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Two polymorphs of 1,8-dichloroanthracene

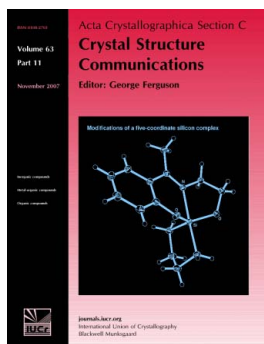
Peter Müller, Frank R. Fronczek, Stacey J. Smith, Teresa Mako and Mindy Levine

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Two polymorphs of 1,8-dichloroanthracene

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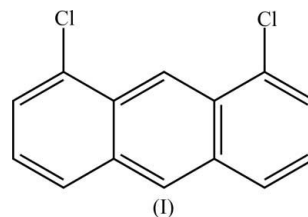
A second, monoclinic, polymorph of the title compound, C₁₄H₈Cl₂, has been found. In addition to the structure of this monoclinic form, the structure of the previously described orthorhombic form [Desvergne, Chekpo & Bouas-Laurent (1978). *J. Chem. Soc. Perkin Trans. 2*, pp. 84–87; Benites, Maverick & Fronczek (1996). *Acta Cryst. C* **52**, 647–648] has been redetermined at low temperature and using modern methods. The low-temperature structure of the orthorhombic form is of significantly higher quality than the previously published structure and additional details can be derived. A comparison of the crystal packing of the two forms with a focus on weak intermolecular C—H···Cl interactions shows the monoclinic structure to have one such interaction linking the molecules into infinite ribbons, while two crystallographically independent C—H···Cl interactions give rise to an interesting infinite three-dimensional network in the orthorhombic crystal form.

Comment

One of the starting materials used in the context of our ongoing research into energy transfer in ternary complexes is the title compound, 1,8-dichloroanthracene, (I). In an attempt to prepare an organometallic platinum clip, crystals of (I) were obtained and the structure was determined to be a previously unreported monoclinic polymorph of 1,8-dichloroanthracene.

The orthorhombic structure of (I) had originally been reported without coordinates by Desvergne *et al.* (1978) and was determined in the space group *Pnma* (with *Z* = 4) from Weissenberg photographs collected at room temperature. This structure was later corrected by Benites *et al.* (1996) to the space group *Pna*2₁ in essentially the same unit cell, determined based on point-detector data collected at room temperature, and refined against *F* values instead of *F*². To better compare the two polymorphs and also to redetermine

the structure using current methods, low-temperature data of the orthorhombic form were collected (the original bulk sample was still available) using a modern diffractometer equipped with an area detector. Figs. 1 and 2 show the molecules of the monoclinic and orthorhombic structures, respectively.



Comparison of the two structures shows that the monoclinic polymorph is somewhat denser than the orthorhombic crystal form (1.548 *versus* 1.533 Mg m⁻³), despite the fact that the orthorhombic unit cell was determined at a slightly lower temperature than the monoclinic one. To test for the possibility of a temperature-dependent phase transition, the unit cell of the monoclinic form was determined at 290 K from a fresh crystal (data not reported here) and was found to be the same monoclinic cell as that reported herein. It also appears that the orthorhombic polymorph does not convert to the denser monoclinic form over time, at least not on a decade timescale, as the orthorhombic crystals used for this study were grown over 16 years ago.

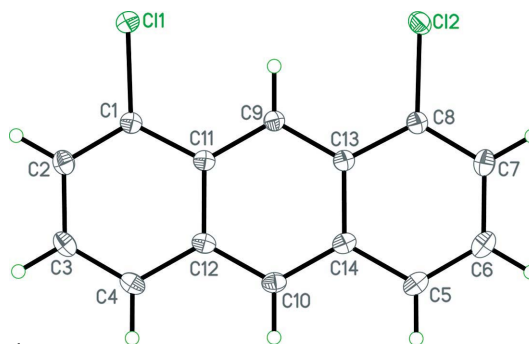


Figure 1

The molecular structure of (I) in the monoclinic form, showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

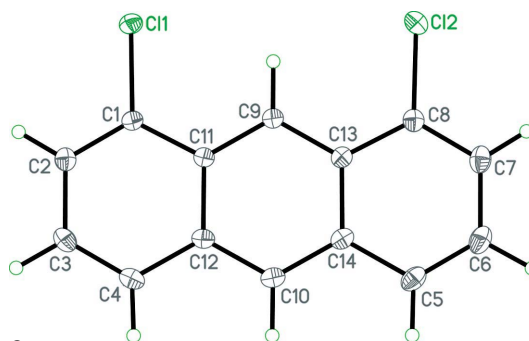
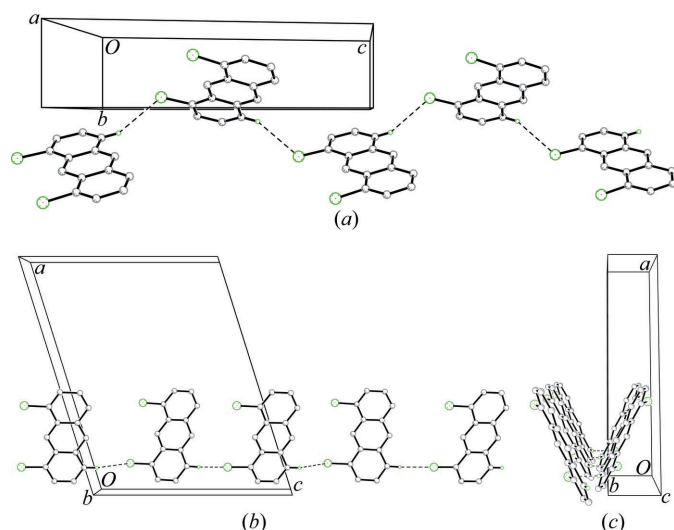


