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## Structure Reports

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## 4-(2-Chloroethoxy)phthalonitrile

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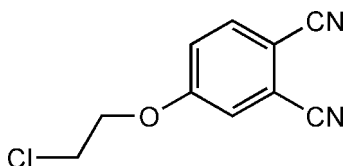
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 Key indicators: single-crystal X-ray study;  $T = 295$  K; mean  $\sigma(\text{C}-\text{C}) = 0.006$  Å; disorder in main residue;  $R$  factor = 0.065;  $wR$  factor = 0.241; data-to-parameter ratio = 11.2.

In the title compound,  $\text{C}_{10}\text{H}_7\text{ClN}_2\text{O}$ , the O and both C atoms of the chloroethoxy group are disordered over two positions, the occupancy factor of the major disorder component refining to 0.54 (2).

## Related literature

For background to the use of phthalonitriles and phthalocyanines, see: McKeown (1998); Leznoff & Lever (1989–1996); Moser & Thomas (1983). For related structures, see: Nesi *et al.* (1998); Dinçer *et al.* (2004); Ocak *et al.* (2004).



## Experimental

## Crystal data

 $\text{C}_{10}\text{H}_7\text{ClN}_2\text{O}$   
 $M_r = 206.63$ 

 Monoclinic,  $P2_1/c$   
 $a = 4.9021$  (8) Å

 $b = 19.014$  (3) Å  
 $c = 10.640$  (3) Å  
 $\beta = 97.123$  (18)°  
 $V = 984.1$  (3) Å<sup>3</sup>  
 $Z = 4$ 

 Mo  $K\alpha$  radiation  
 $\mu = 0.35$  mm<sup>-1</sup>  
 $T = 295$  (2) K  
 $0.6 \times 0.2 \times 0.1$  mm

## Data collection

 Bruker P4 diffractometer  
 Absorption correction: none  
 2496 measured reflections  
 1741 independent reflections  
 890 reflections with  $I > 2\sigma(I)$ 
 $R_{\text{int}} = 0.062$   
 3 standard reflections  
 every 97 reflections  
 intensity decay: none

## Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.065$   
 $wR(F^2) = 0.240$   
 $S = 1.08$   
 1741 reflections  
 155 parameters

 3 restraints  
 H-atom parameters constrained  
 $\Delta\rho_{\text{max}} = 0.27$  e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}} = -0.34$  e Å<sup>-3</sup>

Data collection: *XSCANS* (Bruker, 1997); cell refinement: *XSCANS*; data reduction: *XSCANS*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL* software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SJ2530).

## References

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## supporting information

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## 4-(2-Chloroethoxy)phthalonitrile

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### S1. Comment

Substituted phthalonitriles are generally used for preparing peripherally substituted symmetrical and unsymmetrical phthalocyanine complexes and subphthalocyanines (McKeown, 1998; Leznoff & Lever, 1989–1996). Phthalocyanines were first developed as dyes and pigments (Moser & Thomas, 1983). Over last few years, a great deal of interest has been focused on the synthesis of phthalocyanine derivatives due to their applications in fields, such as chemical sensors, electrochromism, batteries, semiconducting materials, liquid crystals, non-linear optics and photodynamic therapy (PDT) (Leznoff & Lever, 1989–1996). We report here the structure of the title phthalonitrile derivative, (I), (Fig 1).

