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# 2-(3-Chloro-4-hydroxyphenyl)-*N*-(3,4-dimethoxyphenethyl)acetamide

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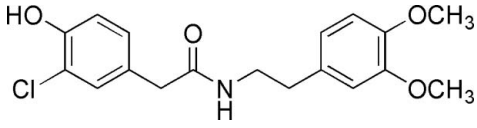
Key indicators: single-crystal X-ray study; $T$ = 295 K; mean $\sigma$(C–C) = 0.005 Å; $R$ factor = 0.057; $wR$ factor = 0.166; data-to-parameter ratio = 14.2.

The title compound, $C_{18}H_{20}ClNO_4$, was synthesized during the generation of a combinatorial library based on the fungal natural product 3-chloro-4-hydroxyphenylacetamide. It crystallizes as discrete molecules linked by intermolecular $C(9)$ chains of N—H···O and O—H···O hydrogen bonds which in turn combine to form chains of $R_2^2(20)$ rings.

## Related literature

For related literature, see: Bernstein *et al.* (1995); Davis *et al.* (2005, 2007).




## Experimental

### Crystal data

$C_{18}H_{20}ClNO_4$
$M_r$ = 349.80
Monoclinic, $P2_1/n$
$a$ = 12.329 (3) Å
$b$ = 12.839 (5) Å
$c$ = 11.062 (3) Å
$\beta$ = 92.18 (2)°
$V$ = 1749.8 (9) Å$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 0.24 mm$^{-1}$
$T$ = 295 K
0.35 × 0.35 × 0.15 mm

### Data collection

Rigaku AFC-7R diffractometer
Absorption correction: none
3463 measured reflections
3078 independent reflections
1596 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.034
3 standard reflections
every 150 reflections
intensity decay: 0.2%

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.056
$wR(F^2)$ = 0.165
$S$ = 0.99
3078 reflections
217 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.21 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.31 e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| N1—H1···O14$^{i}$ | 0.91 | 2.37 | 3.066 (4) | 134 |
| O4—H4···O8$^{i}$ | 0.90 | 1.74 | 2.616 (4) | 165 |

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

Data collection: *MSC/AFC7 Diffractometer Control for Windows* (Molecular Structure Corporation, 1999); cell refinement: *MSC/AFC7 Diffractometer Control for Windows*; data reduction: *TEXSAN for Windows* (Molecular Structure Corporation, 2001); program(s) used to solve structure: *TEXSAN for Windows*; program(s) used to refine structure: *TEXSAN for Windows* and *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *TEXSAN for Windows* and *PLATON* (Spek, 2003).

We acknowledge financial support of this work by Griffith University and the Eskitis Institute for Cell and Molecular Therapies.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: TK2268).

## References

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# supporting information

*Acta Cryst.* (2008). E**64**, o1057 [doi:10.1107/S1600536808013299]

## 2-(3-Chloro-4-hydroxyphenyl)-*N*-(3,4-dimethoxyphenethyl)acetamide

**Rohan A. Davis and Peter C. Healy**

### S1. Comment

The title compound (I) was synthesized during the generation of a combinatorial library based on the fungal natural product 3-chloro-4-hydroxyphenylacetamide (Davis *et al.*, 2005, 2007). Compound (I) was shown to display moderate cytotoxicity towards the human melanoma cell line MM96L with 94% inhibition at 100 $\mu$g ml$^{-1}$ (Davis *et al.*, 2007).

This compound crystallizes as discrete molecules with three planar components: the acetamide group (C7—C8(O8)—N1—C9), the 3-chloro-4-hydroxyphenyl group (C1—C7, O4, Cl3), and the 3,4-dimethoxybenzyl group (C11—C16, C10, O13—C17, O14—C18), Fig. 1. The C9—C10—C11—C16 and C2—C1—C7—C8 torsion angles are -91.8 (4)° and-78.9 (4)° respectively. In the crystal structure the amide (N1) and hydroxy (O4) groups form C(9) chains (Bernstein *et al.*, 1995) of intermolecular N—H···O and O—H···O hydrogen bonds with the methoxy oxygen (O14) and the carbonyl oxygen (O8), respectively (Table 1) which in turn combine to form chains of $R_2^2(20)$ rings (Fig. 2).