Figure 2

The molecular structure of (I) in the orthorhombic form, showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

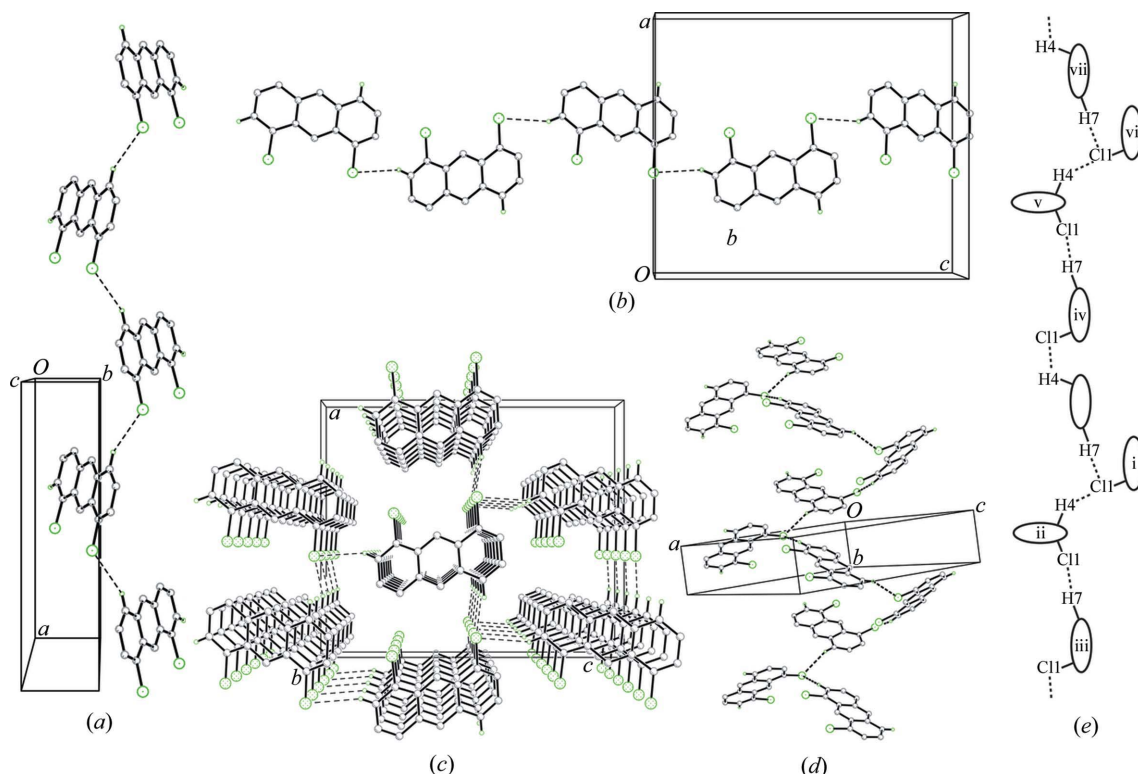

Figure 3

Three views of the infinite ribbon in the monoclinic structure of (I) generated by $C4-H4 \cdots Cl1^i$ interactions [symmetry code: (i) $x, -y + \frac{3}{2}, z + \frac{1}{2}$]. The symmetry operation relating the molecules in this ribbon is the c -glide, and the ribbon consequently extends along the c direction. $C-H \cdots Cl$ interactions are drawn as dashed lines. The views show the ribbon in projection along (a) a , (b) b and (c) c . The angle between neighboring molecules is $55.891 (14)^\circ$.

Besides the obvious intramolecular $C9-H9 \cdots Cl$ contacts occurring in both molecules, which are based more on molecular geometry than attractive forces between H and Cl atoms, both structures show weak intermolecular $C-H \cdots Cl$ interactions. Using the sum of the van der Waals radii (3.0 \AA ; Bondi, 1964) as a cut-off and limiting the search to $C-H \cdots Cl$ angles near a chemically sensible value of 150° , the monoclinic structure has one crystallographically independent van der Waals interaction involving atom Cl1, while the orthorhombic structure has two crystallographically independent interactions of this kind, both involving atom Cl1. These interactions are listed as $C-H \cdots Cl$ hydrogen bonds in Tables 1 and 2.

In the monoclinic polymorph, the $C4-H4 \cdots Cl1^i$ interactions [symmetry code: (i) $x, -y + \frac{3}{2}, z + \frac{1}{2}$] link the molecules into infinite zigzag ribbons extending along the c axis, in which neighboring molecules assume an angle of $55.891 (14)^\circ$ with respect to one another. When viewed in projection along the a axis, the zigzag nature of the ribbons is obvious (Fig. 3a). A projection along the b axis shows the ribbon from the side (Fig. 3b), while in a view along the axis of propagation, *i.e.* the c axis, a V-shaped groove presents itself (Fig. 3c).

The $C-H \cdots Cl$ interaction pattern found in the orthorhombic structure is somewhat more complex. The first of the


Figure 4

Various packing views of the orthorhombic structure. $C-H \cdots Cl$ interactions are drawn as dashed lines. (a) The zigzag ribbon extending along the a axis, generated by the hydrogen bonds of atom H4. The angle between neighboring molecules is $55.816 (14)^\circ$. (b) The zigzag chain extending along the c axis, generated by the hydrogen bonds of atom H7. The angle between neighboring molecules is $51.494 (13)^\circ$. (c) A packing plot in projection along the b axis, providing a view down the helices that are formed by a combination of the motifs shown in (a) and (b). (d) A side view of the helix, which is built using all four general positions of space group $Pna2_1$ for each full turn. (e) Schematics of the anatomy of the helix, showing two turns. The molecules are represented by oval shapes and only those atoms directly interacting with a neighboring molecule protrude from the oval. Each of the four molecules in every full turn of a given helix contributes to that helix in a different way: one molecule binds to its neighbors through atoms H4 and H7, a second one through atoms H4 and Cl1, a third through atoms H7 and Cl1, and the fourth only through atom Cl1. [Symmetry codes in the centers of the ovals in part (e): (i) $-x + 1, -y + 1, z - \frac{1}{2}$; (ii) $-x + \frac{1}{2}, y - \frac{3}{2}, z - \frac{1}{2}$; (iii) $x - \frac{1}{2}, -y - \frac{1}{2}, z$; (iv) $x - \frac{1}{2}, -y + \frac{3}{2}, z$; (v) $-y + \frac{1}{2}, y + \frac{3}{2}, z - \frac{1}{2}$; (vi) $-x + 1, -y + 4, z - \frac{1}{2}$; (vii) $x, y + 3, z$.]

