Crystal structure and Hirshfeld surface analysis of 2-chloro-N-(4-methoxyphenyl)acetamide

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In the title molecule, C9H10ClNO2, the methoxy group lies very close to the plane of the phenyl ring while the acetamido group is twisted out of this plane by 28.87 (5)°. In the crystal, a three-dimensional structure is generated by N—H···O, C—H···O and C—H···Cl hydrogen bonds plus C—H···π(ring) interactions. A Hirshfeld surface analysis of the intermolecular interactions was performed and indicated that C—H···C interactions make the largest contribution to the surface area (33.4%).

1. Chemical context

Amides play a very important role in organic synthesis, including the production of medicines, functional materials, and bioactive molecules (Alcaide et al., 2007; Zhang et al., 2012; García-Álvarez et al., 2013; Ramli & Essassi, 2015; Álvarez-Pérez et al., 2019). In particular, N-arylacetamides are significant intermediates for the synthesis of medicinal, agrochemical, and pharmaceutical compounds (Beccalli et al., 2007; Valeur & Bradley, 2009; Allen & Williams, 2011; Missiou et al., 2021, 2022a,b,c). Given the wide range of therapeutic applications for such compounds, and in a continuation of our research efforts to synthesize more N-arylacetamides (Missiou et al., 2020; Guerrab et al., 2021), we report the synthesis, molecular and crystal structure and Hirshfeld surface analysis of the title compound, 2-chloro-N-(4-methoxyphenyl)acetamide.

2. Structural commentary

The methoxy group lies close to the mean plane of the phenyl ring C3–C8, as indicated by the C7—C6—O2—C9 torsion angle of −174.61 (10)° and atom C9 deviating by only 0.065 (1) Å from the mean plane through the C3–C8 ring. In contrast, the acetamido group is rotated out of the above
plane with the dihedral angle between the mean plane through the C3–C8 ring and that defined by N1/C2/C1/O1 being 28.87 (5)° (Fig. 1). The sum of the angles about N1 is 360.0 (9)°, indicating it to be planar (sp² hybridization). The Cl1—C1—C2—O1 torsion angle is 52.89 (12)°, illustrating a + synclinal (+ gauche) conformation about the C1—C2 bond. This places atom Cl1 at 1.299 (1) Å from the plane defined by C1, C2, N1 and O1.

3. Supramolecular features

In the crystal, N1—H1⋯O1 hydrogen bonds (Table 1) form helical chains along the 2₁ axes. These chains are linked by C1—H1A⋯O2 hydrogen bonds (Table 1), forming layers of molecules parallel to the ab plane (Fig. 2). The layers are linked by weak C4—H4⋯Cl1 hydrogen bonds as well as by C9—H9B⋯Cg1 interactions (Table 1) to generate the final three-dimensional structure (Fig. 3). As the shortest distance between parallel phenyl rings is 5.1075 (7) Å, there are no π–π stacking interactions present.

4. Database survey

A search of the Cambridge Structural Database (CSD, updated to March 2022; Groom et al., 2016) using the fragment A (Fig. 4, R = undefined, X = halogen) yielded 15 hits of which 13 had X = Cl and R = OEt (DELZIE; Zhang et al., 2006), COOEt (HEGLOW; Behbehani & Ibrahim, 2012), F (JODQEZ; Kang et al., 2008), S(O)₂NH(C₃HNO(CH₃)) (NULZEC; Murtaza et al., 2019), SO₂NH₂ (PINXAO; Florke & Saeed, 2018; QUYRIM; Akkurt et al., 2010), SMe (QUGTEU; Mongkholkeaw et al., 2020), H (RIYWIG; Gowda et al., 2008), NO₂ (WEPGEE; Wen et al., 2006; WEPGEE01;...
Gowda et al., 2007a), Cl (WINSUI; Gowda et al., 2007b), MeCl (XABWEF; Ashraf et al., 2016) and Me (XICMA Y; Gowda et al., 2007c). The last two hits had $X = Br$ and $R = Br$ (FOWYIA; Gowda et al., 2009) and CH$_2$CH$_2$O$_2$CC(F)(SPh)(NO$_2$) (VAGCOV; Takeuchi et al., 1988). In general, the conformation of the haloacetamide portion is quite similar in all structures, as is the formation of infinite chains by N—H···O hydrogen bonds and these are comparable to the features found in the title structure. In DELZIE and XABWEF, C—H···C1/C1/C1/C25(ring) interactions assist in the packing, as also observed for the title molecule.

