# organic compounds

# 5-Chloro-2-(4-methoxyphenyl)-1,3-benzothiazole

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In the title compound, $C_{14}H_{10}ClNOS$, the dihedral angle between the benzothiazole ring system and the methoxy-substituted benzene ring is 8.76 (16)°. In the crystal, molecules are stacked in columns along the $c$ axis and no significant intermolecular interactions are observed.

## Related literature

For the biological activity of benzothiazole compounds, see: Chohan *et al.* (2003); Khan *et al.* (2011); Hutchinson *et al.* (2002); Burger & Sawhney (1968); Palmer *et al.* (1971). For related structures, see: Yousuf *et al.* (2012a,b).
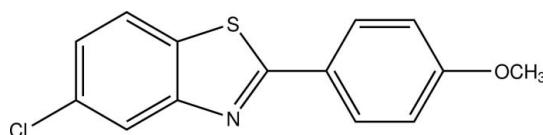


## Experimental

### Crystal data

| | |
|---|---|
| $C_{14}H_{10}ClNOS$ | $b$ = 14.5512 (8) Å |
| $M_r$ = 275.74 | $c$ = 5.8686 (3) Å |
| Orthorhombic, $Pbcn$ | $V$ = 2478.8 (2) Å³ |
| $a$ = 29.0274 (16) Å | $Z$ = 8 |

| | |
|---|---|
| Mo $K\alpha$ radiation | $T$ = 273 K |
| $\mu$ = 0.46 mm⁻¹ | 0.37 × 0.22 × 0.10 mm |

### Data collection

| | |
|---|---|
| Bruker SMART APEX CCD area-detector diffractometer | 13397 measured reflections |
| | 2299 independent reflections |
| Absorption correction: multi-scan (*SADABS*; Bruker, 2000) | 1965 reflections with $I > 2\sigma(I)$ |
| $T_{min}$ = 0.848, $T_{max}$ = 0.955 | $R_{int}$ = 0.052 |

### Refinement

| | |
|---|---|
| $R[F^2 > 2\sigma(F^2)]$ = 0.076 | 164 parameters |
| $wR(F^2)$ = 0.208 | H-atom parameters constrained |
| $S$ = 1.15 | $\Delta\rho_{max}$ = 0.63 e Å⁻³ |
| 2299 reflections | $\Delta\rho_{min}$ = −0.37 e Å⁻³ |

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*, *PARST* (Nardelli, 1996) and *PLATON* (Spek, 2009).

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# supplementary materials

## 5-Chloro-2-(4-methoxyphenyl)-1,3-benzothiazole

**Sammer Yousuf, Shazia Shah, Nida Ambreen, Khalid M. Khan and Shakil Ahmad**

### Comment

Benzothiazole is a well known class of organic compounds with a diverse range of biological activities (Khan *et al.*, 2011; Chohan *et al.*, 2003, Hutchinson *et al.*, 2002; Burger & Sawhney, 1968; Palmer *et al.*, 1971). The title compound is a methoxy phenyl derivative of benzothiazole synthesized as a part of our ongoing project to synthesize bioactive hetereocyclic compounds.

The crystal structure of title compound (Fig. 1), $C_{16}H_{14}ClNOS$, is similar to that our previously published 5-chloro-2-(3,4,5-trimethoxyphenyl)-1,3-benzothiazole (Yousuf *et al.*, 2012*b*) with the difference that the 3,4,5-trimethoxy-phenyl ring is replaced by the 4-methoxyphenyl phenyl ring. The dihedral angle between planner benzothiazole (S1/N1/C1–C7) and methoxy phenyl rings (C8–C13) is 8.76 (16)°. The bond lengths and angle are similar as in previously published benzothiazole compounds (Yousuf *et al.*, 2012*a*,*b*). In the crystal structure the molecules having plane of mirror are arranged in a two-diminesional manner along *a* and *c* axes (Fig. 2).