two C—H···Cl interactions [C4—H4···Cl1ⁱⁱ; symmetry code: (ii) $x - \frac{1}{2}, -y + \frac{5}{2}, z$] links the molecules into infinite zigzag ribbons extending along the *a* direction (Fig. 4*a*), with an angle between neighboring molecules of 55.816 (14)°. These ribbons are similar to those described for the monoclinic structure. Most notably, the angle between molecules is almost identical (only five s.u.s apart) and both ribbons are generated by glide planes (a *c*-glide in the monoclinic structure, corresponding to the propagation of the layer in the *c* direction, and an *a*-glide in the orthorhombic structure, where the chain extends along *a*). It should be noted that the *a* axis in the monoclinic structure is only slightly longer than the *c* axis in the orthorhombic structure [and *d*₁₀₀ of the monoclinic form (18.136 Å) is almost identical to the length of the orthorhombic *c* axis], and the monoclinic *c* and orthorhombic *a* axes are also quite similar (as are, incidentally, the two *b* axes, making the β angle the only notable difference between the two unit cells).

The second interaction in the orthorhombic structure, C7—H7···Cl1ⁱⁱⁱ [symmetry code: (iii) $-x + 1, -y + 1, z - \frac{1}{2}$], adds an interesting dimension of complexity to the packing of that polymorph. When examined on its own, it connects the molecules into infinite zigzag chains extending along the *c* direction, perpendicular to the ribbons generated by the first interaction (Fig. 4*b*). In those chains, the angle between neighboring molecules is 51.494 (13)°. In combination, the two independent C—H···Cl hydrogen bonds give rise to an infinite three-dimensional network (Fig. 4*c* shows a packing plot in projection along the *b* axis). Connection in the third dimension is generated by means of helices extending along the *b* direction (Fig. 4*d*). These helices are built using all four symmetry operators of the space group (including *x, y, z*) and

the four symmetry-equivalent molecules contribute to a given helix in four different ways. One molecule binds to its neighbors through atoms H4 and H7, a second through atoms H4 and Cl1, a third through atoms H7 and Cl1, and the fourth only through atom Cl1, which accepts hydrogen bonds from atoms H4 and H7 of neighboring molecules. Fig. 4(*e*) shows the anatomy of the helix schematically. It is remarkable that, even in the comparatively simple space group *Pna2*₁, just two crystallographically independent hydrogen bonds can give rise to two infinite straight chains in two directions and a complex helix in the third, while interconnecting the molecules into a three-dimensional framework.

Fig. 5 shows packing plots of the two structures and gives the nearest Cl···H distances in views approximately along the respective *b* axes. In both structures, each of the Cl atoms is near five H atoms and all H atoms, except for atom H9 in both molecules, are involved. The respective Cl···H distances are quite similar in the two polymorphs. This statement does not refer to hydrogen bonds or van der Waals interactions, as most of these Cl···H distances are longer than the sum of the van der Waals radii. Nevertheless, the projections shown in Fig. 5 illustrate well the similarities between the two structures. A comparison of the Cl···H distances in the two structures also reflects the higher density found for the monoclinic structure, as the corresponding distances in the orthorhombic polymorph are significantly longer.

Simulated powder patterns of the two structures calculated using *Mercury* (Macrae *et al.*, 2008) did not reveal any similarities between the two polymorphs besides those to be expected with such similar unit cells and packings as described in the paragraphs above.

