



# Three tetracyclic dibenzoazepine derivatives exhibiting different molecular conformations, different patterns of intermolecular hydrogen bonding and different modes of supramolecular aggregation

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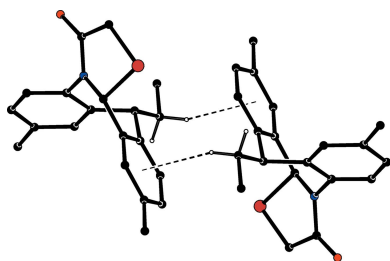
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The biological potential of compounds of the tricyclic dibenzo[*b,e*]azepine system has resulted in considerable synthetic efforts to develop efficient methods for the synthesis of new derivatives of this kind. (9*RS*,15*RS*)-9-Ethyl-11-methyl-9,13*b*-dihydrodibenzo[*c,f*]thiazolo[3,2-*a*]azepin-3(2*H*)-one, C<sub>19</sub>H<sub>19</sub>NOS, (I), crystallizes as a kryptoracemate with  $Z' = 2$  in the space group  $P2_1$ , with one molecule each of the (9*R*,15*R*) and (9*S*,15*S*) configurations in the asymmetric unit, while (9*RS*,15*RS*)-9-ethyl-7,12-dimethyl-9,13*b*-dihydrodibenzo[*c,f*]thiazolo[3,2-*a*]azepin-3(2*H*)-one, C<sub>20</sub>H<sub>21</sub>NOS, (II), crystallizes with  $Z' = 1$  in the space group  $C2/c$ . Ethyl (13*RS*)-2-chloro-13-ethyl-4-oxo-8,13-dihydro-4*H*-benzo[5,6]azepino[3,2,1-*ij*]quinoline-5-carboxylate, C<sub>22</sub>H<sub>20</sub>ClNO<sub>3</sub>, (III), exhibits enantiomeric disorder in the space group  $P\bar{1}$  such that the reference site is occupied by the 13*R* and 13*S* enantiomers, with occupancies of 0.900 (6) and 0.100 (6). In each of the two independent molecules in (I), the five-membered ring adopts an envelope conformation, but the corresponding ring in (II) adopts a half-chair conformation, while the six-membered ring in the major form of (III) adopts a twist-boat conformation. The conformation of the seven-membered ring in each of (I), (II) and the major form of (III) approximates to the twist-boat form. The molecules of compound (I) are linked by two C—H...O hydrogen bonds to form two independent antiparallel  $C(5)$  chains, with each type containing only one enantiomer. These chains are linked into sheets by two C—H... $\pi$ (arene) hydrogen bonds, in which the two donors are both provided by the (9*R*,15*R*) enantiomer and the two acceptor arene rings form part of a molecule of (9*S*,15*S*) configuration, precluding any additional crystallographic symmetry. The molecules of compound (II) are linked by inversion-related C—H... $\pi$ (arene) hydrogen bonds to form isolated cyclic centrosymmetric dimers. The molecules of compound (III) are linked into cyclic centrosymmetric dimers by C—H...O hydrogen bonds and these dimers are linked into chains by a  $\pi$ – $\pi$  stacking interaction. Comparisons are made with some related structures.

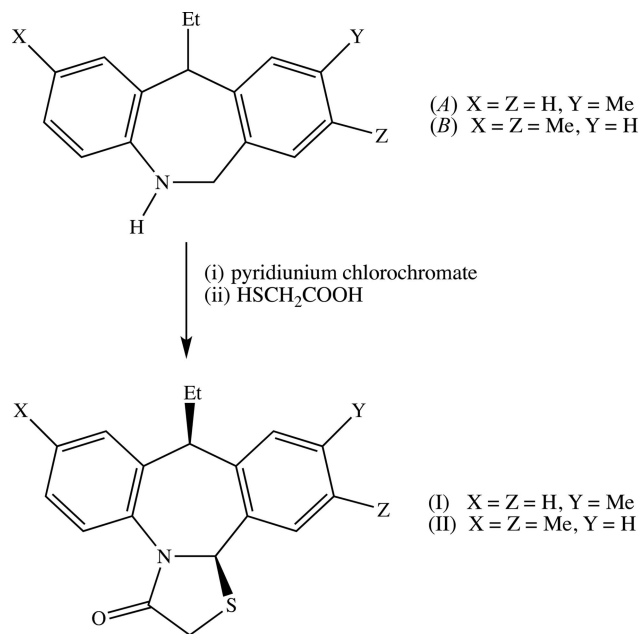


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## 1. Introduction

The tricyclic dibenzo[*b,e*]azepine system constitutes a class of nitrogen-containing heterocyclic compounds whose chemistry continues to be of interest, because of the action of compounds containing this system as analgesics and as anticancer, antidepressive, antihistaminic, antimuscarinic and antipsychotic agents (Al-Qawasmeh *et al.*, 2009). Examples of such compounds in current clinical use include mianserin, racemic 2-methyl-1,2,3,4,10,14*b*-hexahydrodibenzo[*c,f*]pyrazino[1,2-*a*]-

azepine, which is a potent antidepressant (Dinesh *et al.*, 2014), and epinastine, racemic 3-amino-9,13b-dihydro-1*H*-dibenzo[*c,f*]imidazo[1,5-*a*]azepine, which is an antihistaminic used in the treatment of allergic conjunctivitis (Liu *et al.*, 2004). The biological potential of these compounds has resulted in considerable synthetic efforts to develop efficient methods for the synthesis of new derivatives of this kind (Andrés *et al.*, 2002; Stappers *et al.*, 2002; Wikström *et al.*, 2002).

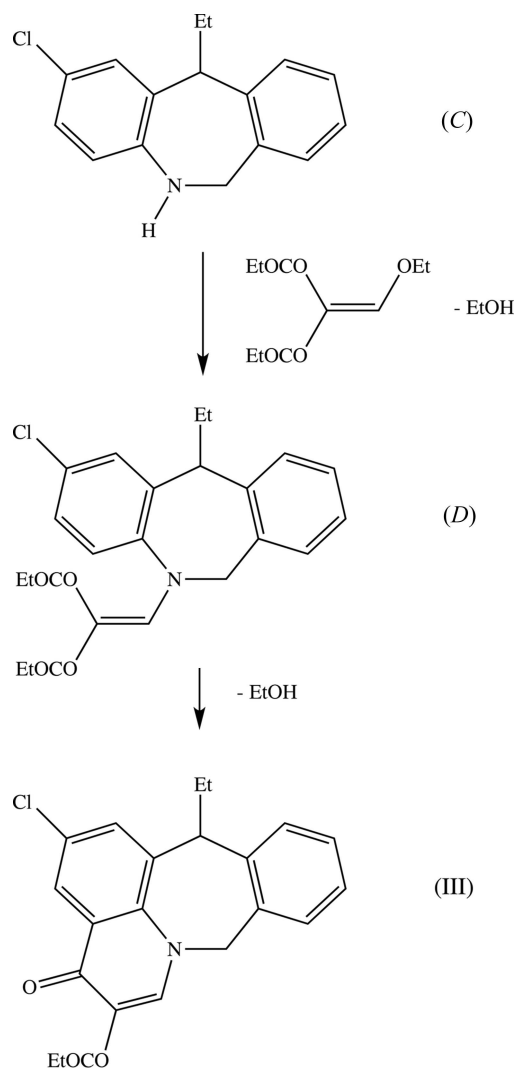


Scheme 1

In this context, and as part of our own interest in the identification of other molecular entities with pharmacological potential, we have for several years studied the chemistry of the synthetically available dihydrodibenzo[*b,e*]azepines (Palma *et al.*, 2004) as building blocks for the construction of novel fused tetracyclic azepine systems. Accordingly, we have reported the synthesis of tetrahydrodibenzo[*c,f*]thiazolo[3,2-*a*]azepine derivatives, compounds in which the dibenzo[*b,e*]azepine nucleus is fused to a thiazolidin-4-one ring (Palma *et al.*, 2010). We are now developing a simple and efficient synthetic methodology for the preparation of derivatives of the type alkyl 4-oxobenzo[5,6]azepino[3,2,1-*ij*]quinoline-5-carboxylate. This is a new heterocyclic system in which a benzazepine nucleus is fused to a 4-quinolone system, which is also of great interest for both the medicinal chemistry and pharmaceutical industries (Mugnaini *et al.*, 2009), mainly because of their antibacterial activity, the best studied biological property of the so-called fluoroquinolone antibiotics.

We report here the molecular and supramolecular structures of three compounds containing fused tetracyclic azepine systems, namely 9-ethyl-11-methyl-9,13b-dihydrodibenzo[*c,f*]thiazolo[3,2-*a*]azepin-3(2*H*)-one, (I), 9-ethyl-7,12-dimethyl-9,13b-dihydrodibenzo[*c,f*]thiazolo[3,2-*a*]azepin-3(2*H*)-one, (II), and ethyl 2-chloro-13-ethyl-4-oxo-8,13-dihydro-4*H*-benzo[5,6]azepino[3,2,1-*ij*]quinoline-5-carboxylate, (III) (Figs. 1–3). Compounds (I) and (II) were synthesized from the corresponding dihydrodibenzo[*b,e*]azepines (A) and (B) (see

Scheme 1) according to a previously described procedure (Palma *et al.*, 2010), in which the tricyclic precursors (A) and (B) were first subjected to oxidation using pyridinium chlorochromate, followed by cyclocondensation with thio-glycolic acid to give (I) and (II). Compound (III) was synthesized from dihydrodibenzo[*b,e*]azepine (C) employing the modified Gould–Jacobs reaction, in which an alkoxy-methylenemalonate derivative, here diethyl 2-(methoxymethylene)malonate, reacts with the amino group of the precursor with displacement of the ethoxy unit by the N atom giving the intermediate (D), followed by benzannulation to give the quinolone derivative (III) (see Scheme 2).



