  0.0256 (7)  0.0264 (7)  −0.0086 (6)  −0.0048 (5)  0.0074 (6)  
C3  0.0184 (6)  0.0235 (7)  0.0202 (6)  −0.0008 (5)  −0.0029 (5)  0.0074 (5)  
C13  0.0228 (6)  0.0281 (7)  0.0193 (6)  −0.0038 (5)  −0.0009 (5)  0.0039 (6)  
C14  0.0176 (5)  0.0173 (6)  0.0150 (6)  −0.0011 (5)  −0.0001 (4)  −0.0002 (5)  
C12  0.0211 (6)  0.0394 (8)  0.0221 (7)  −0.0036 (6)  0.0017 (5)  0.0069 (6)  
C1S  0.0350 (7)  0.0344 (8)  0.0197 (7)  0.0083 (6)  −0.014 (6)  0.0068 (6)  
C11  0.0221 (6)  0.0392 (8)  0.0278 (7)  −0.0122 (6)  −0.0008 (5)  0.0099 (6)  
O4S  0.0308 (5)  0.0296 (5)  0.0182 (5)  −0.0071 (4)  0.0062 (4)  0.0109 (4)  
O3S  0.0309 (5)  0.0308 (5)  0.0244 (7)  −0.0058 (6)  0.0035 (6)  −0.0020 (6)  
C3S  0.0188 (6)  0.0233 (7)  0.0215 (6)  0.0054 (5)  0.0027 (5)  0.0031 (5)  
C6S  0.0226 (7)  0.0481 (9)  0.0332 (8)  0.0057 (6)  −0.0017 (6)  −0.0085 (7)  
C5S  0.0256 (7)  0.0440 (9)  0.0394 (9)  0.0011 (6)  0.0031 (6)  −0.0021 (7)  
C7S  0.0302 (8)  0.0467 (9)  0.0341 (8)  0.0076 (7)  −0.0009 (6)  0.0030 (7)  

Geometric parameters (Å, °)

C1—C7  1.7450 (13)  C16—H16A  0.9700  
O1—C2  1.3936 (15)  C16—H16B  0.9700  
O1—C3  1.3985 (16)  C10—C3  1.3856 (18)  
O2S—C2S  1.2529 (15)  C10—C11  1.387 (2)  
O1S—C2S  1.2623 (15)  C10—H10  0.9300  
N3—C15  1.4918 (15)  C13—C12  1.3866 (18)  
N3—C17  1.4919 (16)  C13—H13  0.9300  
N3—H2N3  0.938 (17)  C14—C15  1.5156 (16)  
N3—H1N3  0.908 (16)  C14—H14A  0.9700  
N2—C5  1.3916 (15)  C14—H14B  0.9700  
N2—C14  1.4618 (15)  C12—C11  1.388 (2)  
N2—C16  1.4716 (15)  C12—H12  0.9300  
N1—C5  1.2863 (16)  C1S—C5  1.527 (2)  
N1—C4  1.4044 (16)  C1S—H1S  0.9600  
C4—C13  1.4006 (19)  C1S—H1S1  0.9700  
C4—C3  1.4010 (19)  O4S—C3S  1.2125 (16)  
C6—C7  1.3852 (17)  O3S—C3S  1.3256 (16)  
C6—C1  1.4005 (17)  O3S—H1S  0.94 (2)  
C6—H6  0.9300  C4S—C3S  1.5019 (19)  
C2S—C1S  1.5027 (17)  C4S—H4S  0.9600  
C5—C1  1.4905 (17)  C4S—H4S2  0.9600  
C2—C9  1.3854 (18)  C4S—H4S3  0.9600  
C2—C1  1.3929 (17)  C6S—C7S  1.520 (2)  
C8—C9  1.3845 (19)  C6S—C5S  1.523 (2)  
C8—C7  1.3867 (19)  C6S—H6S  0.9700  
C8—H8  0.9300  C6S—H6S2  0.9700  
C15—C14  1.5156 (16)  C5S—C7S  1.527 (2)  
C15—H15A  0.9700  C5S—H5S  0.9700  