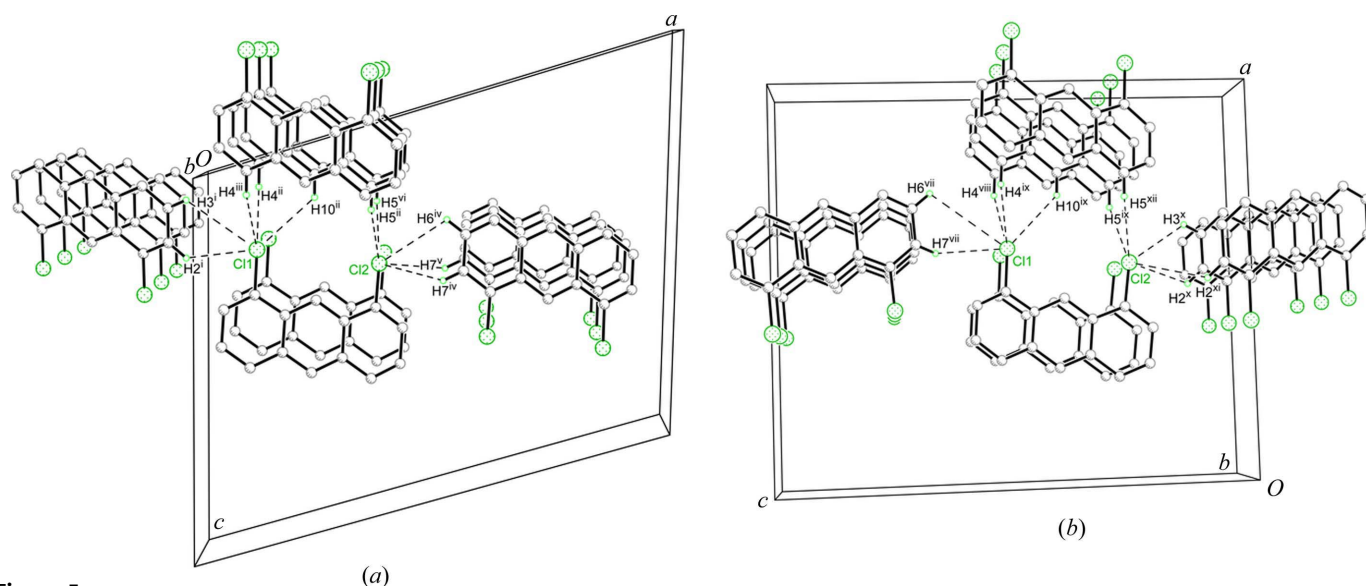


Figure 5

Projections, approximately along the *b* axes, of (a) the monoclinic polymorph and (b) the orthorhombic polymorph, showing the nearest Cl···H distances in the packing of the two structures. Distances around the Cl atoms (in Å), in (a) Cl1···H2ⁱ = 2.98, Cl1···H3ⁱ = 3.33, Cl1···H4ⁱⁱ = 2.99, Cl1···H4ⁱⁱⁱ = 2.95, Cl1···H10ⁱⁱ = 3.05, Cl2···H5ⁱⁱ = 3.07, Cl2···H6^{iv} = 3.46, Cl2···H7^v = 3.28, Cl2···H7^{iv} = 3.16 and Cl2···H5^{vi} = 2.95; in (b) Cl1···H7^{viii} = 2.93, Cl1···H6^{vii} = 3.88, Cl1···H4^{viii} = 2.84, Cl1···H4^{ix} = 3.24, Cl1···H10^{ix} = 3.01, Cl2···H5^{ix} = 3.04, Cl2···H3^x = 3.15, Cl2···H2^x = 3.23, Cl2···H2^{xi} = 3.46 and Cl2···H5^{xii} = 3.10. [Symmetry codes: (i) $-x, y - \frac{1}{2}, -z + \frac{1}{2}$; (ii) $x, -y + \frac{3}{2}, z - \frac{1}{2}$; (iii) $x, -y + \frac{5}{2}, z - \frac{1}{2}$; (iv) $-x + 1, -y + 1, -z + 1$; (v) $-x + 1, -y, -z + 1$; (vi) $x, -y + \frac{1}{2}, z - \frac{1}{2}$; (vii) $-x + 1, -y + 1, z + \frac{1}{2}$; (viii) $x + \frac{1}{2}, -y + \frac{3}{2}, z$; (ix) $x + \frac{1}{2}, -y + \frac{5}{2}, z$; (x) $-x + 1, -y + 2, z - \frac{1}{2}$; (xi) $-x + 1, -y + 1, z - \frac{1}{2}$; (xii) $x + \frac{1}{2}, -y + \frac{1}{2}, z$.]

It is interesting to note that the crystals of both polymorphs were derived by the same method and from the same solvent (slow evaporation from propan-2-ol). The presence of other components, namely Pd(PPh₃)₄ and possible reaction products, during the crystallization of the monoclinic form is the only tangible difference between the two crystallization procedures. However, recrystallization of the 16-year-old orthorhombic sample by slow evaporation from propan-2-ol yielded the monoclinic polymorph (data not presented here). Exhaustive additional crystallization experiments will need to be carried out before a conclusive statement can be made. Nevertheless, it cannot be ruled out that this may be another example of a disappearing polymorph (Dunitz & Bernstein, 1995).

Experimental

For the orthorhombic crystals, compound (I) was prepared according to the procedure of Collman *et al.* (1992) and crystallized from propan-2-ol by slow evaporation. The crystals used for this study were, without recrystallization, taken from the original 16-year-old bulk sample from which a crystal had been used to derive the orthorhombic structure of (I) by Benites *et al.* (1996). For the monoclinic crystals, (I) was prepared according to the procedure of Tauchert *et al.* (2010), chromatographically purified and crystallized by slow evaporation from propan-2-ol in the presence of Pd(PPh₃)₄ and possible reaction products.

Compound (I), monoclinic polymorph

Crystal data

C ₁₄ H ₈ Cl ₂	$V = 1060.25 (14) \text{ \AA}^3$
$M_r = 247.10$	$Z = 4$
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation
$a = 19.0070 (14) \text{ \AA}$	$\mu = 0.57 \text{ mm}^{-1}$
$b = 3.8621 (3) \text{ \AA}$	$T = 100 \text{ K}$
$c = 15.1370 (11) \text{ \AA}$	$0.20 \times 0.05 \times 0.04 \text{ mm}$
$\beta = 107.4103 (15)^\circ$	

Data collection

Bruker APEXII CCD area-detector diffractometer	56651 measured reflections
Absorption correction: multi-scan (SADABS; Sheldrick, 2009)	3700 independent reflections
$T_{\min} = 0.894$, $T_{\max} = 0.977$	3433 reflections with $I > 2\sigma(I)$
	$R_{\text{int}} = 0.027$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.028$	145 parameters
$wR(F^2) = 0.083$	H-atom parameters constrained
$S = 1.15$	$\Delta\rho_{\text{max}} = 0.53 \text{ e \AA}^{-3}$
3700 reflections	$\Delta\rho_{\text{min}} = -0.20 \text{ e \AA}^{-3}$