### S2. Experimental

Compound (I) was prepared as previously reported (Davis *et al.*, 2007). Crystals suitable for X-ray diffraction studies were obtained by slow evaporation of a n-hexane/ethyl acetate (1:1) solution of (I); m.p. 421–423 K.

### S3. Refinement

The carbon-bound H atoms were constrained as riding atoms with C—H = 0.95–0.96 Å. The amide and hydroxyl protons were located in difference Fourier maps and constrained with N—H, O—H = 0.90 Å. $U_{iso}$(H) values were set at 1.2$U_{eq}$ of the parent atom.

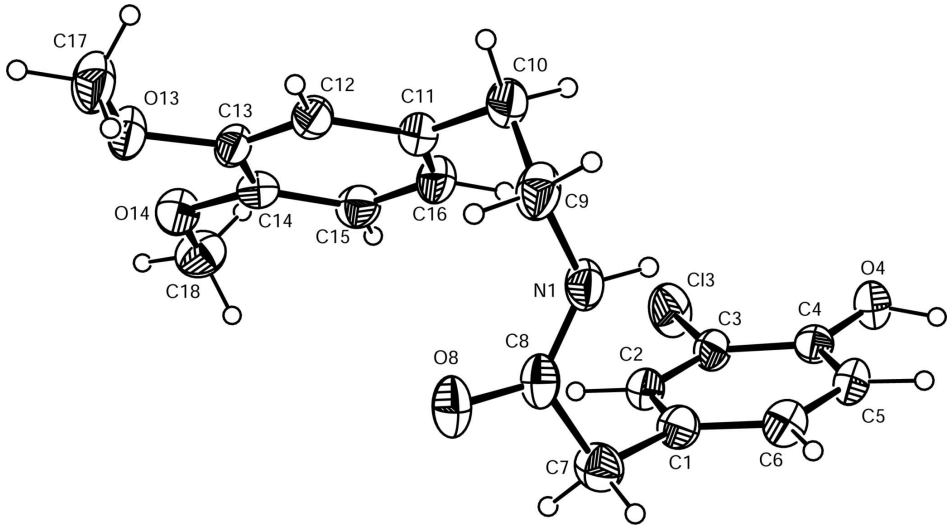


**Figure 1**

View of (I) with the atom numbering scheme. Displacement ellipsoids for non-H atoms are drawn at the 30% probability level.

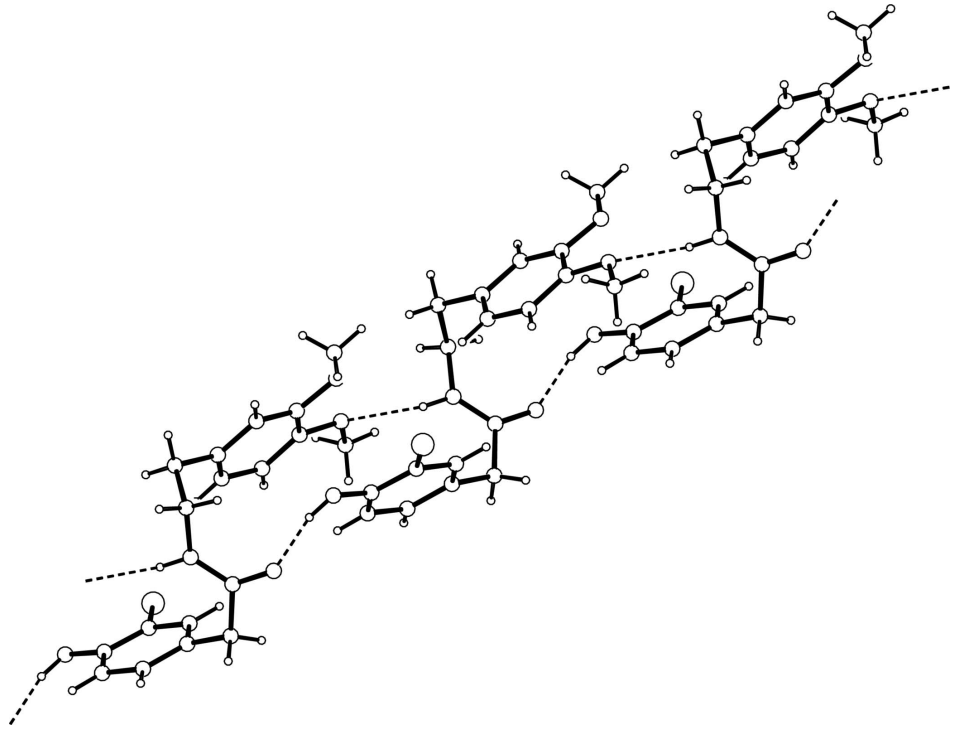

**Figure 2**

View of the intramolecular N—H···O and O—H···O hydrogen bonding (dashed lines) in (I).