### Experimental

A mixture of 2-amino-4-cholorobenzenethiol (0.159 g, 1 mmol), 4-methoxybenzaldehyde (0.136 g, 1 mmol), sodium metabisulfite (0.2 g) and *N*,*N*-dimethylformamide (10 ml) was refluxed for 2 hrs in a round-bottomed flask. The completion of reaction was monitored by TLC and cool to room temperature followed by addition of cold water to obtain white precipitates. Crystallization from ethanol afforded pure crystal of 5-chloro-2-(4-methoxyphenyl) benzothiazole (yield 0.223 g, 81.1%) found suitable for X-ray diffraction studies.

### Refinement

H atoms of phenyl and methyl groups were positioned geometrically with C—H = 0.93 and 0.96 Å, respectively, and constrained to ride on their parent atoms with $U_{iso}(H) = 1.2U_{eq}(C_{phenyl})$ and $1.5U_{eq}(C_{methyl})$. A rotating group model was applied to the methyl group.

### Computing details

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT* (Bruker, 2000); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL* (Sheldrick, 2008), *PARST* (Nardelli, 1996) and *PLATON* (Spek, 2009).
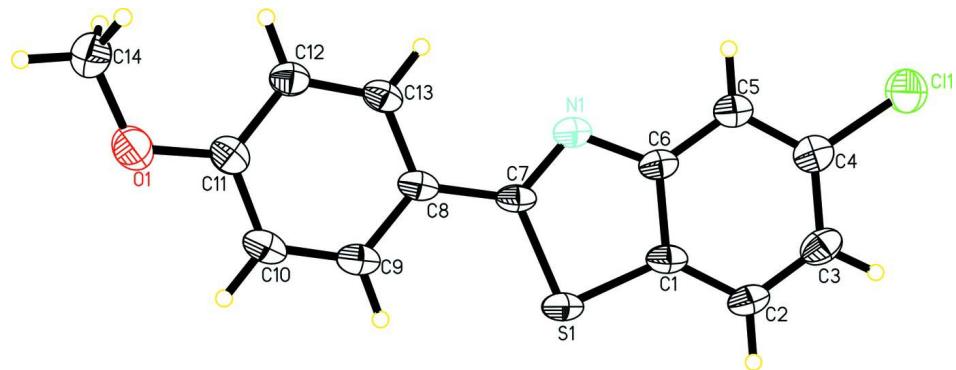
**Figure 1**