Compound (I), orthorhombic polymorph

Crystal data

C ₁₄ H ₈ Cl ₂	$V = 1070.9 (2) \text{ \AA}^3$
$M_r = 247.10$	$Z = 4$
Orthorhombic, $Pna2_1$	Mo $K\alpha$ radiation
$a = 15.331 (2) \text{ \AA}$	$\mu = 0.57 \text{ mm}^{-1}$
$b = 3.8621 (5) \text{ \AA}$	$T = 90 \text{ K}$
$c = 18.087 (2) \text{ \AA}$	$0.33 \times 0.20 \times 0.08 \text{ mm}$

Table 1
Hydrogen-bond geometry (\AA , $^\circ$) for the monoclinic polymorph of (I).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
$C4-H4 \cdots Cl1^i$	0.95	2.95	3.8233 (11)	154

Symmetry code: (i) $x, -y + \frac{5}{2}, z + \frac{1}{2}$

Table 2
Hydrogen-bond geometry (\AA , $^\circ$) for the orthorhombic polymorph of (I).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
$C4-H4 \cdots Cl1^{ii}$	0.95	2.84	3.7207 (11)	154
$C7-H7 \cdots Cl1^{iii}$	0.95	2.93	3.7870 (12)	151

Symmetry codes: (ii) $x - \frac{1}{2}, -y + \frac{5}{2}, z$; (iii) $-x + 1, -y + 1, z - \frac{1}{2}$

Data collection

Nonius KappaCCD area-detector diffractometer	24420 measured reflections
Absorption correction: multi-scan (SCALEPACK; Otwinowski & Minor 1997)	4919 independent reflections
$T_{\min} = 0.835$, $T_{\max} = 0.956$	4717 reflections with $I > 2\sigma(I)$
	$R_{\text{int}} = 0.027$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.028$	H-atom parameters constrained
$wR(F^2) = 0.072$	$\Delta\rho_{\text{max}} = 0.37 \text{ e \AA}^{-3}$
$S = 1.06$	$\Delta\rho_{\text{min}} = -0.27 \text{ e \AA}^{-3}$
4919 reflections	Absolute structure: Flack (1983),
146 parameters	with 2320 Friedel pairs
1 restraint	Flack parameter: 0.42 (3)

Both structures were refined against F^2 on all data by full-matrix least squares with *SHELXL97* (Sheldrick, 2008), following established refinement strategies (Müller, 2009). All H atoms were included in the model at geometrically calculated positions, with $C-H = 0.95 \text{ \AA}$, and refined using a riding model, with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$.

Four outlier reflections with $F_o - F_c/\sigma > 10$ were omitted from the refinement of the monoclinic structure, and two low-resolution reflections with $F_o \ll F_c$ (indicative of obstruction by the beam stop) were omitted from the refinement of the orthorhombic structure. The Flack x parameter (Flack, 1983) of the orthorhombic structure was determined to be close to 0.5 and this structure was refined as a racemic twin. The twin ratio was refined freely and converged at 0.42 (3).

Data collection: *APEX2* (Bruker, 2011) for the monoclinic polymorph; *COLLECT* (Nonius, 2000) for the orthorhombic polymorph. Cell refinement: *SAINT* (Bruker, 2011) for the monoclinic polymorph; *SCALEPACK* (Otwinowski & Minor, 1997) for the orthorhombic polymorph. Data reduction: *SAINT* for the monoclinic polymorph; *DENZO* (Otwinowski & Minor, 1997) and *SCALEPACK* for the orthorhombic polymorph. For both polymorphs, 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*.

The authors thank Andrew W. Maverick for providing (*i.e.* finding) the original 16-year-old orthorhombic bulk sample. PM is grateful to Bruce Foxman (Brandeis University) and Scott Speakman (MIT) for valuable discussions. The

diffractometer used to collect the data for the monoclinic form of (I) was purchased with the help of funding from the National Science Foundation (NSF) under grant No. CHE-0946721. The diffractometer used to collect the data for the orthorhombic form was purchased through grant No. LEQSF(1999–2000)-ENH-TR-13, administered by the Louisiana Board of Regents.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: GZ3224). Services for accessing these data are described at the back of the journal.

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supplementary materials

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Two polymorphs of 1,8-dichloroanthracene

Peter Müller, Frank R. Fronczek, Stacey J. Smith, Teresa Mako and Mindy Levine

(I_mono) 1,8-dichloroanthracene

Crystal data

$C_{14}H_8Cl_2$	$F(000) = 504$
$M_r = 247.10$	$D_x = 1.548 \text{ Mg m}^{-3}$
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$
Hall symbol: $-P 2ybc$	Cell parameters from 9433 reflections
$a = 19.0070 (14) \text{ \AA}$	$\theta = 2.7\text{--}32.0^\circ$
$b = 3.8621 (3) \text{ \AA}$	$\mu = 0.57 \text{ mm}^{-1}$
$c = 15.1370 (11) \text{ \AA}$	$T = 100 \text{ K}$
$\beta = 107.4103 (15)^\circ$	Needle, colourless
$V = 1060.25 (14) \text{ \AA}^3$	$0.20 \times 0.05 \times 0.04 \text{ mm}$
$Z = 4$	

Data collection

Bruker SMART APEX2 CCD area-detector diffractometer	56651 measured reflections
Radiation source: $I\mu S$ micro-focus sealed tube	3700 independent reflections
Incoatec $I\mu S$ multilayer optics monochromator	3433 reflections with $I > 2\sigma(I)$
Detector resolution: $8.3 \text{ pixels mm}^{-1}$	$R_{\text{int}} = 0.027$
φ and ω scans	$\theta_{\text{max}} = 32.0^\circ$, $\theta_{\text{min}} = 2.3^\circ$
Absorption correction: multi-scan (<i>SADABS</i> ; Sheldrick, 2009)	$h = -28 \rightarrow 28$
$T_{\text{min}} = 0.894$, $T_{\text{max}} = 0.977$	$k = -5 \rightarrow 5$
	$l = -22 \rightarrow 22$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.028$	H-atom parameters constrained
$wR(F^2) = 0.083$	$w = 1/[\sigma^2(F_o^2) + (0.0366P)^2 + 0.5831P]$
$S = 1.15$	where $P = (F_o^2 + 2F_c^2)/3$
3700 reflections	$(\Delta/\sigma)_{\text{max}} = 0.001$
145 parameters	$\Delta\rho_{\text{max}} = 0.53 \text{ e \AA}^{-3}$
0 restraints	$\Delta\rho_{\text{min}} = -0.20 \text{ e \AA}^{-3}$
Primary atom site location: structure-invariant direct methods	

Special details

Experimental. Bruker X8 Kappa DUO four-circle diffractometer, Bruker APEX2 CCD. The instrument was purchased with the help of funding from the National Science Foundation (NSF) under Grant Number CHE-0946721.