### 2-(3-Chloro-4-hydroxyphenyl)-*N*-(3,4-dimethoxyphenethyl)acetamide

*Crystal data*

$C_{18}H_{20}ClNO_4$
$M_r = 349.80$

Monoclinic, $P2_1/n$
Hall symbol: -P 2yn

$a$ = 12.329 (3) Å
$b$ = 12.839 (5) Å
$c$ = 11.062 (3) Å
$\beta$ = 92.18 (2)°
$V$ = 1749.8 (9) Å$^3$
$Z$ = 4
$F$(000) = 736
$D_x$ = 1.328 Mg m$^{-3}$

Mo $K\alpha$ radiation, $\lambda$ = 0.7107 Å
Cell parameters from 25 reflections
$\theta$ = 12.7–16.7°
$\mu$ = 0.24 mm$^{-1}$
$T$ = 295 K
Prismatic, colourless
0.35 × 0.35 × 0.15 mm

*Data collection*

Rigaku AFC-7R diffractometer
Radiation source: Rigaku rotating anode
Graphite monochromator
$\omega$–2$\theta$ scans
3463 measured reflections
3078 independent reflections
1596 reflections with $I > 2\sigma(I)$

$R_{int}$ = 0.034
$\theta_{max}$ = 25.0°, $\theta_{min}$ = 2.5°
$h$ = −14→6
$k$ = 0→15
$l$ = −13→13
3 standard reflections every 150 reflections
intensity decay: 0.2%

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.056
$wR(F^2)$ = 0.165
$S$ = 0.99
3078 reflections
217 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-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0802P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.21 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.31 e Å$^{-3}$

*Special details*

**Experimental**. The scan width was (1.68 + 0.30tan$\theta$)° with an $\omega$ scan speed of 16° per minute (up to 4 scans to achieve I/$\sigma$(I) > 10). Stationary background counts were recorded at each end of the scan, and the scan time:background time ratio was 2:1.

**Geometry**. Bond distances, angles *etc*. have been calculated using the rounded fractional coordinates. All su's are estimated from the variances of the (full) variance-covariance matrix. The cell e.s.d.'s are taken into account in the estimation of distances, angles and torsion angles

**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$^2$)*

| | $x$ | $y$ | $z$ | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| Cl3 | 0.45169 (9) | 0.55050 (8) | 0.30062 (9) | 0.0775 (4) |
| O4 | 0.28934 (19) | 0.46680 (18) | 0.13154 (19) | 0.0576 (8) |
| O8 | 0.5970 (2) | 0.0875 (2) | 0.5455 (2) | 0.0730 (10) |
| O13 | 1.02796 (18) | 0.23926 (19) | 0.5658 (2) | 0.0617 (9) |
| O14 | 0.94460 (19) | 0.41834 (18) | 0.6082 (2) | 0.0596 (9) |
| N1 | 0.5696 (2) | 0.1267 (2) | 0.3474 (3) | 0.0582 (11) |
| C1 | 0.3920 (3) | 0.2566 (3) | 0.3989 (3) | 0.0520 (12) |