Scheme 2

## 2. Experimental

### 2.1. Synthesis and crystallization

Compounds (I) and (II) were prepared according to the method reported previously by Palma *et al.* (2010). For the synthesis of compound (III), a solution of 2-chloro-11-ethyl-6,11-dihydro-5*H*-dibenzo[*b,e*]azepine, (C) (0.10 mmol), and diethyl 2-(methoxymethylene)malonate (0.13 mmol) in toluene (10 ml) was heated under reflux for 15 h until the reaction was complete, as indicated by thin-layer chromatography

**Table 1**  
Experimental details.

	(I)	(II)	(III)
<b>Crystal data</b>			
Chemical formula	C <sub>19</sub> H <sub>19</sub> NOS	C <sub>20</sub> H <sub>21</sub> NOS	C <sub>22</sub> H <sub>20</sub> ClNO <sub>3</sub>
<i>M<sub>r</sub></i>	309.41	323.44	381.84
Crystal system, space group	Monoclinic, <i>P</i> 2 <sub>1</sub>	Monoclinic, <i>C</i> 2/ <i>c</i>	Triclinic, <i>P</i> $\bar{1}$
Temperature (K)	100	298	120
<i>a</i> , <i>b</i> , <i>c</i> (Å)	11.4261 (5), 8.1847 (3), 16.8243 (6)	18.1021 (12), 12.4436 (8), 14.8429 (9)	6.8533 (16), 10.612 (5), 13.561 (3)
$\alpha$ , $\beta$ , $\gamma$ (°)	90, 93.067 (2), 90	90, 97.645 (3), 90	72.45 (4), 75.840 (19), 82.46 (2)
<i>V</i> (Å <sup>3</sup> )	1571.14 (11)	3313.7 (4)	910.0 (6)
<i>Z</i>	4	8	2
Radiation type	Mo <i>K</i> α	Mo <i>K</i> α	Mo <i>K</i> α
$\mu$ (mm <sup>-1</sup> )	0.21	0.20	0.23
Crystal size (mm)	0.40 × 0.35 × 0.22	0.23 × 0.22 × 0.20	0.26 × 0.15 × 0.13
<b>Data collection</b>			
Diffractometer	Bruker Kappa APEXII	Bruker Kappa APEXII	Nonius KappaCCD
Absorption correction	Multi-scan ( <i>SADABS</i> ; Bruker, 2006)	Multi-scan ( <i>SADABS</i> ; Bruker, 2006)	Multi-scan ( <i>SADABS</i> ; Sheldrick, 2003)
<i>T</i> <sub>min</sub> – <i>T</i> <sub>max</sub>	0.823, 0.955	0.818, 0.961	0.845, 0.970
No. of measured, independent and observed [ <i>I</i> > 2σ( <i>I</i> )] reflections	61785, 11002, 10697	30952, 3402, 2307	20183, 3776, 2195
<i>R</i> <sub>int</sub>	0.025	0.048	0.154
(sin θ/ <i>λ</i> ) <sub>max</sub> (Å <sup>-1</sup> )	0.757	0.626	0.629
<b>Refinement</b>			
<i>R</i> [ <i>F</i> <sup>2</sup> > 2σ( <i>F</i> <sup>2</sup> )], <i>wR</i> [ <i>F</i> <sup>2</sup> ], <i>S</i>	0.029, 0.079, 1.05	0.060, 0.168, 1.05	0.065, 0.118, 1.06
No. of reflections	11002	3402	3776
No. of parameters	401	211	322
No. of restraints	1	0	74
H-atom treatment	H-atom parameters constrained	H-atom parameters constrained	H-atom parameters constrained
Δρ <sub>max</sub> , Δρ <sub>min</sub> (e Å <sup>-3</sup> )	0.37, -0.21	0.57, -0.45	0.29, -0.31
Absolute structure	See §2.3	–	–

Computer programs: *APEX2* (Bruker, 2006), *COLLECT* (Nonius, 1998), *SAINT* (Bruker, 2006), *DIRAX/LSQ* (Duisenberg *et al.*, 2000), *EVALCCD* (Duisenberg *et al.*, 2003), *SIR92* (Altomare *et al.*, 1994), *SIR2014* (Burla *et al.*, 2015), *SHELXL2014* (Sheldrick, 2015) and *PLATON* (Spek, 2009).

(TLC). The solvent and the excess of diethyl 2-(methoxymethylene)malonate were removed from the reaction mixture under reduced pressure, and Eaton's reagent, *i.e.* a 7.7% solution of phosphorus(V) oxide in methanesulfonic acid (1.6 ml), was added to the remaining crude material. This mixture was heated at 343 K for 40 min, again with TLC monitoring, then cooled to ambient temperature and neutralized with saturated aqueous sodium carbonate solution. The neutralized mixture was extracted with ethyl acetate (3 × 50 ml) and the combined organic extracts were dried over anhydrous sodium sulfate. The solvent was removed under reduced pressure and the crude product was purified by column chromatography on silica gel using heptane–ethyl acetate mixtures (10:1 to 1:3 *v/v*) to give compound (III) (yield 80%, m.p. 466–467 K). Colourless crystals of compounds (I)–(III) suitable for single-crystal X-ray diffraction were grown by slow evaporation, at ambient temperature and in the presence of air, of solutions in heptane–ethyl acetate (2:1 *v/v*) for (I) and (II), and in ethanol–ethyl acetate (7:3 *v/v*) for (III).

## 2.2. Spectroscopic data

*R<sub>F</sub>* = 0.27 (ethyl acetate–heptane, 1:1 *v/v*); IR (cm<sup>-1</sup>): 2965–2926 (C–H), 1687 [C=O(ester)], 1605 [C=O(ketone)], 1482 (C=C), 1146 (C–O); NMR (CDCl<sub>3</sub>): δ(<sup>1</sup>H) 1.02 (*t*, *J* = 7.2 Hz,

3H, 13-CH<sub>2</sub>–CH<sub>3</sub>), 1.41 (*t*, *J* = 7.2 Hz, 3H, O–CH<sub>2</sub>–CH<sub>3</sub>), 2.36–2.24 (*m*, 2H, 13-CH<sub>2</sub>–CH<sub>3</sub>), 4.10 (*br s*, 1H, 13-H), 4.39 (*q*, *J* = 7.2 Hz, 2H, O–CH<sub>2</sub>–), 4.88 (*br s*, 1H, 8-H<sub>B</sub>), 5.88 (*br s*, 1H, 8-H<sub>A</sub>), 7.22 (*dd*, *J* = 7.4, 1.4 Hz, 1H, 12-H), 7.31 (*td*, *J* = 7.4, 1.4 Hz, 1H, 10-H), 7.36 (*td*, *J* = 7.4, 1.4 Hz, 1H, 11-H), 7.40 (*dd*, *J* = 7.4, 1.4 Hz, 1H, 9-H), 7.51 (*d*, *J* = 2.4 Hz, 1H, 1-H), 8.34 (*d*, *J* = 2.4 Hz, 1H, 3-H), 8.53 (*s*, 1H, 6-H); δ(<sup>13</sup>C) 13.1 (13-CH<sub>2</sub>–CH<sub>3</sub>), 14.5 (O–CH<sub>2</sub>–CH<sub>3</sub>), 61.1 (O–CH<sub>2</sub>–), 61.2 (8-C), 109.6 (5-C), 126.3 (3-C), 128.1 (10-C), 129.1 (9-C, 12-C), 129.8 (11-C), 131.2 (2-C), 131.8 (8a-C), 132.3 (3a-C), 134.2 (13a-C), 137.2 (3b-C), 140.5 (12a-C), 150.1 (6-C), 165.6 (COO), 172.8 (4-C); HRMS (EI–MS, 70 eV) *m/z* found 381.1132, C<sub>22</sub>H<sub>20</sub><sup>35</sup>ClNO<sub>3</sub> requires 381.1132.