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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
Cl2	0.382516 (13)	0.29325 (7)	0.371481 (17)	0.01727 (7)
Cl1	0.130329 (14)	0.83680 (7)	0.229521 (17)	0.01825 (7)
C1	0.12833 (5)	0.9344 (3)	0.34099 (7)	0.01406 (17)
C2	0.06746 (6)	1.0937 (3)	0.35174 (7)	0.01650 (18)
H2	0.0270	1.1473	0.2991	0.020*
C3	0.06480 (6)	1.1791 (3)	0.44184 (8)	0.01791 (19)
H3	0.0220	1.2864	0.4494	0.021*
C4	0.12310 (6)	1.1087 (3)	0.51775 (7)	0.01679 (18)
H4	0.1209	1.1719	0.5775	0.020*
C5	0.37151 (6)	0.6169 (3)	0.65498 (7)	0.01720 (19)
H5	0.3702	0.6826	0.7150	0.021*
C6	0.43164 (6)	0.4487 (3)	0.64500 (7)	0.01874 (19)
H6	0.4719	0.3980	0.6982	0.022*
C7	0.43488 (6)	0.3484 (3)	0.55613 (8)	0.01731 (19)
H7	0.4771	0.2309	0.5500	0.021*
C8	0.37733 (5)	0.4207 (3)	0.47958 (7)	0.01412 (17)
C9	0.25283 (5)	0.6755 (3)	0.40811 (7)	0.01312 (17)
H9	0.2546	0.6121	0.3482	0.016*
C10	0.24777 (6)	0.8657 (3)	0.58497 (7)	0.01515 (18)
H10	0.2463	0.9321	0.6448	0.018*
C11	0.19057 (5)	0.8459 (3)	0.41776 (7)	0.01298 (17)
C12	0.18742 (5)	0.9407 (3)	0.50812 (7)	0.01414 (17)
C13	0.31246 (5)	0.5974 (3)	0.48543 (7)	0.01290 (16)
C14	0.31032 (6)	0.6954 (3)	0.57603 (7)	0.01411 (17)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl2	0.01612 (11)	0.01846 (12)	0.01843 (12)	0.00085 (8)	0.00698 (8)	-0.00174 (8)
Cl1	0.01790 (12)	0.02263 (13)	0.01357 (11)	0.00230 (9)	0.00371 (8)	-0.00045 (8)
C1	0.0140 (4)	0.0139 (4)	0.0146 (4)	-0.0012 (3)	0.0049 (3)	-0.0002 (3)
C2	0.0145 (4)	0.0151 (4)	0.0201 (4)	0.0000 (3)	0.0054 (3)	-0.0005 (4)
C3	0.0161 (4)	0.0167 (5)	0.0232 (5)	0.0009 (3)	0.0093 (4)	-0.0015 (4)
C4	0.0184 (4)	0.0159 (4)	0.0190 (4)	-0.0007 (4)	0.0101 (4)	-0.0019 (4)
C5	0.0195 (4)	0.0165 (5)	0.0136 (4)	-0.0032 (4)	0.0018 (3)	0.0009 (4)

C6	0.0192 (4)	0.0163 (5)	0.0172 (4)	-0.0020 (4)	0.0001 (3)	0.0016 (4)
C7	0.0147 (4)	0.0154 (4)	0.0201 (4)	-0.0006 (3)	0.0026 (3)	0.0011 (4)
C8	0.0136 (4)	0.0132 (4)	0.0156 (4)	-0.0012 (3)	0.0044 (3)	-0.0002 (3)
C9	0.0126 (4)	0.0138 (4)	0.0132 (4)	-0.0016 (3)	0.0042 (3)	-0.0003 (3)
C10	0.0174 (4)	0.0155 (4)	0.0134 (4)	-0.0031 (3)	0.0059 (3)	-0.0009 (3)
C11	0.0134 (4)	0.0126 (4)	0.0136 (4)	-0.0020 (3)	0.0051 (3)	-0.0002 (3)
C12	0.0162 (4)	0.0130 (4)	0.0148 (4)	-0.0025 (3)	0.0071 (3)	-0.0013 (3)
C13	0.0130 (4)	0.0124 (4)	0.0135 (4)	-0.0023 (3)	0.0043 (3)	-0.0001 (3)
C14	0.0161 (4)	0.0132 (4)	0.0128 (4)	-0.0035 (3)	0.0040 (3)	0.0000 (3)

Geometric parameters (Å, °)