| | | | | |
|---|---|---|---|---|
| C2 | 0.4348 (3) | 0.3564 (3) | 0.3927 (3) | 0.0531 (14) |
| C3 | 0.3990 (3) | 0.4245 (3) | 0.3042 (3) | 0.0488 (12) |
| C4 | 0.3205 (3) | 0.3961 (3) | 0.2174 (3) | 0.0449 (11) |
| C5 | 0.2765 (3) | 0.2977 (3) | 0.2247 (3) | 0.0506 (12) |
| C6 | 0.3118 (3) | 0.2284 (3) | 0.3136 (3) | 0.0544 (12) |
| C7 | 0.4368 (3) | 0.1806 (3) | 0.4917 (3) | 0.0640 (16) |
| C8 | 0.5425 (3) | 0.1284 (3) | 0.4612 (4) | 0.0555 (12) |
| C9 | 0.6665 (3) | 0.0772 (3) | 0.3040 (4) | 0.0674 (16) |
| C10 | 0.7448 (3) | 0.1555 (3) | 0.2523 (3) | 0.0669 (14) |
| C11 | 0.7947 (3) | 0.2290 (3) | 0.3448 (3) | 0.0541 (14) |
| C12 | 0.8890 (3) | 0.1991 (3) | 0.4103 (3) | 0.0527 (12) |
| C13 | 0.9362 (3) | 0.2626 (3) | 0.4968 (3) | 0.0488 (11) |
| C14 | 0.8904 (3) | 0.3599 (3) | 0.5209 (3) | 0.0506 (11) |
| C15 | 0.7965 (3) | 0.3898 (3) | 0.4579 (3) | 0.0576 (12) |
| C16 | 0.7501 (3) | 0.3244 (3) | 0.3708 (3) | 0.0613 (14) |
| C17 | 1.0790 (3) | 0.1422 (3) | 0.5422 (4) | 0.0773 (17) |
| C18 | 0.8999 (3) | 0.5175 (3) | 0.6355 (4) | 0.0728 (17) |
| H1 | 0.52920 | 0.15340 | 0.28470 | 0.0690* |
| H2 | 0.48940 | 0.37790 | 0.45040 | 0.0640* |
| H4 | 0.22920 | 0.44390 | 0.09130 | 0.0690* |
| H5 | 0.22100 | 0.27710 | 0.16780 | 0.0600* |
| H6 | 0.28090 | 0.16070 | 0.31640 | 0.0650* |
| H7A | 0.44830 | 0.21720 | 0.56580 | 0.0770* |
| H7B | 0.38400 | 0.12770 | 0.50170 | 0.0770* |
| H9A | 0.70220 | 0.04160 | 0.36940 | 0.0810* |
| H9B | 0.64580 | 0.02860 | 0.24260 | 0.0810* |
| H10A | 0.80160 | 0.11820 | 0.21600 | 0.0800* |
| H10B | 0.70660 | 0.19560 | 0.19240 | 0.0800* |
| H12 | 0.92100 | 0.13350 | 0.39450 | 0.0630* |
| H15 | 0.76400 | 0.45500 | 0.47430 | 0.0690* |
| H16 | 0.68580 | 0.34580 | 0.32790 | 0.0730* |
| H17A | 1.14550 | 0.13770 | 0.58820 | 0.0930* |
| H17B | 1.03260 | 0.08680 | 0.56430 | 0.0930* |
| H17C | 1.09290 | 0.13730 | 0.45860 | 0.0930* |
| H18A | 0.82940 | 0.50840 | 0.66600 | 0.0870* |
| H18B | 0.94520 | 0.55140 | 0.69460 | 0.0870* |
| H18C | 0.89500 | 0.55850 | 0.56410 | 0.0870* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| Cl3 | 0.0899 (8) | 0.0688 (7) | 0.0723 (7) | −0.0335 (6) | −0.0161 (5) | 0.0010 (5) |
| O4 | 0.0595 (15) | 0.0560 (15) | 0.0560 (14) | −0.0045 (12) | −0.0141 (12) | 0.0018 (12) |
| O8 | 0.0645 (17) | 0.0801 (19) | 0.0720 (17) | 0.0059 (15) | −0.0273 (14) | 0.0085 (15) |
| O13 | 0.0508 (15) | 0.0643 (17) | 0.0686 (16) | 0.0122 (13) | −0.0165 (12) | −0.0127 (13) |
| O14 | 0.0601 (16) | 0.0540 (16) | 0.0643 (15) | 0.0047 (13) | −0.0015 (12) | −0.0106 (12) |
| N1 | 0.0493 (19) | 0.067 (2) | 0.057 (2) | 0.0096 (16) | −0.0136 (15) | −0.0031 (16) |