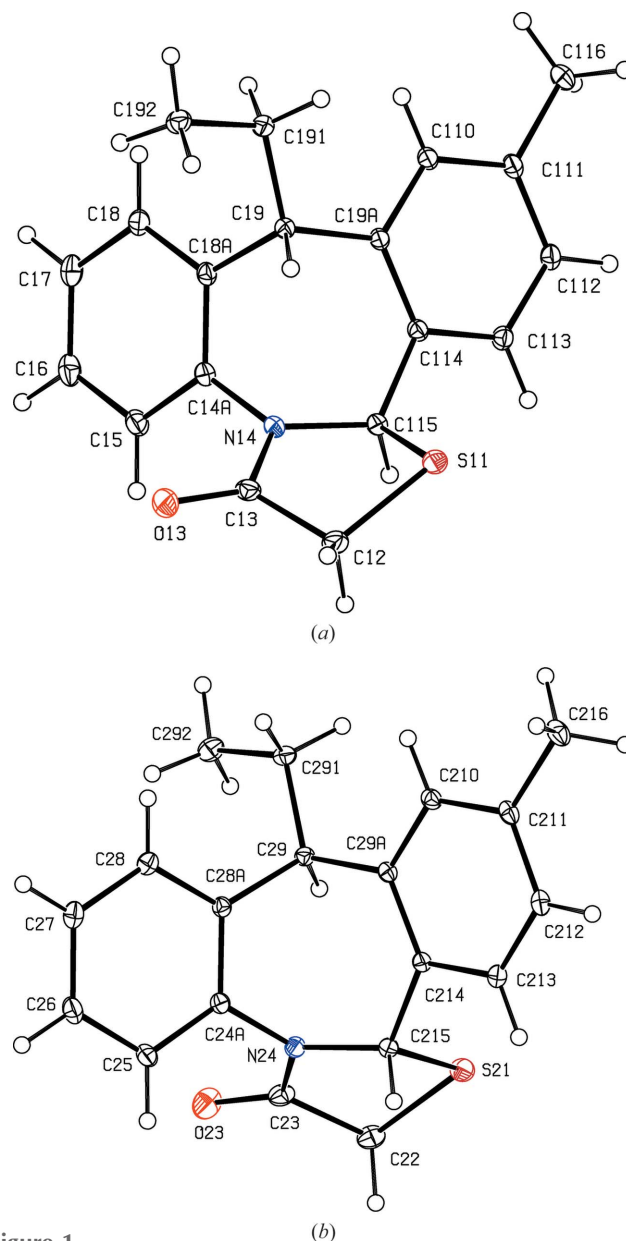
## 2.3. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 1. For compounds (I) and (II), all H atoms were located in difference maps and subsequently treated as riding atoms in geometrically idealized positions, with C–H distances of 0.95 (aromatic), 0.98 (methyl), 0.99 (methylene) or 1.00 Å (methine) for (I), and 0.93, 0.96, 0.97 or 0.98 Å for the corresponding bond types in (II), and with, in each case, *U*<sub>iso</sub>(H) = *kU*<sub>eq</sub>(C), where *k* = 1.5 for the methyl groups, which were permitted to rotate but not to tilt, and 1.2 for all other H atoms. The correct absolute configuration for

compound (I) was established using both the Flack  $x$  parameter (Flack, 1983),  $x = 0.007$  (6), calculated (Parsons *et al.*, 2013) using 4787 quotients of the type  $[(I^+) - (I^-)]/[(I^+) + (I^-)]$ , and the Hooft  $y$  parameter (Hooft *et al.*, 2010)  $y = 0.001$  (7). It was apparent from an early stage in the refinement of compound (III) that the molecules exhibited configurational disorder, such that the reference site was occupied by partial-occupancy molecules of both  $R$  and  $S$  configuration having markedly unequal occupancies. For the minor component, having the  $S$  configuration, the bonded distances and the 1,3 nonbonded distances were restrained to be the same as the corresponding distances in the major component, having an  $R$  configuration, subject to s.u. values of 0.01 and 0.02 Å, respectively; in addition, the anisotropic displacement parameters for pairs of atoms occupying similar regions of physical space were constrained to be identical. The H atoms in the major component were all located in difference maps and then treated as riding atoms in geometrically idealized positions, with C–H = 0.95 (alkenyl and aromatic), 0.98 (methyl), 0.99 (methylene) or 1.00 Å (methine), and with  $U_{\text{iso}}(\text{H})$  defined as for (I) and (II). In the final analysis of variance for compound (II), there was a negative value,  $-0.346$ , of  $K = \text{mean}(F_o^2)/\text{mean}(F_c^2)$  for the group of 409 very weak reflections having  $F_o/F_c(\text{max})$  in the range  $0.000 < F_o/F_c(\text{max}) < 0.006$ , and for compound (III) there was a large value, 5.504, of  $K$  for the group of 382 very weak reflections having  $F_o/F_c(\text{max})$  in the range  $0.000 < F_o/F_c(\text{max}) < 0.015$ .

### 3. Results and discussion

The constitutions of compounds (I) and (II) are rather similar, differing only in the number and location of the methyl substituents, which are at position 11 in (I) and at positions 7 and 12 in (II). Despite this close similarity, compound (I) crystallizes with  $Z' = 2$  in the Sohncke space group  $P2_1$ , while (II) crystallizes in the centrosymmetric space group  $C2/c$ . In molecule 1 of compound (I), containing atom S11 (Fig. 1*a*), there are stereogenic centres at atoms C19 and C115; the reference molecule 1 was selected as one having the  $R$  configuration at atom C19 and on this basis the configuration at atom C115 is also  $R$ , whereas the configurations at atoms C29 and C215 in molecule 2 (Fig. 1*b*) are both  $S$ . Thus, despite crystallizing in the space group  $P2_1$ , compound (I) is a racemic mixture of  $(9R,15R)$  and  $(9S,15S)$  enantiomers and it is therefore a kryptoracemate (Morales & Fronczek, 1996; Fábíán & Brock, 2010; Bernal & Watkins, 2015); a search for possible additional crystallographic symmetry found none. Compound (III) has a stereogenic centre at position 13 and the reference molecule was selected as one having the  $R$  configuration at this site. However, it was apparent that the reference site was in fact occupied by partial-occupancy molecules of both  $R$  and  $S$  configurations (Figs. 3*a* and 3*b*), having occupancies of 0.900 (6) and 0.100 (6), respectively. These two enantiomeric forms occupy similar but not quite identical locations (Fig. 3*c*). The centrosymmetric space groups of compounds (II) and (III) confirm that these compounds have both crystallized as racemic mixtures. That



**Figure 1**  
The molecular structures of the two independent molecules of compound (I), showing (a) molecule 1, which has the  $(9R,15R)$  configuration, and (b) molecule 2, which has the  $(9S,15S)$  configuration. Displacement ellipsoids are drawn at the 30% probability level.

compounds (I)–(III) are racemic is expected from the racemic nature of the precursors (A)–(C) (Palma *et al.*, 2010), but it is interesting to note that for compounds (I) and (II), the stereochemistry at position 15 appears to be wholly controlled by that at position 9 and no evidence was found for the formation of the diastereoisomeric  $(9RS,15SR)$  forms.

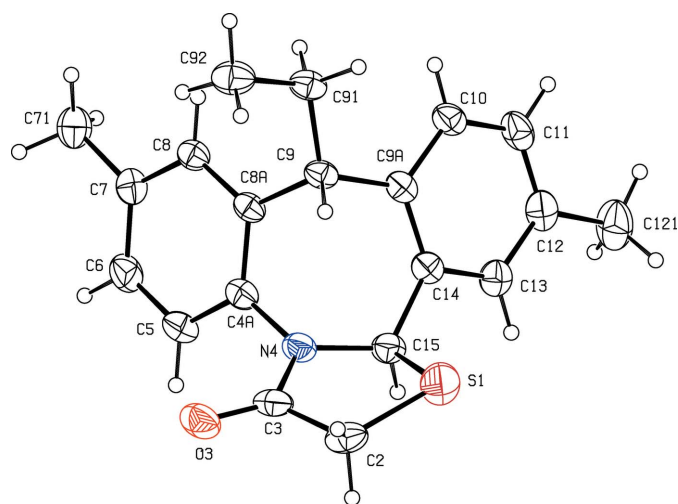
In each of the two independent molecules of compound (I), the five-membered ring is slightly puckered out of planarity, and the ring-puckering parameters (Cremer & Pople, 1975) show that in each molecule this ring adopts an envelope (Evans & Boeyens, 1989) conformation (Table 2), with the ring folded across the line C $x$ 2–C $x$ 15, where  $x = 1$  or 2 in molecules 1 and 2, respectively; the difference of *ca* 180°

**Table 2**  
Selected geometric parameters (Å, °).

	(I), molecule 1	(I), molecule 2	(II)	(III), major	(III), minor
Ring-puckering parameters					
Five-membered ring					
$Q_2$	0.4627 (12)	0.3979 (12)	0.300 (3)		
$\varphi_2$	359.12 (17)	178.1 (2)	339.0 (5)		
Six-membered ring					
$Q$				0.107 (8)	0.16 (7)
$\theta$				87 (4)	112 (26)
$\varphi$				39 (4)	2(30)
Seven-membered rings					
$Q$	0.9540 (13)	0.9418 (12)	0.981 (3)	0.843 (6)	0.85 (5)
$\varphi_2$	38.32 (8)	217.47 (8)	40.16 (16)	271.8 (4)	267 (4)
$\varphi_3$	286.0 (3)	107.8 (3)	281.6 (7)	9.6 (10)	17 (7)
Dihedral angles					
	70.84 (4)	65.44 (4)	76.67 (9)	52.8 (2)	66 (3)
Torsion angles					
Cx9A—Cx9—Cx91—Cx92	−169.01 (11)	170.60 (10)	−173.0 (2)		
Cy2A—Cy13—Cy31—Cy32				−69.4 (5)	42 (5)