C12—C8	1.7395 (10)	C6—C7	1.4190 (16)
C11—C1	1.7404 (10)	C6—H6	0.9500
C1—C2	1.3624 (14)	C7—C8	1.3633 (14)
C1—C11	1.4297 (14)	C7—H7	0.9500
C2—C3	1.4187 (15)	C8—C13	1.4350 (14)
C2—H2	0.9500	C9—C13	1.3970 (13)
C3—C4	1.3638 (16)	C9—C11	1.3995 (14)
C3—H3	0.9500	C9—H9	0.9500
C4—C12	1.4295 (14)	C10—C12	1.3980 (14)
C4—H4	0.9500	C10—C14	1.4003 (15)
C5—C6	1.3616 (16)	C10—H10	0.9500
C5—C14	1.4287 (14)	C11—C12	1.4343 (14)
C5—H5	0.9500	C13—C14	1.4348 (14)
C2—C1—C11	122.45 (9)	C7—C8—C13	122.05 (9)
C2—C1—C11	118.64 (8)	C7—C8—C12	118.99 (8)
C11—C1—C11	118.91 (8)	C13—C8—C12	118.96 (7)
C1—C2—C3	119.71 (10)	C13—C9—C11	120.90 (9)
C1—C2—H2	120.1	C13—C9—H9	119.5
C3—C2—H2	120.1	C11—C9—H9	119.5
C4—C3—C2	120.66 (10)	C12—C10—C14	121.73 (9)
C4—C3—H3	119.7	C12—C10—H10	119.1
C2—C3—H3	119.7	C14—C10—H10	119.1
C3—C4—C12	120.57 (10)	C9—C11—C1	123.20 (9)
C3—C4—H4	119.7	C9—C11—C12	119.83 (9)
C12—C4—H4	119.7	C1—C11—C12	116.98 (9)
C6—C5—C14	120.57 (10)	C10—C12—C4	121.53 (9)
C6—C5—H5	119.7	C10—C12—C11	118.86 (9)
C14—C5—H5	119.7	C4—C12—C11	119.60 (9)
C5—C6—C7	120.76 (10)	C9—C13—C14	119.73 (9)
C5—C6—H6	119.6	C9—C13—C8	123.21 (9)
C7—C6—H6	119.6	C14—C13—C8	117.06 (9)
C8—C7—C6	119.89 (10)	C10—C14—C5	121.40 (9)
C8—C7—H7	120.1	C10—C14—C13	118.94 (9)
C6—C7—H7	120.1	C5—C14—C13	119.66 (10)

Hydrogen-bond geometry (Å, °)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$C4-H4\cdots Cl1^i$	0.95	2.95	3.8233 (11)	154

Symmetry code: (i) $x, -y+5/2, z+1/2$.

(I_ortho) 1,8-dichloroanthracene

Crystal data

$C_{14}H_8Cl_2$

$M_r = 247.10$

Orthorhombic, $Pna2_1$

Hall symbol: $P\ 2c\ -2n$

$a = 15.331\ (2)\ \text{Å}$

$b = 3.8621\ (5)\ \text{Å}$

$c = 18.087\ (2)\ \text{Å}$

$V = 1070.9\ (2)\ \text{Å}^3$

$Z = 4$

$F(000) = 504$

$D_x = 1.533\ \text{Mg m}^{-3}$

Mo $K\alpha$ radiation, $\lambda = 0.71073\ \text{Å}$

Cell parameters from 4800 reflections

$\theta = 2.5\text{--}36.5^\circ$

$\mu = 0.57\ \text{mm}^{-1}$

$T = 90\ \text{K}$

Lath, yellow

$0.33 \times 0.20 \times 0.08\ \text{mm}$

Data collection

Nonius KappaCCD area-detector
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

φ and ω scans

Absorption correction: multi-scan

(SCALEPACK; Otwinowski & Minor 1997)

$T_{\min} = 0.835, T_{\max} = 0.956$

24420 measured reflections

4919 independent reflections

4717 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.027$

$\theta_{\max} = 36.5^\circ, \theta_{\min} = 2.9^\circ$

$h = -25 \rightarrow 24$

$k = -4 \rightarrow 6$

$l = -30 \rightarrow 29$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.028$

$wR(F^2) = 0.072$

$S = 1.06$

4919 reflections

146 parameters

1 restraint

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

$w = 1/[\sigma^2(F_o^2) + (0.0379P)^2 + 0.2409P]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = 0.001$

$\Delta\rho_{\max} = 0.37\ \text{e Å}^{-3}$

$\Delta\rho_{\min} = -0.27\ \text{e Å}^{-3}$

Absolute structure: Flack (1983), with 2320

Friedel pairs

Flack parameter: 0.42 (3)

Special details

Experimental. Nonius KappaCCD. The diffractometer was purchased through grant No. LEQSF(1999–2000)-ENH-TR-13, administered by the Louisiana Board of Regents.