| C1 | 0.050 (2) | 0.052 (2) | 0.054 (2) | 0.0073 (18) | 0.0011 (17) | 0.0026 (17) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C2 | 0.045 (2) | 0.064 (3) | 0.050 (2) | −0.0033 (18) | −0.0018 (16) | −0.0048 (18) |
| C3 | 0.047 (2) | 0.049 (2) | 0.050 (2) | −0.0101 (17) | −0.0029 (16) | −0.0042 (17) |
| C4 | 0.0438 (19) | 0.045 (2) | 0.0461 (19) | 0.0037 (17) | 0.0030 (15) | −0.0058 (16) |
| C5 | 0.040 (2) | 0.050 (2) | 0.061 (2) | −0.0008 (16) | −0.0082 (17) | −0.0053 (17) |
| C6 | 0.053 (2) | 0.045 (2) | 0.065 (2) | 0.0008 (17) | 0.0003 (18) | −0.0048 (18) |
| C7 | 0.069 (3) | 0.063 (3) | 0.060 (2) | 0.012 (2) | 0.0017 (19) | 0.0085 (19) |
| C8 | 0.056 (2) | 0.047 (2) | 0.062 (2) | −0.0032 (18) | −0.017 (2) | −0.0027 (19) |
| C9 | 0.056 (2) | 0.066 (3) | 0.079 (3) | 0.014 (2) | −0.013 (2) | −0.018 (2) |
| C10 | 0.051 (2) | 0.091 (3) | 0.058 (2) | 0.013 (2) | −0.0077 (18) | −0.010 (2) |
| C11 | 0.045 (2) | 0.068 (3) | 0.049 (2) | 0.0019 (19) | −0.0020 (16) | −0.0011 (18) |
| C12 | 0.045 (2) | 0.060 (2) | 0.053 (2) | 0.0025 (17) | 0.0000 (17) | −0.0080 (18) |
| C13 | 0.0361 (19) | 0.056 (2) | 0.054 (2) | 0.0038 (17) | −0.0033 (16) | 0.0037 (17) |
| C14 | 0.047 (2) | 0.058 (2) | 0.0474 (19) | −0.0014 (18) | 0.0082 (16) | 0.0020 (18) |
| C15 | 0.055 (2) | 0.057 (2) | 0.061 (2) | 0.0103 (19) | 0.0056 (19) | 0.0072 (19) |
| C16 | 0.048 (2) | 0.072 (3) | 0.063 (2) | 0.010 (2) | −0.0076 (18) | 0.008 (2) |
| C17 | 0.065 (3) | 0.078 (3) | 0.087 (3) | 0.025 (2) | −0.022 (2) | −0.017 (2) |
| C18 | 0.071 (3) | 0.065 (3) | 0.083 (3) | 0.004 (2) | 0.010 (2) | −0.013 (2) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| Cl3—C3 | 1.744 (4) | C12—C13 | 1.370 (5) |
| O4—C4 | 1.359 (4) | C13—C14 | 1.401 (5) |
| O8—C8 | 1.245 (5) | C14—C15 | 1.383 (5) |
| O13—C13 | 1.374 (4) | C15—C16 | 1.385 (5) |
| O13—C17 | 1.425 (5) | C2—H2 | 0.9500 |
| O14—C14 | 1.376 (4) | C5—H5 | 0.9500 |
| O14—C18 | 1.424 (5) | C6—H6 | 0.9500 |
| O4—H4 | 0.9000 | C7—H7A | 0.9500 |
| N1—C9 | 1.451 (5) | C7—H7B | 0.9500 |
| N1—C8 | 1.315 (5) | C9—H9A | 0.9500 |
| N1—H1 | 0.9100 | C9—H9B | 0.9500 |
| C1—C2 | 1.388 (5) | C10—H10A | 0.9500 |
| C1—C7 | 1.506 (5) | C10—H10B | 0.9500 |
| C1—C6 | 1.389 (5) | C12—H12 | 0.9500 |
| C2—C3 | 1.373 (5) | C15—H15 | 0.9500 |
| C3—C4 | 1.386 (5) | C16—H16 | 0.9500 |
| C4—C5 | 1.379 (5) | C17—H17A | 0.9500 |
| C5—C6 | 1.384 (5) | C17—H17B | 0.9500 |
| C7—C8 | 1.515 (5) | C17—H17C | 0.9500 |
| C9—C10 | 1.520 (5) | C18—H18A | 0.9500 |
| C10—C11 | 1.506 (5) | C18—H18B | 0.9500 |
| C11—C16 | 1.377 (5) | C18—H18C | 0.9500 |
| C11—C12 | 1.400 (5) | | |
| | | | |
| Cl3···O4 | 2.894 (3) | C14···H18C$^{vii}$ | 3.0300 |
| Cl3···C12$^{i}$ | 3.646 (4) | C15···H18A | 2.7800 |
| Cl3···H12$^{i}$ | 2.9200 | C15···H9B$^{i}$ | 2.9500 |
| Cl3···H17C$^{i}$ | 3.1100 | C15···H18C | 2.7300 |