Notes: (i)  $x = 1$  or  $2$  for molecules 1 and 2, respectively, in (I) and  $x = \text{nul}$  for (II);  $y = 1$  or  $2$  for the major- and minor-disorder forms, respectively, in (III); (ii) the ring-puckering angles are calculated for the following atom sequences: five-membered rings Sx1—Cx2—Cx3—Nx4—Cx1, six-membered rings Ny7—Cy3B—Cy3A—Cy4—Cy5—Cy6, and seven-membered rings Nx4—Cx4A—Cx8A—Cx9—Cx9A—Cx14—Cx15 in (I) and (II), and Ny7—Cy3B—Cy3C—Cy13—Cy2A—Cy8A—Cy8 in (III); (iii) the dihedral angles are those between the mean planes of the two aryl rings in each of (I)–(III).

between the  $\varphi_2$  values for the two molecules in (I) confirms their enantiomeric relationship. By contrast with (I), the five-membered ring in compound (II) adopts a half-chair conformation in which the ring is twisted about a line through atom C3 and the approximate mid-point of the S1—C15 bond. For idealized half-chair and envelope conformations, the values of  $\varphi_2$  are  $(36k + 18)^\circ$  and  $36k^\circ$ , respectively, where  $k$  represents an integer. Within the major disorder form of compound (III), the six-membered heterocyclic ring is slightly puckered into a twist-boat conformation; for an idealized twist-boat conformation; the ring-puckering angles are  $\theta = 90^\circ$  and  $\varphi = (60k + 30)^\circ$ , where  $k$  represents an integer. For the seven-membered ring in each of (I), (II) and the major form of (III), the ring conformations are dominated by the twist-boat sin form 2



**Figure 2**  
The molecular structure of the (9*R*,15*R*) enantiomer of compound (II). Displacement ellipsoids are drawn at the 30% probability level.

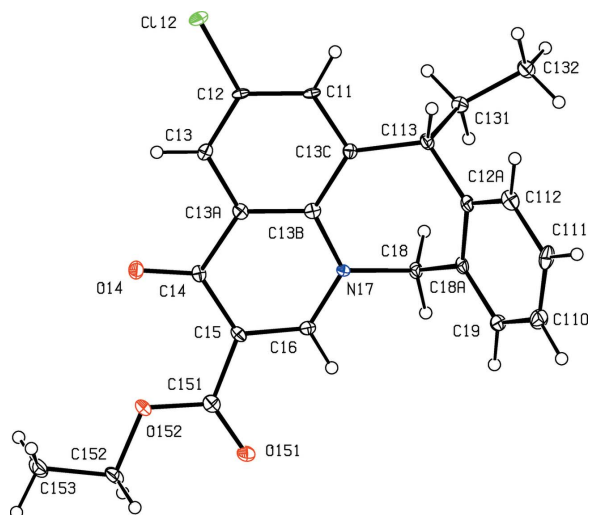
(Evans & Boeyens, 1989). By contrast, the most common conformation of the seven-membered ring in tricyclic dibenzazepines is one intermediate between the boat and twist-boat forms (Sanabría *et al.*, 2014), while the most common form in benzopyrimidoazepines is the boat form (cos form 2) (Acosta *et al.*, 2015; Acosta Quintero, Palma *et al.*, 2016).

In each of the five independent molecular entities reported here, the dihedral angle between the two aryl rings falls within a fairly narrow range of less than  $25^\circ$  (Table 2). The specification of the molecular conformations is completed by the orientation of the ethyl substituent relative to the seven-membered ring; the torsion angles defining this orientation are very similar in compounds (I) and (II), except for the difference in sign between the two independent molecules in (I) consistent with their enantiomeric relationship, whereas in (III) this orientation is entirely different (Table 2 and Figs. 1–3).

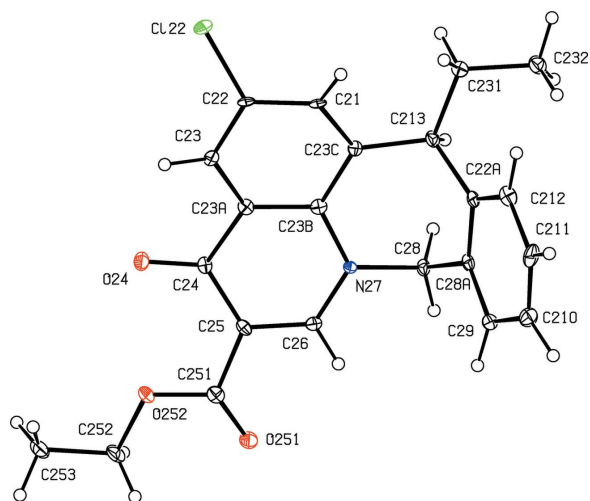
**Table 3**  
Parameters (Å, °) for hydrogen bonds and short intermolecular contacts. Cg1–Cg3 represent the centroids of the C29A/C210–C214, C24A/C25–C28/C28A and C9A/C10–C14 rings, respectively.

Compound	$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
(I)	C115—H115 $\cdots$ O13 <sup>i</sup>	1.00	2.47	3.3084 (18)	141
	C215—H215 $\cdots$ O23 <sup>ii</sup>	1.00	2.37	3.1681 (18)	136
	C112—H112 $\cdots$ Cg1 <sup>iii</sup>	0.95	2.81	3.6738 (13)	152
	C113—H113 $\cdots$ Cg2 <sup>iii</sup>	0.95	2.85	3.7676 (14)	163
(II)	C2—H2B $\cdots$ O3 <sup>iv</sup>	0.97	2.57	3.120 (4)	116
	C91—H91A $\cdots$ Cg3 <sup>v</sup>	0.97	2.82	3.736 (3)	157
(III)	C18—H18B $\cdots$ O151 <sup>vi</sup>	0.99	2.54	3.483 (9)	159
	C19—H19 $\cdots$ O151 <sup>vi</sup>	0.95	2.54	3.396 (12)	149
	C28—H28B $\cdots$ O251 <sup>vi</sup>	0.99	2.10	3.13 (7)	159
	C29—H29 $\cdots$ O251 <sup>vi</sup>	0.95	2.75	3.34 (12)	124

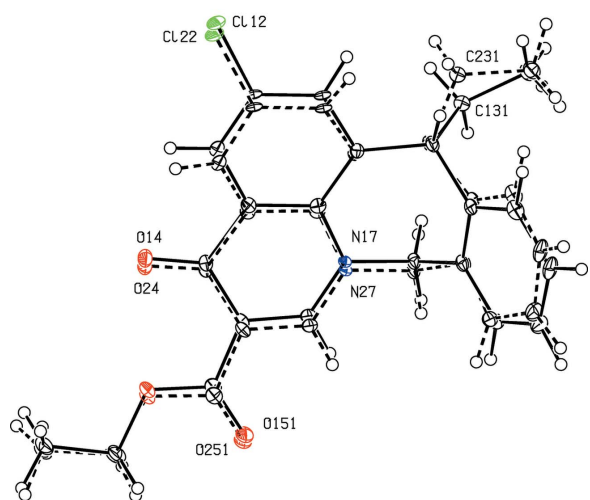
Symmetry codes: (i)  $-x, y + \frac{1}{2}, -z + 1$ ; (ii)  $-x, y - \frac{1}{2}, -z$ ; (iii)  $x, y + 1, z$ ; (iv)  $-x + \frac{1}{2}, -y + \frac{1}{2}, -z + 1$ ; (v)  $-x + 1, -y + 1, -z + 1$ ; (vi)  $-x + 1, -y + 2, -z + 1$ .



(a)



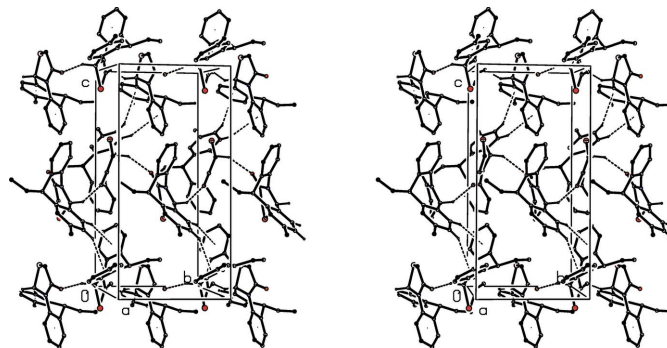
(b)



(c)

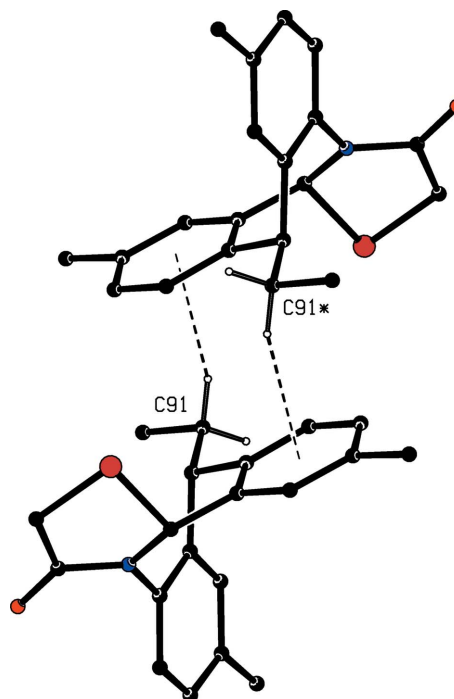
**Figure 3**

The molecular structures of the disordered components of compound (III), showing (a) the major *R* enantiomer, (b) the minor *S* enantiomer and (c) the two disorder components together, with the bonds in the major form shown as full lines and those in the minor form shown as broken lines. Displacement ellipsoids are drawn at the 30% probability level and, for the sake of clarity, the majority of the atom labels have been omitted from part (c).