The molecular structure of title compound with displacement ellipsoids drawn at 30% probability level.
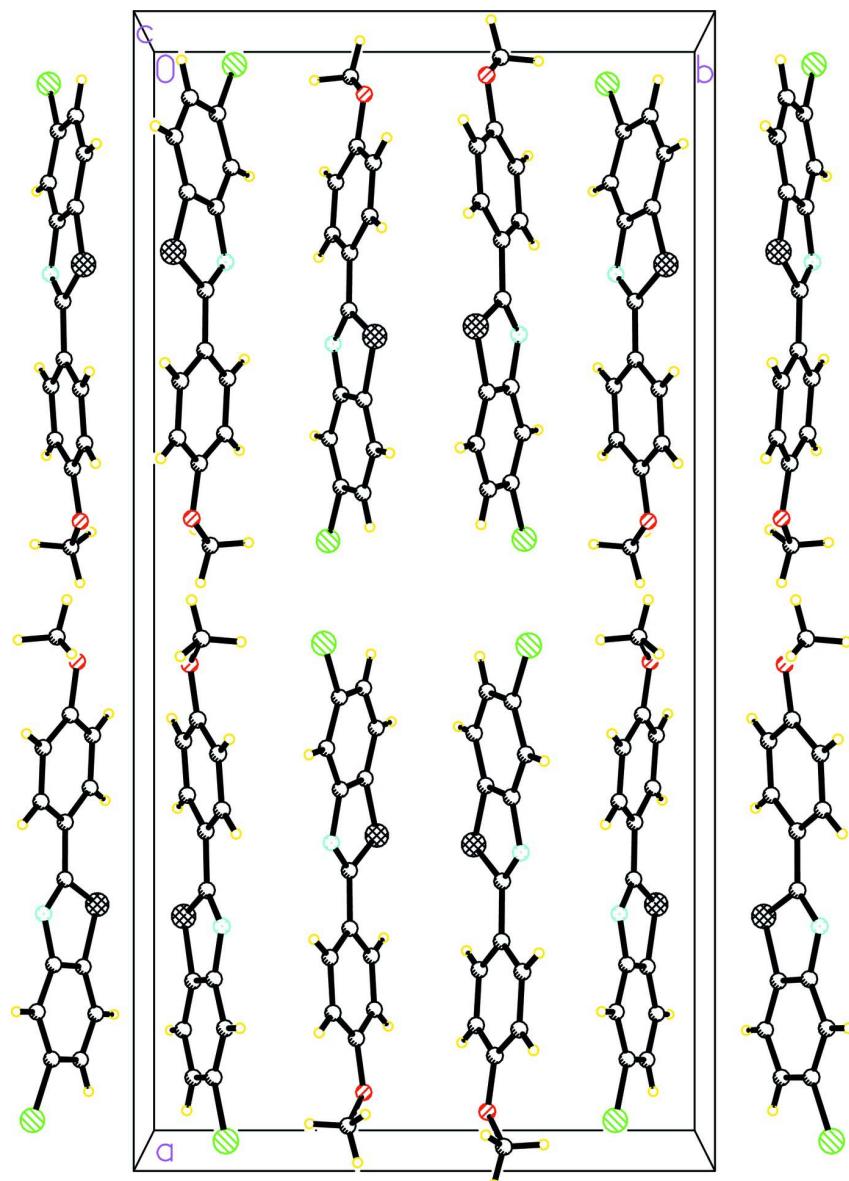
**Figure 2**

A crystal packing diagram of the title compound, viewed along the *c* axis.

**5-Chloro-2-(4-methoxyphenyl)-1,3-benzothiazole**

*Crystal data*

| | |
|---|---|
| $C_{14}H_{10}ClNOS$ | $F(000) = 1136$ |
| $M_r = 275.74$ | $D_x = 1.478$ Mg m$^{-3}$ |
| Orthorhombic, *Pbcn* | Mo *Kα* radiation, $\lambda = 0.71073$ Å |
| Hall symbol: -P 2n 2ab | Cell parameters from 3307 reflections |
| $a = 29.0274$ (16) Å | $\theta = 2.5–26.3°$ |
| $b = 14.5512$ (8) Å | $\mu = 0.46$ mm$^{-1}$ |
| $c = 5.8686$ (3) Å | $T = 273$ K |
| $V = 2478.8$ (2) Å$^3$ | Block, colorles |
| $Z = 8$ | $0.37 \times 0.22 \times 0.10$ mm |

*Data collection*

| | |
|---|---|
| Bruker SMART APEX CCD area-detector diffractometer | 13397 measured reflections |
| Radiation source: fine-focus sealed tube | 2299 independent reflections |
| Graphite monochromator | 1965 reflections with $I > 2\sigma(I)$ |
| $\omega$ scan | $R_{int} = 0.052$ |
| Absorption correction: multi-scan (*SADABS*; Bruker, 2000) | $\theta_{max} = 25.5°$, $\theta_{min} = 1.6°$ |
| $T_{min} = 0.848$, $T_{max} = 0.955$ | $h = -34 \rightarrow 34$ |
| | $k = -17 \rightarrow 17$ |
| | $l = -6 \rightarrow 7$ |

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | Secondary atom site location: difference Fourier map |
| Least-squares matrix: full | Hydrogen site location: inferred from neighbouring sites |
| $R[F^2 > 2\sigma(F^2)] = 0.076$ | H-atom parameters constrained |
| $wR(F^2) = 0.208$ | $w = 1/[\sigma^2(F_o^2) + (0.0983P)^2 + 3.3682P]$ where $P = (F_o^2 + 2F_c^2)/3$ |
| $S = 1.15$ | |
| 2299 reflections | $(\Delta/\sigma)_{max} < 0.001$ |
| 164 parameters | $\Delta\rho_{max} = 0.63$ e Å⁻³ |
| 0 restraints | $\Delta\rho_{min} = -0.37$ e Å⁻³ |
| Primary atom site location: structure-invariant direct methods | |