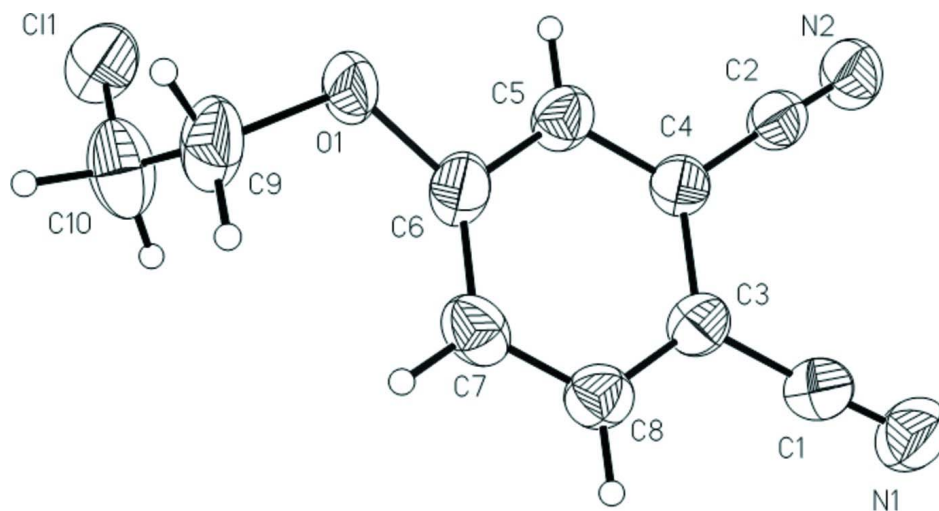
The title compound, C<sub>10</sub>H<sub>7</sub>ClN<sub>2</sub>O, contains a phthalonitrile ring and 2-chloroethoxy substituent in the 4-position. The oxygen and both carbon atoms of this substituent are disordered over two positions. The occupancy factor of the major disorder component refined to 0.54 (2). The C1≡N1 and C2≡N2 bond distances are both 1.138 (4) Å, consistent with N≡C triple-bond character, They are also in good agreement with literature values (Nesi *et al.*, 1998; Dinçer *et al.*, 2004; Ocak *et al.*, 2004).

### S2. Experimental

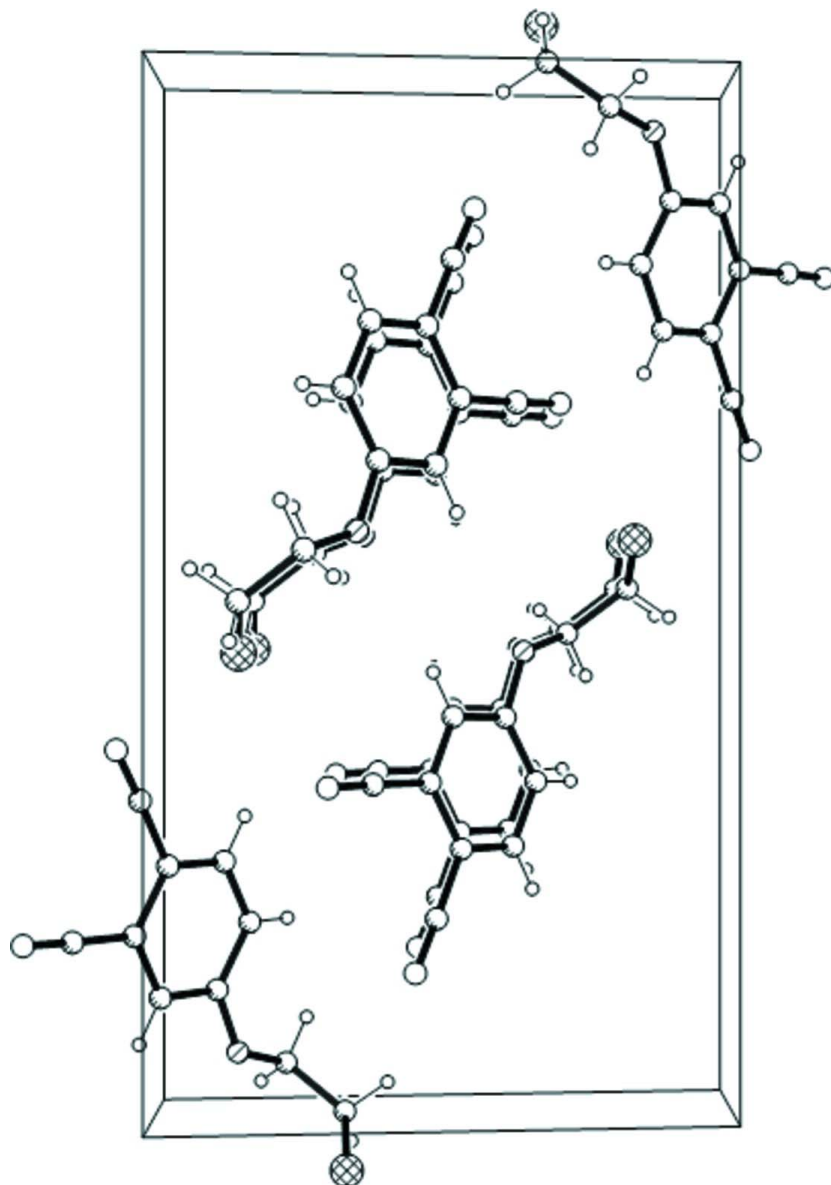
2-chloroethanol (1.6 g, 20 mmol) and 3-nitrophthalonitrile (1.73 g, 10 mmol) were dissolved in dry dimethylformamide (50 ml). After stirring for 1 h at room temperature, dry fine-powdered potassium carbonate (2.76 g, 20 mmol) was added portionwise over a period of 2 h with stirring. The reaction mixture was stirred for 36 h at room temperature and poured into ice-water (300 g). The product was filtered off and washed with water until the filtrate was neutral. Recrystallization from toluene gave a white product (yield 1.6 g, 77.4%). Single crystals were obtained from ethanol at room temperature by slow evaporation. Spectroscopic analysis: IR (KBr,  $\nu$  cm<sup>-1</sup>): 2963, 2868, 2237, 2229; MS(ESI, CH<sub>3</sub>OH):  $m/z$  = 207.2 [M+H]<sup>+</sup>; Anal. Found: C, 58.45; H, 3.72; N, 13.23%. Calcd for C<sub>18</sub>H<sub>16</sub>N<sub>2</sub>O: C, 58.13; H, 3.41; N, 13.56%