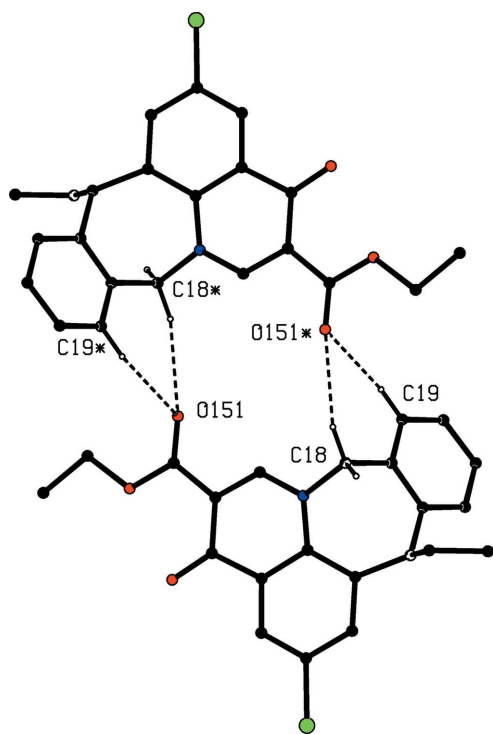

**Figure 4**

A stereoview of part of the crystal structure of compound (I), showing the formation of a hydrogen-bonded sheet parallel to (100) in which chains built from C—H...O hydrogen bonds are linked by C—H... $\pi$ (arene) hydrogen bonds. For the sake of clarity, H atoms which are not involved in the motifs shown have been omitted.

Despite the close similarity between the constitutions of compounds (I) and (II), the supramolecular assembly in these two compounds is entirely different. In compound (I), the molecules are linked into complex sheets by a combination of two C—H...O hydrogen bonds and two C—H... $\pi$ (arene) hydrogen bonds (Table 3), but the formation of the sheet structure is readily analysed in terms of simple substructures (Ferguson *et al.*, 1998*a,b*; Gregson *et al.*, 2000). The molecules of type 1 which are related by the  $2_1$  screw axis along  $(0, y, \frac{1}{2})$  are linked by C—H...O hydrogen bonds to form a  $C(5)$


**Figure 5**

Part of the crystal structure of compound (II), showing the formation of a centrosymmetric hydrogen-bonded dimer. For the sake of clarity, the unit-cell outline and H atoms bonded to C atoms that are not involved in the motif shown have been omitted. The atom marked with an asterisk (\*) is at the symmetry position  $(-x + 1, -y + 1, -z + 1)$ .


**Figure 6**

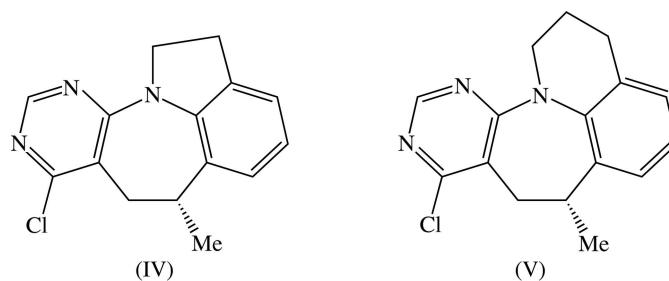
Part of the crystal structure of compound (III), showing the formation by the major-disorder form of a cyclic centrosymmetric hydrogen-bonded dimer. For the sake of clarity, the unit-cell outline and H atoms bonded to C atoms that are not involved in the motif shown have been omitted. Atoms marked with an asterisk (\*) are at the symmetry position  $(-x + 1, -y + 2, -z + 1)$ .

(Bernstein *et al.*, 1995) chain running parallel to the [010] direction. A similar  $C(5)$  chain, antiparallel to the first chain is built from type 2 molecules which are related by the  $2_1$  screw axis along  $(0, y, 0)$ ; thus each type of  $C(5)$  chain contains only a single enantiomeric form. Two independent  $C-H \cdots \pi(\text{arene})$  hydrogen bonds, in which the donors are two adjacent  $C-H$  bonds in a type 1 molecule and the acceptors are the two aryl rings of a type 2 molecule, thus precluding the possibility of any additional crystallographic symmetry, link the type 1 chains along  $(0, y, n + \frac{1}{2})$  to the type 2 chains along  $(0, y, n)$ , where  $n$  represents an integer in each case, to form a sheet lying parallel to (100) (Fig. 4); however, there are no direction-specific interactions between adjacent sheets.

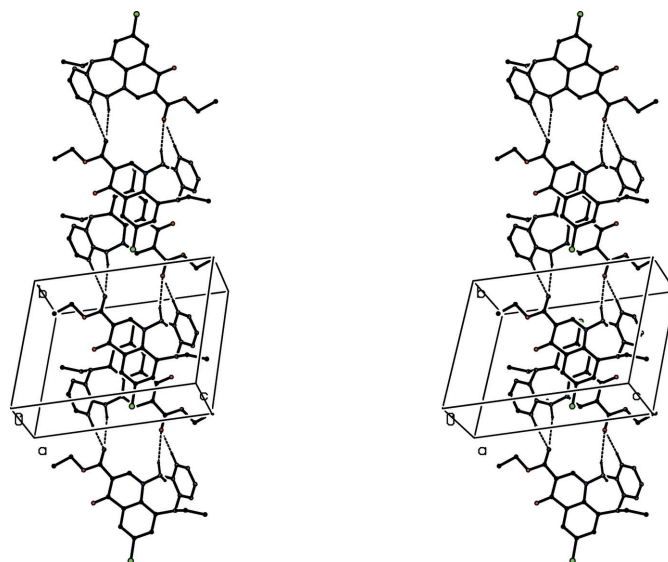
In contrast to the complex supramolecular assembly in compound (I), that in compound (II) is extremely simple. Inversion-related pairs of molecules are linked by paired  $C-H \cdots \pi(\text{arene})$  hydrogen bonds to form centrosymmetric dimers, each containing an enantiomeric pair (Fig. 5), but there are no direction-specific interactions between adjacent dimers, so that the supramolecular assembly is finite and zero-dimensional.

The molecules of compound (III) are linked by  $C-H \cdots O$  hydrogen bonds to form centrosymmetric dimers (Fig. 6). For the major-disorder form, the hydrogen bonds generate a dimer, centred at  $(\frac{1}{2}, \frac{1}{2})$ , characterized by an outer  $R_2^2(18)$  ring, which encloses an inner  $R_2^2(14)$  ring flanked by two inversion-

related  $R_2^1(6)$  rings; for the minor-disorder component, only the  $R_2^2(14)$  ring is present as the  $H29 \cdots O251^i$  separation [symmetry code: (i)  $-x + 1, -y + 2, -z + 1$ ] of 2.75 Å is above the sum of the van der Waals radii (Rowland & Taylor, 1996), so that the corresponding  $C-H \cdots O$  contact cannot be regarded as a hydrogen bond. Dimers of this type are linked into a chain by a single  $\pi-\pi$  stacking interaction. The chlorinated aryl rings of the molecules at  $(x, y, z)$  and  $(-x + 2, -y + 1, -z + 1)$  are strictly parallel, with an interplanar spacing of 3.368 (3) Å; the ring-centroid separation is 3.649 (4) Å, corresponding to a ring-centroid offset of 1.404 (4) Å. This interaction links hydrogen-bonded dimers related by translation into a  $\pi$ -stacked chain running parallel to the  $[1\bar{1}0]$  direction (Fig. 7).


**Scheme 3**

It is interesting briefly to compare compounds (I)–(III) reported here with the related tetracyclic benzopyrimidoazepine derivatives (IV) and (V) (see Scheme 3). Firstly, the syntheses of (IV) and (V) utilized a completely different approach (Acosta Quintero *et al.*, 2015; Acosta Quintero, Burgos *et al.*, 2016) from that employed for the preparation of (I)–(III); the synthesis of compounds (I)–(III) appended an additional ring to a preformed dibenzazepine skeleton, while those for (IV) and (V) were based on the formation of the


**Figure 7**

A stereoview of part of the crystal structure of compound (III), showing the formation of a  $\pi$ -stacked chain of hydrogen-bonded dimers running parallel to the  $[1\bar{1}0]$  direction. For the sake of clarity, the minor-disorder component and H atoms bonded to C atoms that are not involved in the motif shown have been omitted.

azepine ring as the final step using an *N*-pyrimidoindole precursor for (IV) and an *N*-pyrimidoquinoline precursor for (V). Secondly, the conformation of the azepine ring in compound (IV) differs from the twist-boat form which predominates in (I)–(III) and (V), as this ring contains a significant contribution from the twist-chair form. As a consequence of this, the *C*-methyl group occupies a quasi-equatorial position in (V), as expected, but a quasi-axial site in (IV) (Acosta Quintero, Palma *et al.*, 2016). Thirdly, the supramolecular aggregation in the structures of (IV) and (V) differs from that in (I)–(III). The molecules of compound (IV) are linked into *C*(5) chains by C–H···N hydrogen bonds, although interactions of this type are wholly absent from the structures of (I)–(III), and inversion-related chains of this type are linked into pairs by a  $\pi$ – $\pi$  stacking interaction involving the pyrimidine ring. The molecules of compound (V) are linked by C–H··· $\pi$ (pyrimidine) interactions into cyclic centrosymmetric dimers, somewhat similar to those in the structure of compound (II).