5. Hirshfeld surface analysis

The analysis was performed with CrystalExplorer 21.5 (Spackman et al., 2021) with the details of the pictorial output described in a recent publication (Tan et al., 2019). Fig. 5 shows the $d_{\text{norm}}$ surface for the asymmetric unit plotted over the range −0.5547 to 0.9665 arbitrary units together with two adjacent molecules that are part of one infinite chain and two in adjacent chains (cf. Fig. 2). The bright-red spots at the top and bottom indicate the N—H···O hydrogen bonds (blue arrows) while the fainter ones at the far right and left indicate the C—H···O hydrogen bonds linking the chains (curved black lines) while that below and to the right of the Cl atom represents the weak C—H···Cl hydrogen bonds. Fig. 6a is the fingerprint plot showing all intermolecular interactions while Fig. 6b–6d show these resolved into C···H/H···C (33.4%), O···H/H···O (19.5%) and Cl···H/H···Cl (20%) interactions, respectively.

6. Synthesis and crystallization

0.047 mol of 4-methoxyaniline were dissolved in 40 mL of pure acetic acid and put in an ice bath. Subsequently, chloroacetyl chloride (0.047 mol) was added portionwise under stirring. At the end of the reaction, a solution of sodium acetate (35 mL) was added and a solid precipitate appeared after 30 min of stirring at room temperature. The resulting solid was filtered and washed with cold water, dried and recrystallized from ethanol to give the title compound as colourless crystals.

Yield 80%, m.p. = 398.6–400.3 K, FT–IR (ATR, $\nu$, cm$^{-1}$) 3292 ($\nu$ N–H amide), 1029 ($\nu$ N–C amide), 1660 ($\nu$ C=O amide), 3073 ($\nu$ C–H$_{\text{arom}}$), 827 ($\nu$ C–Cl), 2959 ($\nu$ C–H$_{\text{CH}_3}$).$^1$H NMR (DMSO–d$_6$) $\delta$ ppm: 3.74 (3H, s, CH$_3$); 4.24 (2H, s, CH$_2$), 6.93–7.5 (4H, m, $J = 1.3$ Hz, H$_{\text{arom}}$), 10.23 (1H, s, NH).$^{13}$C NMR (DMSO–d$_6$) $\delta$ ppm: 43.48 (CH$_2$), 55.23 (CH$_3$), 131.53 (C$_{\text{arom}}$–N), 155.51 (C$_{\text{arom}}$–O), 113.92–120.92 (C$_{\text{arom}}$), 164.13 (C=O); HRMS (ESI–MS) ($m/z$) calculated for C$_9$H$_{10}$ClNO$_2$ 199.04, found 199.0105.

7. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. Hydrogen atoms attached to carbon were placed in idealized positions and included as
riding contributions with isotropic displacement parameters fixed at 1.2U_{eq}(C) (1.5 for the methyl group). The N-bound H atom was found in a difference-Fourier map and refined with a DFIX 0.91 0.01 instruction and an independent isotropic displacement parameter.