### S3. Refinement

The oxygen and both carbon atoms of the chloroethoxy group were disordered over two positions. The occupancy factor of the major disorder component refined to 0.52 (2). All H-atoms were positioned geometrically and refined using a riding model with d(C—H) = 0.93 Å,  $U_{\text{iso}}=1.2U_{\text{eq}}$  (C) for aromatic and 0.96 Å,  $U_{\text{iso}}=1.2U_{\text{eq}}$  (C) for CH<sub>2</sub> atoms.

**Figure 1**

The structure of the title compound, (I), showing 35% probability ellipsoids and the atom numbering scheme. For clarity only atoms of the major disorder component are shown.

**Figure 2**

The molecular packing of (I) viewed along the *a* axis. H atoms and atoms of the minor disorder component have been omitted.

**(I)***Crystal data* $C_{10}H_7ClN_2O$  $M_r = 206.63$ Monoclinic,  $P2_1/c$ Hall symbol:  $-P\ 2_1/c$  $a = 4.9021(8)\ \text{\AA}$  $b = 19.014(3)\ \text{\AA}$  $c = 10.640(3)\ \text{\AA}$  $\beta = 97.123(18)^\circ$  $V = 984.1(3)\ \text{\AA}^3$  $Z = 4$  $F(000) = 424$  $D_x = 1.395\ \text{Mg m}^{-3}$ Mo  $K\alpha$  radiation,  $\lambda = 0.71073\ \text{\AA}$ 

Cell parameters from 43 reflections

 $\theta = 4.9\text{--}12.4^\circ$  $\mu = 0.35\ \text{mm}^{-1}$

$T = 295$  K  
Prism, colorless

$0.6 \times 0.2 \times 0.1$  mm

*Data collection*

Bruker P4  
diffractometer  
Radiation source: fine-focus sealed tube  
Graphite monochromator  
 $\omega$  scans  
2496 measured reflections  
1741 independent reflections  
890 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.062$   
 $\theta_{\text{max}} = 25.1^\circ$ ,  $\theta_{\text{min}} = 2.1^\circ$   
 $h = -5 \rightarrow 1$   
 $k = -1 \rightarrow 22$   
 $l = -12 \rightarrow 12$   
3 standard reflections every 97 reflections  
intensity decay: none