| | | | |
|---|---|---|---|
| Cl3···H2$^{ii}$ | 2.9700 | C17···H12 | 2.5000 |
| O4···Cl3 | 2.894 (3) | C17···H10B$^{iv}$ | 3.0600 |
| O4···C17$^{i}$ | 3.411 (5) | C18···H15 | 2.5300 |
| O4···O8$^{iii}$ | 2.616 (4) | H1···C1 | 2.5200 |
| O8···C4$^{iv}$ | 3.295 (4) | H1···C6 | 2.8800 |
| O8···C5$^{iv}$ | 3.266 (5) | H1···H10B | 2.5100 |
| O8···O4$^{iv}$ | 2.616 (4) | H1···O13$^{iii}$ | 2.7900 |
| O8···C8$^{v}$ | 3.262 (5) | H1···O14$^{iii}$ | 2.3700 |
| O13···O14 | 2.569 (3) | H2···H7A | 2.4900 |
| O14···C6$^{iv}$ | 3.417 (4) | H2···Cl3$^{ii}$ | 2.9700 |
| O14···O13 | 2.569 (3) | H4···H5 | 2.3100 |
| O14···N1$^{iv}$ | 3.066 (4) | H4···O8$^{iii}$ | 1.7400 |
| O4···H17C$^{i}$ | 2.8300 | H4···C8$^{iii}$ | 2.8200 |
| O4···H18A$^{ii}$ | 2.7400 | H4···H9A$^{iii}$ | 2.4700 |
| O4···H6$^{vi}$ | 2.7100 | H5···H4 | 2.3100 |
| O8···H9A | 2.4500 | H5···O8$^{iii}$ | 2.6500 |
| O8···H4$^{iv}$ | 1.7400 | H6···H7B | 2.4100 |
| O8···H7B$^{v}$ | 2.8200 | H6···O4$^{ix}$ | 2.7100 |
| O8···H5$^{iv}$ | 2.6500 | H7A···H2 | 2.4900 |
| O13···H1$^{iv}$ | 2.7900 | H7B···H6 | 2.4100 |
| O13···H10B$^{iv}$ | 2.7000 | H7B···O8$^{v}$ | 2.8200 |
| O14···H18C$^{vii}$ | 2.8100 | H9A···O8 | 2.4500 |
| O14···H1$^{iv}$ | 2.3700 | H9A···C12 | 3.0900 |
| N1···C2 | 3.431 (5) | H9A···H4$^{iv}$ | 2.4700 |
| N1···C6 | 3.443 (5) | H9B···C15$^{viii}$ | 2.9500 |
| N1···C16 | 3.379 (5) | H10A···H12 | 2.4300 |
| N1···O14$^{iii}$ | 3.066 (4) | H10B···H1 | 2.5100 |
| C2···N1 | 3.431 (5) | H10B···H16 | 2.4600 |
| C4···C18$^{ii}$ | 3.406 (5) | H10B···O13$^{iii}$ | 2.7000 |
| C4···O8$^{iii}$ | 3.295 (4) | H10B···C17$^{iii}$ | 3.0600 |
| C4···C13$^{iii}$ | 3.522 (5) | H10B···H17A$^{iii}$ | 2.5300 |
| C5···C14$^{iii}$ | 3.375 (5) | H12···C17 | 2.5000 |
| C5···C13$^{iii}$ | 3.348 (5) | H12···H10A | 2.4300 |
| C5···O8$^{iii}$ | 3.266 (5) | H12···H17B | 2.3600 |
| C6···C14$^{iii}$ | 3.598 (5) | H12···H17C | 2.2100 |
| C6···N1 | 3.443 (5) | H12···Cl3$^{viii}$ | 2.9200 |
| C6···O14$^{iii}$ | 3.417 (4) | H15···C18 | 2.5300 |
| C8···C8$^{v}$ | 3.574 (6) | H15···H18A | 2.3400 |
| C8···O8$^{v}$ | 3.262 (5) | H15···H18C | 2.2900 |
| C12···Cl3$^{viii}$ | 3.646 (4) | H16···H10B | 2.4600 |
| C13···C4$^{iv}$ | 3.522 (5) | H17A···H10B$^{iv}$ | 2.5300 |
| C13···C5$^{iv}$ | 3.348 (5) | H17B···C12 | 2.8100 |
| C14···C18$^{vii}$ | 3.535 (6) | H17B···H12 | 2.3600 |
| C14···C6$^{iv}$ | 3.598 (5) | H17C···C12 | 2.6700 |
| C14···C5$^{iv}$ | 3.375 (5) | H17C···H12 | 2.2100 |
| C16···N1 | 3.379 (5) | H17C···Cl3$^{viii}$ | 3.1100 |
| C17···O4$^{viii}$ | 3.411 (5) | H17C···O4$^{viii}$ | 2.8300 |
| C18···C4$^{ii}$ | 3.406 (5) | H18A···C15 | 2.7800 |