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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
C11	0.604407 (15)	0.89356 (7)	0.499979 (15)	0.01757 (5)
C12	0.551245 (16)	0.33625 (6)	0.248928 (15)	0.01766 (5)
C1	0.49318 (6)	0.9843 (3)	0.50488 (6)	0.01308 (15)
C2	0.46168 (7)	1.1505 (3)	0.56601 (6)	0.01533 (16)
H2	0.5000	1.2154	0.6049	0.018*
C3	0.37096 (8)	1.2256 (3)	0.57101 (6)	0.01685 (17)
H3	0.3488	1.3388	0.6137	0.020*
C4	0.31555 (7)	1.1368 (3)	0.51515 (6)	0.01620 (17)
H4	0.2552	1.1881	0.5194	0.019*
C5	0.26485 (7)	0.6215 (3)	0.26894 (6)	0.01811 (18)
H5	0.2046	0.6772	0.2722	0.022*
C6	0.29585 (8)	0.4562 (3)	0.20762 (6)	0.01972 (19)
H6	0.2570	0.3983	0.1686	0.024*
C7	0.38568 (8)	0.3698 (3)	0.20149 (6)	0.01817 (18)
H7	0.4067	0.2555	0.1585	0.022*
C8	0.44159 (7)	0.4517 (3)	0.25761 (6)	0.01466 (16)
C9	0.46959 (6)	0.7135 (3)	0.38141 (5)	0.01289 (15)
H9	0.5298	0.6574	0.3779	0.015*
C10	0.29172 (7)	0.8810 (3)	0.39160 (6)	0.01500 (16)
H10	0.2316	0.9390	0.3949	0.018*
C11	0.43834 (6)	0.8835 (2)	0.44444 (5)	0.01204 (14)
C12	0.34733 (6)	0.9675 (3)	0.45031 (5)	0.01332 (15)
C13	0.41335 (7)	0.6252 (2)	0.32344 (5)	0.01301 (15)
C14	0.32225 (7)	0.7117 (3)	0.32837 (5)	0.01399 (15)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C11	0.01212 (9)	0.02056 (10)	0.02002 (9)	0.00001 (7)	-0.00155 (8)	-0.00242 (9)
C12	0.01851 (10)	0.01666 (9)	0.01781 (9)	0.00126 (8)	0.00319 (8)	-0.00108 (9)
C1	0.0122 (3)	0.0125 (3)	0.0145 (4)	-0.0002 (3)	-0.0002 (3)	0.0011 (3)
C2	0.0178 (4)	0.0132 (4)	0.0149 (4)	-0.0002 (3)	-0.0002 (3)	0.0000 (3)
C3	0.0204 (4)	0.0141 (4)	0.0160 (4)	0.0017 (3)	0.0041 (3)	0.0001 (3)
C4	0.0151 (4)	0.0146 (4)	0.0189 (4)	0.0019 (3)	0.0042 (3)	0.0020 (3)
C5	0.0173 (4)	0.0160 (4)	0.0210 (4)	-0.0015 (3)	-0.0057 (3)	0.0034 (3)
C6	0.0242 (5)	0.0156 (4)	0.0194 (4)	-0.0016 (4)	-0.0077 (4)	0.0012 (3)
C7	0.0249 (5)	0.0149 (4)	0.0148 (4)	-0.0013 (3)	-0.0030 (3)	0.0008 (3)
C8	0.0173 (4)	0.0122 (3)	0.0146 (4)	-0.0003 (3)	-0.0004 (3)	0.0010 (3)
C9	0.0127 (4)	0.0124 (4)	0.0136 (3)	-0.0003 (3)	0.0005 (3)	0.0015 (3)
C10	0.0122 (4)	0.0145 (4)	0.0183 (4)	0.0001 (3)	-0.0004 (3)	0.0027 (3)
C11	0.0122 (3)	0.0108 (3)	0.0131 (3)	-0.0004 (3)	0.0010 (3)	0.0015 (3)
C12	0.0125 (3)	0.0122 (4)	0.0153 (3)	0.0003 (3)	0.0015 (3)	0.0029 (3)
C13	0.0142 (4)	0.0112 (4)	0.0136 (3)	-0.0005 (3)	-0.0007 (3)	0.0022 (3)
C14	0.0136 (4)	0.0120 (4)	0.0164 (4)	-0.0013 (3)	-0.0019 (3)	0.0030 (3)

Geometric parameters (Å, °)

C11—C1	1.7431 (10)	C6—C7	1.4214 (18)
C12—C8	1.7463 (11)	C6—H6	0.9500
C1—C2	1.3666 (15)	C7—C8	1.3657 (15)
C1—C11	1.4330 (15)	C7—H7	0.9500
C2—C3	1.4237 (17)	C8—C13	1.4334 (14)
C2—H2	0.9500	C9—C13	1.3996 (14)
C3—C4	1.3637 (16)	C9—C11	1.4001 (14)
C3—H3	0.9500	C9—H9	0.9500
C4—C12	1.4284 (15)	C10—C14	1.3981 (15)
C4—H4	0.9500	C10—C12	1.4022 (15)
C5—C6	1.3650 (17)	C10—H10	0.9500
C5—C14	1.4322 (15)	C11—C12	1.4364 (14)
C5—H5	0.9500	C13—C14	1.4388 (15)
C2—C1—C11	122.51 (9)	C7—C8—C13	122.42 (10)
C2—C1—C11	118.77 (8)	C7—C8—C12	118.57 (8)
C11—C1—C11	118.72 (8)	C13—C8—C12	119.00 (8)
C1—C2—C3	119.49 (10)	C13—C9—C11	120.89 (9)
C1—C2—H2	120.3	C13—C9—H9	119.6
C3—C2—H2	120.3	C11—C9—H9	119.6
C4—C3—C2	120.68 (10)	C14—C10—C12	121.83 (9)
C4—C3—H3	119.7	C14—C10—H10	119.1
C2—C3—H3	119.7	C12—C10—H10	119.1
C3—C4—C12	120.72 (10)	C9—C11—C1	123.21 (9)
C3—C4—H4	119.6	C9—C11—C12	119.90 (9)
C12—C4—H4	119.6	C1—C11—C12	116.89 (9)
C6—C5—C14	120.64 (11)	C10—C12—C4	121.57 (9)
C6—C5—H5	119.7	C10—C12—C11	118.74 (9)
C14—C5—H5	119.7	C4—C12—C11	119.69 (9)
C5—C6—C7	120.70 (10)	C9—C13—C8	123.37 (9)
C5—C6—H6	119.6	C9—C13—C14	119.68 (9)
C7—C6—H6	119.6	C8—C13—C14	116.95 (9)
C8—C7—C6	119.72 (10)	C10—C14—C5	121.47 (10)
C8—C7—H7	120.1	C10—C14—C13	118.96 (9)
C6—C7—H7	120.1	C5—C14—C13	119.57 (10)

Hydrogen-bond geometry (Å, °)

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
C4—H4 \cdots C11 ⁱ	0.95	2.84	3.7207 (11)	154
C7—H7 \cdots C11 ⁱⁱ	0.95	2.93	3.7870 (12)	151

Symmetry codes: (i) $x-1/2, -y+5/2, z$; (ii) $-x+1, -y+1, z-1/2$.