Finally, we note that although the racemic compound (IV) crystallizes in the Sohncke space group  $P2_1$ , it does so as a conglomerate rather than as a kryptoracemate.

## Acknowledgements

The authors thank ‘Centro de Instrumentación Científico-Técnica de Universidad de Jaén’ and Professor Pascal Roussel (University of Lille, France) for data collection. They also thank Vicerrectoría de Investigación y Extensión of Universidad Industrial de Santander (grant No. 9310), the Consejería de Innovación, Ciencia y Empresa (Junta de Andalucía, Spain) and the Universidad de Jaén for financial support.

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## supporting information

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## Three tetracyclic dibenzoazepine derivatives exhibiting different molecular conformations, different patterns of intermolecular hydrogen bonding and different modes of supramolecular aggregation

**Jeferson B. Mateus-Ruiz, Lina M. Acosta Quintero, Alirio Palma, Mario A. Macías, Justo Cobo and Christopher Glidewell**

### Computing details

Data collection: *APEX2* (Bruker, 2006) for (I), (II); *COLLECT* (Nonius, 1998) for (III). Cell refinement: *SAINT* (Bruker, 2006) for (I), (II); *DIRAX/LSQ* (Duisenberg *et al.*, 2000) for (III). Data reduction: *SAINT* (Bruker, 2006) for (I), (II);  *EVALCCD* (Duisenberg *et al.*, 2003) for (III). Program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994) for (I), (II); *SIR2014* (Burla *et al.*, 2015) for (III). For all compounds, program(s) used to refine structure: *SHELXL2014* (Sheldrick, 2015); molecular graphics: *PLATON* (Spek, 2009); software used to prepare material for publication: *SHELXL2014* (Sheldrick, 2015) and *PLATON* (Spek, 2009).

### (I) (9*RS*,15*RS*)-9-Ethyl-11-methyl-9,13*b*-dihydrodibenzo[*c,f*]thiazolo[3,2-*a*]azepin-3(2*H*)-one

#### Crystal data

C <sub>19</sub> H <sub>19</sub> NOS	$F(000) = 656$
$M_r = 309.41$	$D_x = 1.308 \text{ Mg m}^{-3}$
Monoclinic, $P2_1$	Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$
$a = 11.4261 (5) \text{ \AA}$	Cell parameters from 11002 reflections
$b = 8.1847 (3) \text{ \AA}$	$\theta = 1.8\text{--}32.5^\circ$
$c = 16.8243 (6) \text{ \AA}$	$\mu = 0.21 \text{ mm}^{-1}$
$\beta = 93.067 (2)^\circ$	$T = 100 \text{ K}$
$V = 1571.14 (11) \text{ \AA}^3$	Block, colourless
$Z = 4$	$0.40 \times 0.35 \times 0.22 \text{ mm}$

#### Data collection

Bruker Kappa APEXII diffractometer	61785 measured reflections
Radiation source: high brilliance microfocus sealed tube	11002 independent reflections
$\varphi$ and $\omega$ scans	10697 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (SADABS; Bruker, 2006)	$R_{\text{int}} = 0.025$
$T_{\text{min}} = 0.823$ , $T_{\text{max}} = 0.955$	$\theta_{\text{max}} = 32.5^\circ$ , $\theta_{\text{min}} = 1.8^\circ$
	$h = -17 \rightarrow 16$
	$k = -12 \rightarrow 12$
	$l = -25 \rightarrow 25$

#### Refinement

Refinement on $F^2$	$wR(F^2) = 0.079$
Least-squares matrix: full	$S = 1.05$
$R[F^2 > 2\sigma(F^2)] = 0.029$	11002 reflections

401 parameters  
 1 restraint  
 Hydrogen site location: inferred from  
 neighbouring sites  
 H-atom parameters constrained  
 $w = 1/[\sigma^2(F_o^2) + (0.0527P)^2 + 0.1585P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = 0.001$   
 $\Delta\rho_{\max} = 0.37 \text{ e } \text{Å}^{-3}$   
 $\Delta\rho_{\min} = -0.21 \text{ e } \text{Å}^{-3}$   
 Absolute structure: Flack  $x$  determined using  
 4787 quotients [(I+)-(I-)]/[(I+)+(I-)] (Parsons *et al.*, 2013)  
 Absolute structure parameter: 0.007 (6)

*Special details*

**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.

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å<sup>2</sup>)*

	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> <sub>iso</sub> */ <i>U</i> <sub>eq</sub>
S11	0.04257 (3)	0.64856 (4)	0.31677 (2)	0.02072 (7)
C12	-0.06240 (11)	0.6542 (2)	0.39359 (8)	0.0221 (2)
H12A	-0.0975	0.7643	0.3973	0.026*
H12B	-0.1258	0.5737	0.3825	0.026*
C13	0.00808 (11)	0.61139 (17)	0.46981 (8)	0.0198 (2)
O13	-0.03273 (10)	0.55030 (17)	0.52867 (7)	0.0298 (2)
N14	0.12297 (9)	0.65093 (16)	0.46388 (6)	0.01701 (19)
C14A	0.20563 (11)	0.63483 (17)	0.52973 (7)	0.0170 (2)
C15	0.19005 (13)	0.72209 (19)	0.59932 (8)	0.0221 (2)
H15	0.1246	0.7927	0.6032	0.027*
C16	0.27158 (15)	0.7045 (2)	0.66311 (8)	0.0256 (3)
H16	0.2618	0.7623	0.7113	0.031*
C17	0.36731 (14)	0.6021 (2)	0.65618 (8)	0.0243 (3)
H17	0.4241	0.5927	0.6993	0.029*
C18	0.38124 (11)	0.51298 (19)	0.58668 (7)	0.0195 (2)
H18	0.4466	0.4422	0.5832	0.023*
C18A	0.29966 (11)	0.52719 (16)	0.52223 (7)	0.0158 (2)
C19	0.30872 (10)	0.43915 (16)	0.44299 (7)	0.0151 (2)
H19	0.2274	0.4054	0.4252	0.018*
C19A	0.34743 (11)	0.56585 (16)	0.38285 (7)	0.0148 (2)
C110	0.45748 (11)	0.55481 (17)	0.35032 (7)	0.0164 (2)
H110	0.5071	0.4655	0.3650	0.020*
C111	0.49666 (11)	0.67094 (17)	0.29695 (7)	0.0173 (2)
C112	0.42386 (11)	0.80170 (18)	0.27585 (7)	0.0185 (2)
H112	0.4482	0.8808	0.2388	0.022*
C113	0.31535 (11)	0.81690 (17)	0.30887 (7)	0.0177 (2)
H113	0.2672	0.9083	0.2951	0.021*
C114	0.27587 (10)	0.70040 (16)	0.36181 (7)	0.0155 (2)
C115	0.15382 (11)	0.72903 (16)	0.38914 (7)	0.0160 (2)
H115	0.1415	0.8495	0.3942	0.019*
C116	0.61535 (12)	0.6532 (2)	0.26305 (8)	0.0246 (3)
H16A	0.6057	0.6325	0.2057	0.037*