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|---|---|---|---|
| C18···C14$^{vii}$ | 3.535 (6) | H18A···H15 | 2.3400 |
| C1···H1 | 2.5200 | H18A···O4$^{ii}$ | 2.7400 |
| C3···H18A$^{ii}$ | 2.9800 | H18A···C3$^{ii}$ | 2.9800 |
| C4···H18A$^{ii}$ | 2.6000 | H18A···C4$^{ii}$ | 2.6000 |
| C5···H18A$^{ii}$ | 3.0800 | H18A···C5$^{ii}$ | 3.0800 |
| C6···H1 | 2.8800 | H18C···C15 | 2.7300 |
| C8···H4$^{iv}$ | 2.8200 | H18C···H15 | 2.2900 |
| C12···H17B | 2.8100 | H18C···O14$^{vii}$ | 2.8100 |
| C12···H17C | 2.6700 | H18C···C14$^{vii}$ | 3.0300 |
| C12···H9A | 3.0900 | | |

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|---|---|---|---|
| C13—O13—C17 | 116.7 (3) | C4—C5—H5 | 119.00 |
| C14—O14—C18 | 117.1 (3) | C6—C5—H5 | 119.00 |
| C4—O4—H4 | 109.00 | C1—C6—H6 | 120.00 |
| C8—N1—C9 | 124.5 (3) | C5—C6—H6 | 120.00 |
| C9—N1—H1 | 110.00 | C1—C7—H7A | 108.00 |
| C8—N1—H1 | 125.00 | C1—C7—H7B | 108.00 |
| C2—C1—C7 | 120.1 (3) | C8—C7—H7A | 108.00 |
| C6—C1—C7 | 121.8 (3) | C8—C7—H7B | 108.00 |
| C2—C1—C6 | 118.0 (3) | H7A—C7—H7B | 109.00 |
| C1—C2—C3 | 120.7 (3) | N1—C9—H9A | 109.00 |
| Cl3—C3—C2 | 119.8 (3) | N1—C9—H9B | 109.00 |
| Cl3—C3—C4 | 118.6 (3) | C10—C9—H9A | 109.00 |
| C2—C3—C4 | 121.6 (4) | C10—C9—H9B | 109.00 |
| O4—C4—C3 | 118.7 (3) | H9A—C9—H9B | 109.00 |
| O4—C4—C5 | 123.5 (3) | C9—C10—H10A | 108.00 |
| C3—C4—C5 | 117.8 (3) | C9—C10—H10B | 108.00 |
| C4—C5—C6 | 121.1 (3) | C11—C10—H10A | 108.00 |
| C1—C6—C5 | 120.8 (4) | C11—C10—H10B | 108.00 |
| C1—C7—C8 | 115.5 (3) | H10A—C10—H10B | 109.00 |
| O8—C8—N1 | 124.1 (3) | C11—C12—H12 | 119.00 |
| O8—C8—C7 | 117.7 (4) | C13—C12—H12 | 119.00 |
| N1—C8—C7 | 118.2 (3) | C14—C15—H15 | 120.00 |
| N1—C9—C10 | 112.2 (3) | C16—C15—H15 | 120.00 |
| C9—C10—C11 | 114.1 (3) | C11—C16—H16 | 119.00 |
| C10—C11—C16 | 122.9 (3) | C15—C16—H16 | 119.00 |
| C10—C11—C12 | 119.3 (3) | O13—C17—H17A | 109.00 |
| C12—C11—C16 | 117.8 (3) | O13—C17—H17B | 109.00 |
| C11—C12—C13 | 121.4 (4) | O13—C17—H17C | 110.00 |
| O13—C13—C12 | 125.3 (3) | H17A—C17—H17B | 109.00 |
| O13—C13—C14 | 114.7 (3) | H17A—C17—H17C | 109.00 |
| C12—C13—C14 | 120.0 (3) | H17B—C17—H17C | 109.00 |
| O14—C14—C15 | 125.3 (3) | O14—C18—H18A | 109.00 |
| O14—C14—C13 | 115.6 (3) | O14—C18—H18B | 109.00 |
| C13—C14—C15 | 119.2 (3) | O14—C18—H18C | 109.00 |
| C14—C15—C16 | 119.9 (4) | H18A—C18—H18B | 109.00 |
| C11—C16—C15 | 121.8 (3) | H18A—C18—H18C | 109.00 |
| C1—C2—H2 | 120.00 | H18B—C18—H18C | 110.00 |