*Refinement*

Refinement on  $F^2$   
Least-squares matrix: full  
 $R[F^2 > 2\sigma(F^2)] = 0.065$   
 $wR(F^2) = 0.240$   
 $S = 1.08$   
1741 reflections  
155 parameters  
3 restraints  
Primary atom site location: structure-invariant  
direct methods

Secondary atom site location: difference Fourier  
map  
Hydrogen site location: inferred from  
neighbouring sites  
H-atom parameters constrained  
 $w = 1/[\sigma^2(F_o^2) + (0.1087P)^2 + 0.4256P]$   
where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\text{max}} < 0.001$   
 $\Delta\rho_{\text{max}} = 0.27 \text{ e } \text{\AA}^{-3}$   
 $\Delta\rho_{\text{min}} = -0.34 \text{ e } \text{\AA}^{-3}$

*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 ( $\text{\AA}^2$ )*

|     | $x$         | $y$         | $z$          | $U_{\text{iso}}^*/U_{\text{eq}}$ | Occ. (<1) |
|-----|-------------|-------------|--------------|----------------------------------|-----------|
| C11 | 0.5427 (3)  | 0.44767 (7) | 0.83121 (16) | 0.1010 (7)                       |           |
| N1  | 0.8645 (10) | 0.8557 (2)  | 0.4503 (5)   | 0.1033 (15)                      |           |
| N2  | 1.0442 (9)  | 0.6789 (2)  | 0.3064 (4)   | 0.0871 (13)                      |           |
| C1  | 0.7656 (10) | 0.8069 (2)  | 0.4867 (5)   | 0.0759 (13)                      |           |
| C2  | 0.8952 (9)  | 0.6778 (2)  | 0.3795 (5)   | 0.0689 (12)                      |           |
| C3  | 0.6468 (8)  | 0.7433 (2)  | 0.5291 (4)   | 0.0662 (11)                      |           |
| C4  | 0.7097 (8)  | 0.6785 (2)  | 0.4738 (4)   | 0.0621 (11)                      |           |
| C5  | 0.5977 (8)  | 0.6167 (2)  | 0.5135 (4)   | 0.0707 (12)                      |           |
| H5A | 0.6393      | 0.5738      | 0.4785       | 0.085*                           |           |
| C6  | 0.4228 (9)  | 0.6196 (2)  | 0.6063 (5)   | 0.0761 (13)                      |           |
| C7  | 0.3573 (9)  | 0.6827 (3)  | 0.6596 (4)   | 0.0755 (13)                      |           |
| H7A | 0.2388      | 0.6837      | 0.7214       | 0.091*                           |           |
| C8  | 0.4698 (9)  | 0.7442 (2)  | 0.6201 (5)   | 0.0717 (12)                      |           |
| H8A | 0.4254      | 0.7869      | 0.6553       | 0.086*                           |           |