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| Bond          | Distance  | Bond          | Distance  | Bond          | Distance  |
|---------------|-----------|---------------|-----------|---------------|-----------|
| C15—H15B     | 0.9700    | C5S—H5S2     | 0.9700    | C7S—C5S       | 1.527 (2) |
| C17—C16      | 1.5164 (16) | C7S—H7S1     | 0.9700    | C7S—H7S2      | 0.9700    |
| C17—H17A     | 0.9700    |               |           |               |           |
| C17—H17B     | 0.9700    |               |           |               |           |
| C9—H9        | 0.9300    |               |           |               |           |
| C2—O1—C3     | 111.72 (9) | C3—C10—C11   | 119.77 (13) |               |           |
| C15—N3—C17   | 110.86 (9) | C3—C10—C10   | 120.1      |               |           |
| C15—N3—H2N3  | 110.0 (10) | C11—C10—H10  | 120.1      |               |           |
| C17—N3—H2N3  | 111.0 (10) | C10—C3—O1    | 118.21 (12) |               |           |
| C15—N3—H1N3  | 109.7 (9)  | C10—C3—C4    | 121.72 (13) |               |           |
| C17—N3—H1N3  | 110.1 (10) | O1—C3—C4     | 119.99 (11) |               |           |
| H2N3—N3—H1N3 | 105.1 (13) |               |           |               |           |
| C5—N2—C14    | 116.76 (10)| C12—C13—C4   | 119.4      |               |           |
| C5—N2—C16    | 117.53 (10)| C12—C13—H13  | 119.4      |               |           |
| C14—N2—C16   | 112.18 (9) |               |           |               |           |
| C5—N1—C4     | 123.41 (11)|               |           |               |           |
| C13—C4—C3    | 117.37 (12)|               |           |               |           |
| C13—C4—N1    | 117.62 (12)|               |           |               |           |
| C3—C4—N1     | 124.68 (12)|               |           |               |           |
| C7—C6—C1     | 119.10 (12)|               |           |               |           |
| C7—C6—H6     | 120.4      |               |           |               |           |
| C1—C6—H6     | 120.4      |               |           |               |           |
| O2S—C2S—O1S  | 122.84 (11) |               |           |               |           |
| O2S—C2S—C1S  | 119.38 (11)|               |           |               |           |
| O1S—C2S—C1S  | 117.79 (11)|               |           |               |           |
| N1—C5—N2     | 118.38 (11)|               |           |               |           |
| N1—C5—C1     | 126.41 (11)|               |           |               |           |
| N2—C5—C1     | 114.71 (10)|               |           |               |           |
| C9—C2—C1     | 121.55 (12)|               |           |               |           |
| C9—C2—O1     | 118.71 (11)|               |           |               |           |
| C1—C2—O1     | 119.72 (11)|               |           |               |           |
| C9—C8—C7     | 119.01 (12)|               |           |               |           |
| C9—C8—H8     | 120.5      |               |           |               |           |
| C7—C8—H8     | 120.5      |               |           |               |           |
| N3—C10—C14   | 109.97 (9) |               |           |               |           |