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|---|---|---|---|
| C3—C2—H2 | 120.00 | | |
| | | | |
| C17—O13—C13—C12 | 1.8 (5) | C3—C4—C5—C6 | −1.9 (5) |
| C17—O13—C13—C14 | −178.4 (3) | C4—C5—C6—C1 | 0.7 (6) |
| C18—O14—C14—C13 | −179.7 (3) | C1—C7—C8—O8 | 161.4 (3) |
| C18—O14—C14—C15 | 0.1 (5) | C1—C7—C8—N1 | −20.3 (5) |
| C9—N1—C8—O8 | 0.0 (6) | N1—C9—C10—C11 | 66.8 (4) |
| C9—N1—C8—C7 | −178.2 (3) | C9—C10—C11—C12 | 86.5 (4) |
| C8—N1—C9—C10 | −115.1 (4) | C9—C10—C11—C16 | −91.8 (4) |
| C6—C1—C2—C3 | −0.4 (5) | C10—C11—C12—C13 | −178.9 (3) |
| C7—C1—C2—C3 | 176.3 (3) | C16—C11—C12—C13 | −0.5 (5) |
| C2—C1—C6—C5 | 0.5 (5) | C10—C11—C16—C15 | 178.8 (3) |
| C7—C1—C6—C5 | −176.1 (3) | C12—C11—C16—C15 | 0.5 (5) |
| C2—C1—C7—C8 | −78.9 (4) | C11—C12—C13—O13 | 179.5 (3) |
| C6—C1—C7—C8 | 97.7 (4) | C11—C12—C13—C14 | −0.3 (5) |
| C1—C2—C3—Cl3 | 178.4 (3) | O13—C13—C14—O14 | 1.0 (4) |
| C1—C2—C3—C4 | −0.9 (6) | O13—C13—C14—C15 | −178.7 (3) |
| Cl3—C3—C4—O4 | 1.3 (5) | C12—C13—C14—O14 | −179.2 (3) |
| Cl3—C3—C4—C5 | −177.3 (3) | C12—C13—C14—C15 | 1.1 (5) |
| C2—C3—C4—O4 | −179.4 (3) | O14—C14—C15—C16 | 179.2 (3) |
| C2—C3—C4—C5 | 2.0 (5) | C13—C14—C15—C16 | −1.1 (5) |
| O4—C4—C5—C6 | 179.5 (3) | C14—C15—C16—C11 | 0.3 (5) |

Symmetry codes: (i) −*x*+3/2, *y*+1/2, −*z*+1/2; (ii) −*x*+1, −*y*+1, −*z*+1; (iii) *x*−1/2, −*y*+1/2, *z*−1/2; (iv) *x*+1/2, −*y*+1/2, *z*+1/2; (v) −*x*+1, −*y*, −*z*+1; (vi) −*x*+1/2, *y*+1/2, −*z*+1/2; (vii) −*x*+2, −*y*+1, −*z*+1; (viii) −*x*+3/2, *y*−1/2, −*z*+1/2; (ix) −*x*+1/2, *y*−1/2, −*z*+1/2.

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N1—H1···O14$^{iii}$ | 0.91 | 2.37 | 3.066 (4) | 134 |
| O4—H4···O8$^{iii}$ | 0.90 | 1.74 | 2.616 (4) | 165 |

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