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References

Akkturk, M., Yalcin, S. P., Turkmen, H. & Buyukcungur, O. (2010). Acta Cryst. E66, o1596.
Alcaide, B., Almendros, P. & Aragoncillo, C. (2007). Chem. Rev. 107, 4437–4492.
Allen, C. L. & Williams, J. M. J. (2011). Chem. Soc. Rev. 40, 3405.
Alvarez-Perez, A., Estebanias, M. A., Izquierdo, S., Varela, J. A. & Saá, C. (2019). Org. Lett. 21, 5346–5350.
Ashraf, Z., Kim, D., Seo, S.-Y. & Kang, S. K. (2016). Acta Cryst. C72, 94–98.
Beccalli, E. M., Broggi, G., Martinelli, M. & Sottocornola, S. (2007). Chem. Rev. 107, 5318–5365.
Behbouhi, H. & Ibrahim, H. M. (2012). Molecules, 17, 6362–6385.
Brandenburg, K. & Putz, H. (2012). DIAMOND. Crystal Impact GbR, Bonn, Germany.
Buker (2020). APEX3 and SAINT. Bruker AXS LLC, Madison, Wisconsin, USA.
Florea, U. & Saeed, A. (2018). Private Communication (refcode PINXAO). CCDC, Cambridge, England.
Garcia-Alvarez, R., Crochet, P. & Cadierno, V. (2013). Green Chem. 15, 46–66.
Gowda, B. T., Foro, S. & Fuess, H. (2007a). Acta Cryst. E63, o2335–o2336.
Gowda, B. T., Foro, S. & Fuess, H. (2007b). Acta Cryst. E63, o4488.
Gowda, B. T., Foro, S. & Fuess, H. (2007c). Acta Cryst. E63, o2333–o2334.
Gowda, B. T., Kozišek, J., Tokarčík, M. & Fuess, H. (2008). Acta Cryst. E64, o987.
Gowda, B. T., Svoboda, I., Foro, S., Suchetan, P. A. & Fuess, H. (2009). Acta Cryst. E65, o1955.
Groom, C. R., Bruno, I. J., Lightfoot, M. P. & Ward, S. C. (2016). Acta Cryst. B72, 171–179.
Guerrab, W., Missioui, M., Zaoui, Y., Mague, J. T. & Ramli, Y. (2021). Z. Kristallogr. New Cryst. Struct. 236, 133–134.
Kang, S., Zeng, H., Li, H. & Wang, H. (2008). Acta Cryst. E64, o1194.
Krause, L., Herbst-Irmer, R., Sheldrick, G. M. & Stalke, D. (2015). J. Appl. Cryst. 48, 3–10.
Missioui, M., Guerrab, W., Mague, J. T. & Ramli, Y. (2020). Z. Kristallogr. New Cryst. Struct. 235, 1429–1430.
Missioui, M., Lgaz, H., Guerrab, W., Lee, H., Warad, I., Mague, J. T., Ali, I. H., Essassi, E. M. & Ramli, Y. (2022a). J. Mol. Struct. 1253, 132132.
Missioui, M., Mortada, S., Guerrab, W., Serdaroğlu, G., Kaya, S., Mague, J. T., Essassi, E. M., Faouzi, M. E. A. & Ramli, Y. (2021). J. Mol. Struct. 1239, 130484.
Missioui, M., Said, M. A., Demirtaş, G., Mague, J. T., Al-Sulami, A., Al-Kaff, N. S. & Ramli, Y. (2022b). Arab. J. Chem. 15, 103595.
Missioui, M., Said, M. A., Demirtaş, G., Mague, J. T. & Ramli, Y. (2022c). J. Mol. Struct. 1247, 131420.
Mongkolkeaw, S., Songasan, A., Duangthongyoo, T., Chainok, K., Suramit, S., Wattanathana, W. & Wannaler, B. (2020). Acta Cryst. E76, 594–598.
Murtaza, S., Altaf, A. A., Hamayun, M., Itikhar, K., Tahir, M. N., Tarig, J. & Faiz, K. (2019). Eur. J. Chem. 10, 358–366.
Ramli, Y. & Essassi, E. M. (2015). Adv. Chem. Res. 27, 109–160.
Sheldrick, G. M. (2008). Acta Cryst. A64, 112–122.
Sheldrick, G. M. (2015a). Acta Cryst. A71, 3–8.
Sheldrick, G. M. (2015b). Acta Cryst. A71, 3–8.
Spackman, P. R., Turner, M. J., McKinnon, J. J., Wolff, S. K., Grimwood, D. J., Jayatilaka, D. & Spackman, M. A. (2021). J. Appl. Cryst. 54, 1006–1011.
Takeuchi, Y., Nojiri, M., Koizumi, T. & Itakura, Y. (1988). Tetrahedron Lett. 29, 4727–4730.
Tan, S. L., Jotani, M. M. & Tiekink, E. R. T. (2019). Acta Cryst. E75, 308–318.
Valeur, E. & Bradley, M. (2009). Chem. Soc. Rev. 38, 606–631.
Wen, Y.-H., Li, X.-M., Xu, L.-L., Tang, X.-F. & Zhang, S.-S. (2006). Acta Cryst. E62, o4427–o4428.
Zhang, D., Zhao, X., Hou, J. & Li, Z. (2012). Chem. Rev. 112, 5271–5316.
Zhang, S.-S., Wen, H.-L., Li, X.-M., Xu, L.-L. & Wen, Y.-H. (2006). Acta Cryst. E62, o3412–o3413.
Crystalline structure and Hirshfeld surface analysis of 2-chloro-N-(4-methoxy-phenyl)acetamide