*Special details*

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

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

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

| | $x$ | $y$ | $z$ | $U_{iso}*/U_{eq}$ |
|---|---|---|---|---|
| Cl1 | 0.04504 (4) | 0.17240 (11) | −0.0360 (2) | 0.0787 (5) |
| S1 | 0.21904 (4) | 0.08514 (7) | 0.52664 (16) | 0.0494 (4) |
| O1 | 0.43838 (11) | 0.1057 (2) | 0.1916 (6) | 0.0656 (9) |
| N1 | 0.21907 (12) | 0.1613 (2) | 0.1301 (5) | 0.0437 (8) |
| C1 | 0.16642 (15) | 0.1048 (2) | 0.4009 (6) | 0.0450 (9) |
| C2 | 0.12234 (17) | 0.0857 (3) | 0.4807 (7) | 0.0502 (10) |
| H2A | 0.1179 | 0.0592 | 0.6231 | 0.060* |
| C3 | 0.08560 (16) | 0.1072 (3) | 0.3433 (7) | 0.0556 (11) |
| H3A | 0.0558 | 0.0946 | 0.3927 | 0.067* |
| C4 | 0.09246 (15) | 0.1475 (3) | 0.1304 (8) | 0.0530 (10) |
| C5 | 0.13595 (15) | 0.1688 (3) | 0.0488 (7) | 0.0471 (9) |
| H5A | 0.1400 | 0.1968 | −0.0922 | 0.057* |
| C6 | 0.17346 (14) | 0.1466 (2) | 0.1872 (6) | 0.0420 (9) |
| C7 | 0.24620 (14) | 0.1318 (2) | 0.2888 (6) | 0.0409 (9) |
| C8 | 0.29667 (14) | 0.1305 (2) | 0.2692 (6) | 0.0420 (9) |
| C9 | 0.32493 (16) | 0.0941 (3) | 0.4380 (7) | 0.0511 (10) |
| H9A | 0.3118 | 0.0730 | 0.5727 | 0.061* |

| | | | |
|---|---|---|---|
| C10 | 0.37175 (16) | 0.0885 (3) | 0.4104 (7) | 0.0535 (11) |
| H10A | 0.3901 | 0.0646 | 0.5261 | 0.064* |
| C11 | 0.39169 (15) | 0.1187 (3) | 0.2077 (7) | 0.0487 (10) |
| C12 | 0.36468 (15) | 0.1576 (3) | 0.0376 (7) | 0.0479 (9) |
| H12A | 0.3780 | 0.1795 | −0.0959 | 0.057* |
| C13 | 0.31762 (15) | 0.1631 (2) | 0.0709 (7) | 0.0461 (9) |
| H13A | 0.2994 | 0.1893 | −0.0421 | 0.055* |
| C14 | 0.46063 (17) | 0.1316 (4) | −0.0114 (9) | 0.0717 (14) |
| H14A | 0.4926 | 0.1149 | −0.0032 | 0.108* |
| H14B | 0.4580 | 0.1969 | −0.0316 | 0.108* |
| H14C | 0.4465 | 0.1007 | −0.1379 | 0.108* |