H16B	0.6572	0.5615	0.2890	0.037*
H16C	0.6603	0.7539	0.2724	0.037*
C191	0.38285 (11)	0.28333 (17)	0.44701 (7)	0.0178 (2)
H19A	0.3929	0.2431	0.3923	0.021*
H19B	0.4615	0.3093	0.4713	0.021*
C192	0.32763 (11)	0.14856 (19)	0.49539 (8)	0.0211 (2)
H19C	0.3245	0.1837	0.5509	0.032*
H19D	0.3749	0.0490	0.4929	0.032*
H19E	0.2480	0.1265	0.4734	0.032*
S21	0.22593 (3)	0.00063 (5)	-0.08726 (2)	0.02161 (7)
C22	0.06874 (13)	0.02911 (19)	-0.08922 (8)	0.0224 (3)
H22A	0.0435	0.1113	-0.1298	0.027*
H22B	0.0276	-0.0750	-0.1016	0.027*
C23	0.04285 (12)	0.08804 (18)	-0.00633 (8)	0.0205 (2)
O23	-0.04535 (10)	0.16254 (18)	0.00951 (7)	0.0317 (3)
N24	0.13069 (9)	0.04697 (14)	0.04793 (6)	0.01589 (19)
C24A	0.12426 (10)	0.08689 (16)	0.13011 (7)	0.0151 (2)
C25	0.03501 (11)	0.02280 (18)	0.17405 (8)	0.0196 (2)
H25	-0.0202	-0.0517	0.1503	0.024*
C26	0.02786 (12)	0.06939 (18)	0.25329 (9)	0.0219 (2)
H26	-0.0332	0.0281	0.2838	0.026*
C27	0.11024 (12)	0.17634 (18)	0.28750 (8)	0.0207 (2)
H27	0.1057	0.2075	0.3416	0.025*
C28	0.19976 (11)	0.23845 (17)	0.24303 (7)	0.0175 (2)
H28	0.2562	0.3104	0.2674	0.021*
C28A	0.20726 (10)	0.19610 (15)	0.16314 (7)	0.0141 (2)
C29	0.30020 (10)	0.26061 (15)	0.10960 (7)	0.0140 (2)
H29	0.2589	0.2824	0.0567	0.017*
C29A	0.38728 (10)	0.12397 (15)	0.09590 (7)	0.0140 (2)
C210	0.50569 (10)	0.13608 (17)	0.12168 (7)	0.0157 (2)
H210	0.5318	0.2316	0.1494	0.019*
C211	0.58688 (10)	0.01222 (18)	0.10797 (7)	0.0170 (2)
C212	0.54650 (11)	-0.12996 (18)	0.06995 (7)	0.0183 (2)
H212	0.5995	-0.2166	0.0609	0.022*
C213	0.42911 (11)	-0.14551 (17)	0.04519 (7)	0.0167 (2)
H213	0.4027	-0.2437	0.0201	0.020*
C214	0.34913 (10)	-0.01951 (16)	0.05650 (7)	0.01384 (19)
C215	0.22787 (10)	-0.04930 (15)	0.01916 (7)	0.0144 (2)
H215	0.2088	-0.1678	0.0248	0.017*
C216	0.71526 (11)	0.0308 (2)	0.13229 (8)	0.0233 (3)
H26A	0.7347	0.1472	0.1370	0.035*
H26B	0.7625	-0.0195	0.0920	0.035*
H26C	0.7319	-0.0230	0.1837	0.035*
C291	0.35367 (11)	0.42370 (16)	0.13740 (8)	0.0172 (2)
H29A	0.3887	0.4117	0.1922	0.021*
H29B	0.4168	0.4547	0.1023	0.021*
C292	0.26090 (13)	0.55806 (18)	0.13589 (8)	0.0222 (2)
H29C	0.2227	0.5652	0.0824	0.033*

H29D	0.2982	0.6628	0.1498	0.033*
H29E	0.2023	0.5324	0.1745	0.033*

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

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
S11	0.01711 (13)	0.02520 (16)	0.01967 (13)	0.00113 (12)	-0.00079 (10)	-0.00009 (12)
C12	0.0153 (5)	0.0253 (7)	0.0256 (6)	0.0012 (5)	0.0011 (4)	0.0039 (5)
C13	0.0151 (5)	0.0196 (6)	0.0251 (6)	0.0016 (4)	0.0046 (4)	0.0031 (4)
O13	0.0208 (5)	0.0394 (7)	0.0298 (5)	-0.0007 (5)	0.0080 (4)	0.0110 (5)
N14	0.0143 (4)	0.0211 (5)	0.0160 (4)	0.0011 (4)	0.0037 (3)	0.0025 (4)
C14A	0.0177 (5)	0.0183 (6)	0.0153 (4)	-0.0010 (4)	0.0030 (4)	0.0013 (4)
C15	0.0276 (6)	0.0210 (6)	0.0183 (5)	0.0014 (5)	0.0063 (5)	-0.0008 (5)
C16	0.0371 (8)	0.0240 (7)	0.0159 (5)	-0.0034 (6)	0.0032 (5)	-0.0014 (5)
C17	0.0291 (7)	0.0283 (7)	0.0151 (5)	-0.0049 (5)	-0.0018 (5)	0.0008 (5)
C18	0.0183 (5)	0.0243 (6)	0.0159 (5)	-0.0018 (5)	0.0004 (4)	0.0033 (4)
C18A	0.0158 (5)	0.0179 (6)	0.0139 (4)	-0.0018 (4)	0.0026 (4)	0.0019 (4)
C19	0.0138 (5)	0.0174 (5)	0.0142 (4)	0.0006 (4)	0.0015 (4)	0.0014 (4)
C19A	0.0147 (5)	0.0173 (5)	0.0126 (4)	-0.0003 (4)	0.0019 (4)	0.0004 (4)
C110	0.0152 (5)	0.0205 (6)	0.0137 (4)	0.0000 (4)	0.0020 (4)	0.0001 (4)
C111	0.0160 (5)	0.0225 (6)	0.0136 (4)	-0.0019 (4)	0.0028 (4)	0.0001 (4)
C112	0.0192 (5)	0.0220 (6)	0.0145 (5)	-0.0024 (5)	0.0020 (4)	0.0028 (4)
C113	0.0182 (5)	0.0186 (6)	0.0162 (5)	-0.0007 (4)	0.0013 (4)	0.0028 (4)
C114	0.0143 (5)	0.0180 (5)	0.0143 (4)	-0.0004 (4)	0.0021 (4)	0.0003 (4)
C115	0.0153 (5)	0.0169 (5)	0.0159 (5)	0.0003 (4)	0.0026 (4)	0.0014 (4)
C116	0.0188 (5)	0.0323 (7)	0.0234 (6)	0.0010 (6)	0.0087 (4)	0.0042 (5)
C191	0.0158 (5)	0.0195 (6)	0.0181 (5)	0.0020 (4)	0.0025 (4)	0.0030 (4)
C192	0.0181 (5)	0.0204 (6)	0.0250 (5)	0.0006 (5)	0.0023 (4)	0.0051 (5)
S21	0.02265 (14)	0.02804 (18)	0.01408 (12)	-0.00534 (12)	0.00057 (10)	-0.00045 (11)
C22	0.0241 (6)	0.0233 (7)	0.0189 (5)	0.0003 (5)	-0.0065 (4)	-0.0005 (5)
C23	0.0192 (5)	0.0192 (6)	0.0223 (5)	0.0020 (5)	-0.0061 (4)	-0.0026 (5)
O23	0.0241 (5)	0.0360 (6)	0.0339 (5)	0.0138 (5)	-0.0088 (4)	-0.0078 (5)
N24	0.0131 (4)	0.0182 (5)	0.0162 (4)	0.0020 (4)	-0.0013 (3)	-0.0022 (4)
C24A	0.0133 (5)	0.0158 (5)	0.0164 (5)	0.0011 (4)	0.0010 (4)	-0.0006 (4)
C25	0.0141 (5)	0.0205 (6)	0.0245 (5)	-0.0009 (4)	0.0041 (4)	-0.0009 (5)
C26	0.0197 (6)	0.0215 (6)	0.0253 (6)	0.0009 (5)	0.0093 (5)	0.0024 (5)
C27	0.0238 (6)	0.0210 (6)	0.0180 (5)	0.0034 (5)	0.0062 (4)	0.0004 (4)
C28	0.0199 (5)	0.0174 (6)	0.0155 (5)	0.0009 (4)	0.0016 (4)	-0.0006 (4)
C28A	0.0133 (4)	0.0142 (5)	0.0150 (4)	0.0012 (4)	0.0014 (4)	-0.0002 (4)
C29	0.0149 (5)	0.0132 (5)	0.0140 (4)	-0.0013 (4)	0.0012 (4)	-0.0002 (4)
C29A	0.0132 (4)	0.0152 (5)	0.0138 (4)	-0.0006 (4)	0.0021 (3)	0.0012 (4)
C210	0.0149 (4)	0.0183 (5)	0.0140 (4)	-0.0023 (4)	0.0016 (3)	0.0014 (4)
C211	0.0134 (4)	0.0232 (6)	0.0145 (4)	0.0002 (4)	0.0023 (4)	0.0034 (4)
C212	0.0159 (5)	0.0226 (6)	0.0167 (5)	0.0034 (4)	0.0037 (4)	0.0007 (4)
C213	0.0171 (5)	0.0175 (6)	0.0159 (5)	0.0013 (4)	0.0032 (4)	-0.0010 (4)
C214	0.0133 (4)	0.0148 (5)	0.0136 (4)	-0.0005 (4)	0.0019 (3)	0.0000 (4)
C215	0.0139 (5)	0.0149 (5)	0.0144 (4)	0.0001 (4)	0.0008 (4)	-0.0009 (4)
C216	0.0131 (5)	0.0336 (8)	0.0231 (6)	0.0003 (5)	0.0002 (4)	0.0015 (5)

C291	0.0190 (5)	0.0149 (5)	0.0178 (5)	-0.0027 (4)	-0.0001 (4)	-0.0013 (4)
C292	0.0274 (6)	0.0155 (6)	0.0235 (6)	0.0006 (5)	0.0005 (5)	-0.0005 (4)