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Computing details

Data collection: APEX3 (Bruker, 2020); cell refinement: SAINT (Bruker, 2020); data reduction: SAINT (Bruker, 2020); program(s) used to solve structure: SHELXT (Sheldrick, 2015a); program(s) used to refine structure: SHELXL2018/1 (Sheldrick, 2015b); molecular graphics: DIAMOND (Brandenburg & Putz, 2012); software used to prepare material for publication: SHELXTL (Sheldrick, 2008).

2-Chloro-N-(4-methoxyphenyl)acetamide

Crystal data

C₉H₁₀ClNO₂

F(000) = 416

Mₚ = 199.63

Dₐ = 1.469 Mg m⁻³

Monoclinic, P₂₁/c

Mo Kα radiation, λ = 0.71073 Å

a = 10.0939 (5) Å

Cell parameters from 9903 reflections

b = 9.6423 (5) Å

θ = 2.2–31.1°

c = 10.2799 (5) Å

µ = 0.39 mm⁻¹

β = 115.531 (2)°

T = 172 K

V = 902.83 (8) Å³

Plate, colourless

Z = 4

0.29 × 0.25 × 0.09 mm

Data collection

Bruker D8 QUEST PHOTON 3 diffractometer

43210 measured reflections

Radiation source: fine-focus sealed tube

2508 independent reflections

Graphite monochromator

2θmax = 31.1°, θmin = 3.1°

Detector resolution: 7.3910 pixels mm⁻¹

h = −14→14

φ and ω scans

k = −13→13

l = −14→14

Absorption correction: numerical

(SADABS; Krause et al., 2015)

Refinement

Refinement on F²

Tmin = 0.91, Tmax = 0.97

Least-squares matrix: full

R[F² > 2σ(F²)] = 0.032

Primary atom site location: dual

wR(F²) = 0.091

Secondary atom site location: difference Fourier map

S = 1.10

Hydrogen site location: mixed

2871 reflections

H atoms treated by a mixture of independent and constrained refinement

123 parameters

w = 1/[σ(F²) + (0.0462P)² + 0.2203P]

1 restraint

where P = (F²+c(2)F²)/3
(Δ/σ)_{max} = 0.001
Δρ_{max} = 0.38 \text{ e Å}^{-3}

Special details
Experimental. The diffraction data were obtained from 7 sets of frames, each of width 0.5° in ω or φ, collected with scan parameters determined by the "strategy" routine in APEX3. The scan time was 6 sec/frame.

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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 > 2σ(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. H-atoms attached to carbon were placed in calculated positions (C—H = 0.95 - 0.99 Å) and were included as riding contributions with isotropic displacement parameters 1.2 - 1.5 times those of the attached atoms. That attached to nitrogen was placed in a location derived from a difference map and refined with a DFIX 0.91 0.01 instruction.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