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

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| Cl1 | 0.0538 (7) | 0.1036 (11) | 0.0787 (9) | 0.0116 (7) | 0.0019 (6) | 0.0106 (7) |
| S1 | 0.0692 (7) | 0.0454 (6) | 0.0336 (5) | −0.0036 (5) | 0.0005 (4) | 0.0084 (4) |
| O1 | 0.0598 (19) | 0.072 (2) | 0.065 (2) | 0.0054 (15) | −0.0097 (16) | 0.0031 (16) |
| N1 | 0.058 (2) | 0.0384 (16) | 0.0347 (16) | −0.0028 (14) | 0.0022 (15) | 0.0019 (13) |
| C1 | 0.069 (3) | 0.0347 (18) | 0.0308 (18) | −0.0014 (17) | 0.0019 (18) | 0.0005 (14) |
| C2 | 0.068 (3) | 0.044 (2) | 0.039 (2) | −0.0038 (19) | 0.0129 (19) | 0.0029 (16) |
| C3 | 0.066 (3) | 0.051 (2) | 0.050 (2) | −0.004 (2) | 0.020 (2) | −0.0059 (19) |
| C4 | 0.061 (3) | 0.046 (2) | 0.052 (2) | 0.0044 (18) | 0.007 (2) | −0.0042 (18) |
| C5 | 0.061 (2) | 0.0386 (19) | 0.042 (2) | 0.0006 (17) | 0.0043 (19) | −0.0007 (16) |
| C6 | 0.061 (2) | 0.0313 (16) | 0.0334 (18) | −0.0018 (16) | 0.0071 (17) | −0.0023 (14) |
| C7 | 0.064 (2) | 0.0315 (17) | 0.0269 (17) | −0.0024 (16) | 0.0010 (17) | −0.0002 (14) |
| C8 | 0.065 (2) | 0.0298 (16) | 0.0314 (18) | −0.0007 (15) | −0.0052 (17) | −0.0006 (14) |
| C9 | 0.072 (3) | 0.045 (2) | 0.036 (2) | −0.0032 (19) | −0.0038 (19) | 0.0056 (16) |
| C10 | 0.071 (3) | 0.047 (2) | 0.043 (2) | 0.0017 (19) | −0.016 (2) | 0.0052 (17) |
| C11 | 0.056 (2) | 0.043 (2) | 0.047 (2) | 0.0021 (17) | −0.0078 (19) | −0.0040 (17) |
| C12 | 0.059 (2) | 0.042 (2) | 0.043 (2) | −0.0012 (18) | −0.0009 (18) | 0.0047 (16) |
| C13 | 0.061 (2) | 0.0376 (19) | 0.040 (2) | 0.0003 (17) | −0.0088 (18) | 0.0040 (15) |
| C14 | 0.055 (3) | 0.092 (4) | 0.068 (3) | −0.007 (3) | 0.001 (2) | 0.004 (3) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| Cl1—C4 | 1.726 (5) | C5—H5A | 0.9300 |
| S1—C1 | 1.720 (4) | C7—C8 | 1.470 (6) |
| S1—C7 | 1.741 (4) | C8—C9 | 1.391 (5) |
| O1—C11 | 1.372 (5) | C8—C13 | 1.396 (5) |
| O1—C14 | 1.407 (5) | C9—C10 | 1.371 (7) |
| N1—C7 | 1.293 (5) | C9—H9A | 0.9300 |
| N1—C6 | 1.382 (5) | C10—C11 | 1.394 (6) |
| C1—C2 | 1.391 (6) | C10—H10A | 0.9300 |
| C1—C6 | 1.409 (5) | C11—C12 | 1.390 (5) |
| C2—C3 | 1.373 (7) | C12—C13 | 1.382 (6) |
| C2—H2A | 0.9300 | C12—H12A | 0.9300 |
| C3—C4 | 1.394 (6) | C13—H13A | 0.9300 |
| C3—H3A | 0.9300 | C14—H14A | 0.9600 |
| C4—C5 | 1.386 (6) | C14—H14B | 0.9600 |