|      |             |             |             |           |          |
|------|-------------|-------------|-------------|-----------|----------|
| O1   | 0.297 (3)   | 0.5510 (8)  | 0.6161 (17) | 0.084 (4) | 0.54 (2) |
| C9   | 0.134 (4)   | 0.5396 (9)  | 0.719 (2)   | 0.098 (6) | 0.54 (2) |
| H9A  | 0.0513      | 0.5837      | 0.7401      | 0.118*    | 0.54 (2) |
| H9B  | -0.0127     | 0.5068      | 0.6922      | 0.118*    | 0.54 (2) |
| C10  | 0.294 (4)   | 0.5130 (10) | 0.8277 (15) | 0.101 (6) | 0.54 (2) |
| H10A | 0.1641      | 0.4968      | 0.8830      | 0.121*    | 0.54 (2) |
| H10B | 0.3863      | 0.5534      | 0.8694      | 0.121*    | 0.54 (2) |
| O1'  | 0.352 (4)   | 0.5616 (10) | 0.6669 (13) | 0.080 (4) | 0.46 (2) |
| C9'  | 0.255 (3)   | 0.5600 (6)  | 0.7816 (13) | 0.059 (4) | 0.46 (2) |
| H9C  | 0.0815      | 0.5851      | 0.7771      | 0.071*    | 0.46 (2) |
| H9D  | 0.3849      | 0.5819      | 0.8458      | 0.071*    | 0.46 (2) |
| C10' | 0.2133 (14) | 0.4813 (7)  | 0.8148 (17) | 0.070 (4) | 0.46 (2) |
| H10C | 0.0970      | 0.4576      | 0.7474      | 0.085*    | 0.46 (2) |
| H10D | 0.1318      | 0.4767      | 0.8930      | 0.085*    | 0.46 (2) |

*Atomic displacement parameters (Å<sup>2</sup>)*

|      | $U^{11}$    | $U^{22}$   | $U^{33}$    | $U^{12}$    | $U^{13}$    | $U^{23}$   |
|------|-------------|------------|-------------|-------------|-------------|------------|
| C11  | 0.0865 (10) | 0.0857 (9) | 0.1351 (14) | 0.0058 (7)  | 0.0306 (8)  | 0.0097 (8) |
| N1   | 0.114 (4)   | 0.077 (3)  | 0.121 (4)   | -0.011 (3)  | 0.024 (3)   | 0.005 (3)  |
| N2   | 0.091 (3)   | 0.088 (3)  | 0.090 (3)   | 0.007 (2)   | 0.041 (2)   | 0.009 (2)  |
| C1   | 0.074 (3)   | 0.070 (3)  | 0.084 (3)   | -0.001 (2)  | 0.012 (2)   | -0.008 (3) |
| C2   | 0.075 (3)   | 0.058 (2)  | 0.076 (3)   | 0.001 (2)   | 0.021 (2)   | 0.001 (2)  |
| C3   | 0.064 (2)   | 0.061 (2)  | 0.074 (3)   | 0.0028 (19) | 0.008 (2)   | 0.003 (2)  |
| C4   | 0.060 (2)   | 0.062 (2)  | 0.066 (3)   | 0.0064 (19) | 0.0145 (19) | 0.003 (2)  |
| C5   | 0.073 (3)   | 0.059 (2)  | 0.083 (3)   | 0.008 (2)   | 0.023 (2)   | 0.006 (2)  |
| C6   | 0.079 (3)   | 0.068 (3)  | 0.086 (3)   | 0.008 (2)   | 0.027 (2)   | 0.017 (2)  |
| C7   | 0.072 (3)   | 0.091 (3)  | 0.066 (3)   | 0.019 (2)   | 0.019 (2)   | 0.002 (2)  |
| C8   | 0.070 (3)   | 0.070 (3)  | 0.076 (3)   | 0.009 (2)   | 0.010 (2)   | -0.002 (2) |
| O1   | 0.115 (8)   | 0.067 (6)  | 0.083 (9)   | 0.010 (5)   | 0.062 (7)   | 0.012 (6)  |
| C9   | 0.088 (9)   | 0.096 (9)  | 0.120 (13)  | 0.007 (7)   | 0.053 (9)   | 0.033 (8)  |
| C10  | 0.073 (8)   | 0.141 (15) | 0.089 (8)   | 0.017 (10)  | 0.015 (7)   | 0.041 (10) |
| O1'  | 0.125 (8)   | 0.056 (5)  | 0.066 (8)   | -0.004 (5)  | 0.041 (8)   | -0.004 (6) |
| C9'  | 0.046 (6)   | 0.063 (7)  | 0.071 (8)   | -0.009 (5)  | 0.017 (6)   | -0.018 (5) |
| C10' | 0.051 (6)   | 0.056 (7)  | 0.109 (10)  | -0.020 (5)  | 0.029 (6)   | 0.009 (6)  |