|     | x      | y      | z      | U_{iso}*/U_{eq} |
|-----|--------|--------|--------|-----------------|
| Cl1 | 0.30989(3) | 0.41222(3) | 0.90827(3) | 0.03169(9)      |
| O1  | 0.43630(10) | 0.27410(8)  | 0.71560(10) | 0.03148(19)     |
| O2  | 0.99240(9)  | 0.34016(9)  | 0.56309(9)  | 0.03316(19)     |
| N1  | 0.53618(9)  | 0.48405(8)  | 0.70583(9)  | 0.02160(17)     |
| H1  | 0.5353(17)  | 0.5749(9)   | 0.7222(16)  | 0.034(4)*       |
| C1  | 0.32272(11) | 0.47958(10) | 0.75260(11) | 0.02423(19)     |
| H1A | 0.226444    | 0.470886    | 0.667909    | 0.029*          |
| H1B | 0.348725    | 0.579198    | 0.767150    | 0.029*          |
| C2  | 0.43834(11) | 0.40146(10) | 0.72459(10) | 0.02147(19)     |
| C3  | 0.64850(11) | 0.44121(10) | 0.66649(10) | 0.02036(18)     |
| C4  | 0.63724(11) | 0.32251(10) | 0.58566(11) | 0.02264(19)     |
| H4  | 0.552126    | 0.265906    | 0.555512    | 0.027*          |
| C5  | 0.75029(11) | 0.28609(10) | 0.54860(10) | 0.02332(19)     |
| H5  | 0.742024    | 0.204773    | 0.493437    | 0.028*          |
| C6  | 0.87484(11) | 0.36845(11) | 0.59218(11) | 0.02389(19)     |
| C7  | 0.88452(12) | 0.48995(11) | 0.66987(12) | 0.0275(2)       |
| H7  | 0.968462    | 0.547843    | 0.697762    | 0.033*          |
| C8  | 0.77218(12) | 0.52620(10) | 0.70637(11) | 0.0249(2)       |
| H8  | 0.779097    | 0.609174    | 0.758744    | 0.030*          |
| C9  | 0.97987(13) | 0.22369(13) | 0.47330(13) | 0.0325(2)       |
| H9A | 1.070019    | 0.214361    | 0.460154    | 0.049*          |
| H9B | 0.896160    | 0.237045    | 0.379304    | 0.049*          |
| H9C | 0.964803    | 0.139514    | 0.518658    | 0.049*          |

Atomic displacement parameters (Å^2)

|     | U_{11} | U_{22} | U_{33} | U_{12} | U_{13} | U_{23} |
|-----|--------|--------|--------|--------|--------|--------|
| Cl1 | 0.03574(16) | 0.03273(15) | 0.03447(15) | 0.00576(10) | 0.02258(12) | 0.00686(10) |
| O1  | 0.0429(5)   | 0.0163(3)  | 0.0466(5)  | −0.0005(3)  | 0.0300(4)   | 0.0011(3)  |

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O2 0.0278 (4) 0.0390 (5) 0.0389 (4) −0.0049 (3) 0.0202 (3) −0.0107 (3)
N1 0.0247 (4) 0.0154 (4) 0.0269 (4) −0.0001 (3) 0.0133 (3) −0.0006 (3)
C1 0.0259 (5) 0.0214 (4) 0.0278 (5) 0.0031 (3) 0.0139 (4) 0.0043 (4)
C2 0.0253 (4) 0.0181 (4) 0.0226 (4) 0.0010 (3) 0.0118 (4) 0.0022 (3)
C3 0.0228 (4) 0.0177 (4) 0.0213 (4) 0.0002 (3) 0.0102 (3) 0.0013 (3)
C4 0.0239 (4) 0.0204 (4) 0.0240 (4) −0.0033 (3) 0.0107 (3) −0.0022 (3)
C5 0.0275 (5) 0.0206 (4) 0.0232 (4) −0.0013 (3) 0.0121 (4) −0.0027 (3)
C6 0.0252 (4) 0.0255 (5) 0.0229 (4) −0.0010 (4) 0.0122 (4) −0.0002 (3)
C7 0.0278 (5) 0.0256 (5) 0.0320 (5) −0.0071 (4) 0.0156 (4) −0.0051 (4)
C8 0.0286 (5) 0.0194 (4) 0.0283 (5) −0.0041 (4) 0.0139 (4) −0.0042 (3)
C9 0.0336 (6) 0.0345 (6) 0.0336 (6) 0.0051 (4) 0.0184 (5) −0.0027 (4)