| | | | |
|---|---|---|---|
| C5—C6 | 1.396 (6) | C14—H14C | 0.9600 |
| | | | |
| C1—S1—C7 | 89.62 (18) | C9—C8—C13 | 117.8 (4) |
| C11—O1—C14 | 118.4 (4) | C9—C8—C7 | 122.5 (4) |
| C7—N1—C6 | 110.9 (3) | C13—C8—C7 | 119.7 (3) |
| C2—C1—C6 | 121.3 (4) | C10—C9—C8 | 121.5 (4) |
| C2—C1—S1 | 129.8 (3) | C10—C9—H9A | 119.2 |
| C6—C1—S1 | 109.0 (3) | C8—C9—H9A | 119.2 |
| C3—C2—C1 | 118.2 (4) | C9—C10—C11 | 119.6 (4) |
| C3—C2—H2A | 120.9 | C9—C10—H10A | 120.2 |
| C1—C2—H2A | 120.9 | C11—C10—H10A | 120.2 |
| C2—C3—C4 | 120.7 (4) | O1—C11—C12 | 124.4 (4) |
| C2—C3—H3A | 119.6 | O1—C11—C10 | 115.2 (4) |
| C4—C3—H3A | 119.6 | C12—C11—C10 | 120.5 (4) |
| C5—C4—C3 | 122.3 (4) | C13—C12—C11 | 118.7 (4) |
| C5—C4—Cl1 | 118.9 (3) | C13—C12—H12A | 120.7 |
| C3—C4—Cl1 | 118.8 (3) | C11—C12—H12A | 120.7 |
| C4—C5—C6 | 117.2 (4) | C12—C13—C8 | 121.9 (4) |
| C4—C5—H5A | 121.4 | C12—C13—H13A | 119.0 |
| C6—C5—H5A | 121.4 | C8—C13—H13A | 119.0 |
| N1—C6—C5 | 124.7 (3) | O1—C14—H14A | 109.5 |
| N1—C6—C1 | 114.9 (4) | O1—C14—H14B | 109.5 |
| C5—C6—C1 | 120.3 (4) | H14A—C14—H14B | 109.5 |
| N1—C7—C8 | 123.7 (3) | O1—C14—H14C | 109.5 |
| N1—C7—S1 | 115.5 (3) | H14A—C14—H14C | 109.5 |
| C8—C7—S1 | 120.6 (3) | H14B—C14—H14C | 109.5 |
| | | | |
| C7—S1—C1—C2 | 179.4 (4) | C1—S1—C7—N1 | 1.1 (3) |
| C7—S1—C1—C6 | −0.7 (3) | C1—S1—C7—C8 | −174.8 (3) |
| C6—C1—C2—C3 | 1.2 (6) | N1—C7—C8—C9 | −176.7 (3) |
| S1—C1—C2—C3 | −178.9 (3) | S1—C7—C8—C9 | −1.1 (5) |
| C1—C2—C3—C4 | −0.4 (6) | N1—C7—C8—C13 | 0.5 (5) |
| C2—C3—C4—C5 | −0.9 (6) | S1—C7—C8—C13 | 176.0 (3) |
| C2—C3—C4—Cl1 | 179.6 (3) | C13—C8—C9—C10 | −1.2 (6) |
| C3—C4—C5—C6 | 1.3 (6) | C7—C8—C9—C10 | 176.0 (4) |
| Cl1—C4—C5—C6 | −179.2 (3) | C8—C9—C10—C11 | −0.9 (6) |
| C7—N1—C6—C5 | −178.4 (3) | C14—O1—C11—C12 | −1.3 (6) |
| C7—N1—C6—C1 | 0.7 (4) | C14—O1—C11—C10 | 177.8 (4) |
| C4—C5—C6—N1 | 178.6 (3) | C9—C10—C11—O1 | −176.6 (4) |
| C4—C5—C6—C1 | −0.4 (5) | C9—C10—C11—C12 | 2.5 (6) |
| C2—C1—C6—N1 | −179.9 (3) | O1—C11—C12—C13 | 177.1 (4) |
| S1—C1—C6—N1 | 0.2 (4) | C10—C11—C12—C13 | −1.9 (6) |
| C2—C1—C6—C5 | −0.8 (5) | C11—C12—C13—C8 | −0.2 (6) |
| S1—C1—C6—C5 | 179.3 (3) | C9—C8—C13—C12 | 1.8 (5) |
| C6—N1—C7—C8 | 174.5 (3) | C7—C8—C13—C12 | −175.5 (3) |
| C6—N1—C7—S1 | −1.2 (4) | | |