| N3—C15—H15A  | 109.8      | H4S1—C4S—H4S2 | 109.5    |               |           |
| C14—C15—H15A | 109.8      | H3S—C4S—H4S3 | 109.5    |               |           |
| C14—C15—H15B | 108.2      | O4S—C3S—O3S  | 119.90 (12) |               |           |
| H15A—C15—H15B| 118.71 (11)|               |           |               |           |
| C2—C1—C6     | 121.06 (11)|               |           |               |           |
| C2—C1—C5     | 120.13 (11)|               |           |               |           |
| C3—C1—C5     | 109.7      |               |           |               |           |
| N3—C17—C16   | 109.7      |               |           |               |           |
| C16—C17—H17A | 109.7      |               |           |               |           |
| N3—C17—H17B  | 109.7      |               |           |               |           |
C16—C17—H17B 109.7  H6S1—C6S—H6S2 108.0
H17A—C17—H17B 108.2  C6S—C5S—C7Si 110.96 (13)
C6—C7—C8 121.92 (12)  C6S—C5S—H5S1 109.4
C6—C7—Cl 118.96 (10)  C7Si—C5S—H5S1 109.4
C8—C7—Cl 119.13 (10)  C6S—C5S—H5S2 109.4
C8—C9—C2 119.70 (12)  C7Si—C5S—H5S2 109.4
C8—C9—H9 120.2   H5S1—C5S—H5S2 108.0
C2—C9—H9 120.2   C6S—C7Si—C5S 111.37 (13)
N2—C16—C17 110.55 (10)  C5Si—C7Si—H7Si 109.4
N2—C16—H16A 109.5  C5Si—C7Si—H7Si 109.4
C17—C16—H16A 109.5  C6S—C7Si—H7Si 109.4
C17—C16—H16B 109.5  C5Si—C7Si—H7Si 109.4
C5—N1—C4—C13 148.72 (12)  C9—C8—C7—C6 178.58 (9)
C5—N1—C4—C3 −38.18 (18)  C7—C8—C9—C2 0.34 (18)
C4—N1—C5—N2 −175.55 (11)  O1—C2—C9—C8 187.70 (11)
C4—N1—C5—C1 −4.1 (2)  C5Si—C7Si—C5Si—C7Si −55.20 (19)
C14—N2—C5—N1 10.69 (16)  C9—C8—C7—Cl −1.29 (18)
C16—N2—C5—N1 −127.00 (12)  C5Si—C7Si—C5Si—C7Si 55.42 (18)
C14—N2—C5—C1 −161.70 (10)  N3—C17—C16—N2 −54.59 (12)
C16—N2—C5—C1 60.61 (14)  N3—C17—C16—N2 58.13 (12)
C3—O1—C2—C9 111.88 (12)  N1—C4—C13—C12 173.70 (12)
C3—O1—C2—C1 −69.92 (13)  C5—N2—C14—C15 159.85 (10)
C17—N3—C15—C14 −59.51 (12)  C16—N2—C14—C15 −60.29 (12)
C9—C2—C1—C6 −0.46 (17)  N1—C5—C1—C2 38.92 (18)
O1—C2—C1—C6 −178.62 (10)  N1—C5—C1—C2 −137.27 (13)
C7—C6—C1—C2 −0.46 (17)  N1—C5—C1—C2 34.41 (16)
C7—C6—C1—C5 175.81 (11)  N1—C5—C1—C2 38.92 (18)
N1—C5—C1—C2 38.92 (18)  N1—C5—C1—C2 −176.70 (11)
N2—C5—C1—C2 −149.41 (11)  N1—C5—C1—C2 −137.27 (13)
N2—C5—C1—C2 34.41 (16)  C15—N3—C17—C16 −55.20 (19)
C15—N3—C17—C16 56.29 (12)  C1—C6—C7—Cl −55.20 (19)
C1—C6—C7—Cl −178.52 (9)  C9—C8—C7—Cl 55.42 (18)
C1—C6—C7—C8 1.35 (18)  C5Si—C6S—C7S—C5Si 55.42 (18)
C9—C8—C7—C6 −1.29 (18)  C5Si—C6S—C7S—C5Si 55.42 (18)

Symmetry code: (i) −x+1, −y+1, −z+1.

Hydrogen-bond geometry (Å, °)

| D—H···A | D—H | H···A | D···A | D—H···A |
|---------|------|------|-------|---------|
| N3—H1N3···O1Si | 0.91 (2) | 1.86 (2) | 2.7664 (13) | 175 (2) |
| O3Si—H1Si···O2Si | 0.94 (2) | 1.61 (2) | 2.5375 (13) | 171 (2) |

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### Supporting Information

|          | D     | r     | D     | θ     |
|----------|-------|-------|-------|-------|
| N3—H2N3···O1S<sup>iii</sup> | 0.94 (2) | 1.82 (2) | 2.7292 (14) | 162 (1) |
| C1S—H1S1···O3S<sup>iii</sup> | 0.96  | 2.42  | 3.3778 (18) | 172   |
| C14—H14···O1<sup>iv</sup> | 0.97  | 2.59  | 3.2448 (15) | 125   |
| C17—H17···O4S<sup>iv</sup> | 0.97  | 2.32  | 3.2314 (15) | 155   |

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