Geometric parameters (Å, °)

| Bond | Distance (Å) | Angle (°) |
|------|--------------|-----------|
| C1—C2 | 1.7828 (10) | C4—C5    | 1.3947 (14) |
| O1—C2 | 1.2310 (12) | C4—H4    | 0.9500      |
| O2—C6 | 1.3704 (13) | C5—C6    | 1.3880 (14) |
| O2—C9 | 1.4243 (14) | C5—H5    | 0.9500      |
| N1—C2 | 1.3457 (13) | C6—C7    | 1.3973 (15) |
| N1—H1 | 0.893 (9)   | C6—C7    | 1.3840 (15) |
| C1—C2 | 1.5172 (14) | C7—H7    | 0.9500      |
| C1—H1A| 0.9900      | C7—H7    | 0.9500      |
| C3—C4 | 1.3900 (13) | C8—H8    | 0.9800      |
| C3—C8 | 1.3989 (14) | C8—H8    | 0.9800      |
| C6—O2 | 117.06 (9)  | C6—C5—C4| 120.11 (9)  |
| C2—N1 | 126.46 (8)  | C6—C5—H5| 119.9       |
| C2—Cl1| 119.0 (10)  | C4—C5—H5| 119.9       |
| C3—N1 | 114.5 (10)  | O2—C6—C5| 124.39 (9)  |
| C2—Cl1| 110.42 (7)  | O2—C6—C7| 115.96 (9)  |
| C2—Cl1—H1A| 109.6 | C5—C6—C7| 119.65 (9)  |
| C1—Cl1—H1A| 109.6 | C8—C7—C6| 120.20 (9)  |
| C2—Cl1—H1B| 109.6 | C8—C7—H7| 119.9       |
| C1—Cl1—H1B| 109.6 | C6—C7—H7| 119.9       |
| H1A—Cl1—H1B| 108.1 | C7—C8—C3| 120.30 (9)  |
| O1—C2—N1 | 124.69 (9) | C7—C8—H8| 119.8       |
| O1—C2—Cl1| 121.35 (9) | C3—C8—H8| 119.8       |
| N1—C2—C1 | 113.91 (8) | O2—C9—H9A| 109.5       |
| C4—C3—C8 | 119.36 (9) | O2—C9—H9B| 109.5       |
| C4—C3—N1 | 122.71 (9) | H9A—C9—H9B| 109.5      |
| C8—C3—N1 | 117.88 (9) | O2—C9—H9C| 109.5       |
| C3—C4—C5 | 120.33 (9) | H9A—C9—H9C| 109.5      |
| C3—C4—H4 | 119.8 | H9B—C9—H9C| 109.5       |
| C5—C4—H4 | 119.8 | C9—O2—C6—C5| 4.72 (16)  |
C3—N1—C2—C1  −174.47 (9)  C9—O2—C6—C7  −174.61 (10)
C11—C1—C2—O1  52.89 (12)  C4—C5—C6—O2  178.93 (10)
C11—C1—C2—N1  −129.47 (8)  C4—C5—C6—C7  −1.77 (15)
C2—N1—C3—C4  27.78 (15)  O2—C6—C7—C8  −179.00 (10)
C2—N1—C3—C8  −154.97 (10)  C5—C6—C7—C8  1.64 (16)
C8—C3—C4—C5  2.04 (15)  C6—C7—C8—C3  0.34 (16)
N1—C3—C4—C5  179.25 (9)  C4—C3—C8—C7  −2.17 (15)
C3—C4—C5—C6  −0.08 (15)  N1—C3—C8—C7  −179.52 (9)

Hydrogen-bond geometry (Å, °)

| D—H···A | D—H | H···A | D···A | D—H···A |
|---------|------|-------|-------|---------|
| N1—H1···O1i | 0.89 (1) | 2.01 (1) | 2.8910 (11) | 171 (1) |
| C1—H1A···O2ii | 0.99 | 2.48 | 3.3347 (13) | 145 |
| C4—H4···Cl1iii | 0.95 | 2.83 | 3.7646 (10) | 167 |
| C9—H9B···Cg1iv | 0.98 | 2.72 | 3.5020 (13) | 137 |

Symmetry codes: (i) −x+1, y+1/2, −z+3/2; (ii) x−1, y, z; (iii) x, −y+1/2, z−1/2; (iv) x, −y−1/2, z−3/2.