*Geometric parameters (Å, °)*

|          |           |          |            |
|----------|-----------|----------|------------|
| C11—C10' | 1.726 (5) | C7—H7A   | 0.9300     |
| C11—C10  | 1.738 (5) | C8—H8A   | 0.9300     |
| N1—C1    | 1.137 (6) | O1—C9    | 1.450 (16) |
| N2—C2    | 1.132 (5) | C9—C10   | 1.41 (3)   |
| C1—C3    | 1.439 (6) | C9—H9A   | 0.9700     |
| C2—C4    | 1.435 (6) | C9—H9B   | 0.9700     |
| C3—C8    | 1.378 (6) | C10—H10A | 0.9700     |
| C3—C4    | 1.416 (5) | C10—H10B | 0.9700     |
| C4—C5    | 1.386 (6) | O1'—C9'  | 1.366 (5)  |
| C5—C6    | 1.386 (6) | C9'—C10' | 1.556 (19) |

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|           |            |                |            |
|-----------|------------|----------------|------------|
| C5—H5A    | 0.9300     | C9'—H9C        | 0.9700     |
| C6—O1'    | 1.344 (17) | C9'—H9D        | 0.9700     |
| C6—C7     | 1.382 (6)  | C10'—H10C      | 0.9700     |
| C6—O1     | 1.452 (15) | C10'—H10D      | 0.9700     |
| C7—C8     | 1.381 (6)  |                |            |
|           |            |                |            |
| N1—C1—C3  | 177.5 (5)  | C10—C9—H9A     | 109.3      |
| N2—C2—C4  | 178.3 (5)  | O1—C9—H9A      | 109.3      |
| C8—C3—C4  | 119.5 (4)  | C10—C9—H9B     | 109.3      |
| C8—C3—C1  | 121.6 (4)  | O1—C9—H9B      | 109.3      |
| C4—C3—C1  | 118.9 (4)  | H9A—C9—H9B     | 107.9      |
| C5—C4—C3  | 119.8 (4)  | C9—C10—C11     | 126.4 (13) |
| C5—C4—C2  | 121.0 (4)  | C9—C10—H10A    | 105.7      |
| C3—C4—C2  | 119.2 (4)  | C11—C10—H10A   | 105.7      |
| C4—C5—C6  | 119.1 (4)  | C9—C10—H10B    | 105.7      |
| C4—C5—H5A | 120.4      | C11—C10—H10B   | 105.7      |
| C6—C5—H5A | 120.4      | H10A—C10—H10B  | 106.2      |
| O1'—C6—C7 | 115.4 (7)  | C6—O1'—C9'     | 125.9 (13) |
| O1'—C6—C5 | 121.8 (8)  | O1'—C9'—C10'   | 107.1 (12) |
| C7—C6—C5  | 121.5 (4)  | O1'—C9'—H9C    | 110.3      |
| C7—C6—O1  | 128.9 (7)  | C10'—C9'—H9C   | 110.3      |
| C5—C6—O1  | 108.7 (7)  | O1'—C9'—H9D    | 110.3      |
| C8—C7—C6  | 119.2 (4)  | C10'—C9'—H9D   | 110.3      |
| C8—C7—H7A | 120.4      | H9C—C9'—H9D    | 108.5      |
| C6—C7—H7A | 120.4      | C9'—C10'—C11   | 103.4 (9)  |
| C3—C8—C7  | 120.9 (4)  | C9'—C10'—H10C  | 111.1      |
| C3—C8—H8A | 119.6      | C11—C10'—H10C  | 111.1      |
| C7—C8—H8A | 119.6      | C9'—C10'—H10D  | 111.1      |
| C6—O1—C9  | 117.8 (11) | C11—C10'—H10D  | 111.1      |
| C10—C9—O1 | 111.8 (13) | H10C—C10'—H10D | 109.0      |

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