*Geometric parameters (Å, °)*

S11—C12	1.8102 (14)	S21—C22	1.8096 (15)
S11—C115	1.8348 (13)	S21—C215	1.8354 (12)
C12—C13	1.5184 (19)	C22—C23	1.520 (2)
C12—H12A	0.9900	C22—H22A	0.9900
C12—H12B	0.9900	C22—H22B	0.9900
C13—O13	1.2238 (16)	C23—O23	1.2196 (17)
C13—N14	1.3609 (16)	C23—N24	1.3623 (16)
N14—C14A	1.4229 (15)	N24—C24A	1.4264 (16)
N14—C115	1.4702 (16)	N24—C215	1.4648 (16)
C14A—C15	1.3911 (18)	C24A—C25	1.3937 (17)
C14A—C18A	1.4003 (18)	C24A—C28A	1.3968 (17)
C15—C16	1.390 (2)	C25—C26	1.3933 (19)
C15—H15	0.9500	C25—H25	0.9500
C16—C17	1.387 (2)	C26—C27	1.388 (2)
C16—H16	0.9500	C26—H26	0.9500
C17—C18	1.3947 (19)	C27—C28	1.3951 (18)
C17—H17	0.9500	C27—H27	0.9500
C18—C18A	1.3964 (17)	C28—C28A	1.3951 (16)
C18—H18	0.9500	C28—H28	0.9500
C18A—C19	1.5238 (17)	C28A—C29	1.5232 (17)
C19—C191	1.5305 (18)	C29—C29A	1.5226 (17)
C19—C19A	1.5306 (17)	C29—C291	1.5308 (17)
C19—H19	1.0000	C29—H29	1.0000
C19A—C110	1.4009 (16)	C29A—C210	1.4020 (16)
C19A—C114	1.4056 (18)	C29A—C214	1.4063 (17)
C110—C111	1.3975 (17)	C210—C211	1.4015 (18)
C110—H110	0.9500	C210—H210	0.9500
C111—C112	1.3899 (19)	C211—C212	1.3946 (19)
C111—C116	1.5061 (18)	C211—C216	1.5094 (17)
C112—C113	1.3911 (17)	C212—C213	1.3890 (18)
C112—H112	0.9500	C212—H212	0.9500
C113—C114	1.3960 (17)	C213—C214	1.3976 (17)
C113—H113	0.9500	C213—H213	0.9500
C114—C115	1.5098 (17)	C214—C215	1.5102 (16)
C115—H115	1.0000	C215—H215	1.0000
C116—H16A	0.9800	C216—H26A	0.9800
C116—H16B	0.9800	C216—H26B	0.9800
C116—H16C	0.9800	C216—H26C	0.9800
C191—C192	1.5271 (19)	C291—C292	1.527 (2)
C191—H19A	0.9900	C291—H29A	0.9900
C191—H19B	0.9900	C291—H29B	0.9900
C192—H19C	0.9800	C292—H29C	0.9800
C192—H19D	0.9800	C292—H29D	0.9800

C192—H19E	0.9800	C292—H29E	0.9800
C12—S11—C115	88.80 (6)	C22—S21—C215	90.38 (6)
C13—C12—S11	104.79 (9)	C23—C22—S21	105.42 (9)
C13—C12—H12A	110.8	C23—C22—H22A	110.7
S11—C12—H12A	110.8	S21—C22—H22A	110.7
C13—C12—H12B	110.8	C23—C22—H22B	110.7
S11—C12—H12B	110.8	S21—C22—H22B	110.7
H12A—C12—H12B	108.9	H22A—C22—H22B	108.8
O13—C13—N14	124.56 (13)	O23—C23—N24	124.38 (13)
O13—C13—C12	124.74 (12)	O23—C23—C22	124.26 (12)
N14—C13—C12	110.69 (11)	N24—C23—C22	111.36 (12)
C13—N14—C14A	121.57 (11)	C23—N24—C24A	121.25 (11)
C13—N14—C115	116.26 (10)	C23—N24—C215	117.10 (10)
C14A—N14—C115	121.78 (10)	C24A—N24—C215	121.52 (10)
C15—C14A—C18A	122.15 (12)	C25—C24A—C28A	122.04 (11)
C15—C14A—N14	119.88 (12)	C25—C24A—N24	120.33 (11)
C18A—C14A—N14	117.95 (11)	C28A—C24A—N24	117.59 (11)
C16—C15—C14A	119.06 (14)	C26—C25—C24A	119.11 (12)
C16—C15—H15	120.5	C26—C25—H25	120.4
C14A—C15—H15	120.5	C24A—C25—H25	120.4
C17—C16—C15	119.77 (13)	C27—C26—C25	119.76 (12)
C17—C16—H16	120.1	C27—C26—H26	120.1
C15—C16—H16	120.1	C25—C26—H26	120.1
C16—C17—C18	120.80 (13)	C26—C27—C28	120.53 (12)
C16—C17—H17	119.6	C26—C27—H27	119.7
C18—C17—H17	119.6	C28—C27—H27	119.7
C17—C18—C18A	120.40 (13)	C27—C28—C28A	120.72 (12)
C17—C18—H18	119.8	C27—C28—H28	119.6
C18A—C18—H18	119.8	C28A—C28—H28	119.6
C18—C18A—C14A	117.78 (12)	C28—C28A—C24A	117.83 (11)
C18—C18A—C19	124.48 (12)	C28—C28A—C29	124.36 (11)
C14A—C18A—C19	117.69 (11)	C24A—C28A—C29	117.81 (10)
C18A—C19—C19I	114.75 (10)	C29A—C29—C28A	108.47 (10)
C18A—C19—C19A	107.07 (10)	C29A—C29—C29I	115.68 (10)
C19I—C19—C19A	114.52 (10)	C28A—C29—C29I	113.64 (10)
C18A—C19—H19	106.6	C29A—C29—H29	106.1
C19I—C19—H19	106.6	C28A—C29—H29	106.1
C19A—C19—H19	106.6	C29I—C29—H29	106.1
C110—C19A—C114	118.32 (11)	C210—C29A—C214	118.33 (11)
C110—C19A—C19	120.78 (11)	C210—C29A—C29	121.93 (11)
C114—C19A—C19	120.83 (10)	C214—C29A—C29	119.74 (10)
C111—C110—C19A	122.16 (12)	C211—C210—C29A	122.29 (12)
C111—C110—H110	118.9	C211—C210—H210	118.9
C19A—C110—H110	118.9	C29A—C210—H210	118.9
C112—C111—C110	118.68 (11)	C212—C211—C210	118.26 (11)
C112—C111—C116	121.09 (12)	C212—C211—C216	120.09 (12)
C110—C111—C116	120.23 (12)	C210—C211—C216	121.64 (12)

C111—C112—C113	120.06 (12)	C213—C212—C211	120.30 (12)
C111—C112—H112	120.0	C213—C212—H212	119.9
C113—C112—H112	120.0	C211—C212—H212	119.9
C112—C113—C114	121.27 (12)	C212—C213—C214	121.30 (12)
C112—C113—H113	119.4	C212—C213—H213	119.3
C114—C113—H113	119.4	C214—C213—H213	119.3
C113—C114—C19A	119.49 (11)	C213—C214—C29A	119.47 (11)
C113—C114—C115	115.06 (11)	C213—C214—C215	114.60 (11)
C19A—C114—C115	125.39 (11)	C29A—C214—C215	125.82 (11)
N14—C115—C114	117.43 (10)	N24—C215—C214	118.07 (10)
N14—C115—S11	102.84 (8)	N24—C215—S21	103.46 (8)
C114—C115—S11	111.14 (8)	C214—C215—S21	109.44 (8)
N14—C115—H115	108.4	N24—C215—H215	108.5
C114—C115—H115	108.4	C214—C215—H215	108.5
S11—C115—H115	108.4	S21—C215—H215	108.5
C111—C116—H16A	109.5	C211—C216—H26A	109.5
C111—C116—H16B	109.5	C211—C216—H26B	109.5
H16A—C116—H16B	109.5	H26A—C216—H26B	109.5
C111—C116—H16C	109.5	C211—C216—H26C	109.5
H16A—C116—H16C	109.5	H26A—C216—H26C	109.5
H16B—C116—H16C	109.5	H26B—C216—H26C	109.5
C192—C191—C19	112.46 (10)	C292—C291—C29	110.95 (10)
C192—C191—H19A	109.1	C292—C291—H29A	109.4
C19—C191—H19A	109.1	C29—C291—H29A	109.4
C192—C191—H19B	109.1	C292—C291—H29B	109.4
C19—C191—H19B	109.1	C29—C291—H29B	109.4
H19A—C191—H19B	107.8	H29A—C291—H29B	108.0
C191—C192—H19C	109.5	C291—C292—H29C	109.5
C191—C192—H19D	109.5	C291—C292—H29D	109.5
H19C—C192—H19D	109.5	H29C—C292—H29D	109.5
C191—C192—H19E	109.5	C291—C292—H29E	109.5
H19C—C192—H19E	109.5	H29C—C292—H29E	109.5
H19D—C192—H19E	109.5	H29D—C292—H29E	109.5
C115—S11—C12—C13	-35.23 (10)	C215—S21—C22—C23	30.15 (10)
S11—C12—C13—O13	-154.57 (14)	S21—C22—C23—O23	158.58 (14)
S11—C12—C13—N14	25.70 (15)	S21—C22—C23—N24	-21.43 (15)
O13—C13—N14—C14A	-4.8 (2)	O23—C23—N24—C24A	1.3 (2)
C12—C13—N14—C14A	174.94 (13)	C22—C23—N24—C24A	-178.71 (12)
O13—C13—N14—C115	-177.78 (14)	O23—C23—N24—C215	177.26 (14)
C12—C13—N14—C115	1.96 (18)	C22—C23—N24—C215	-2.73 (17)
C13—N14—C14A—C15	-60.13 (19)	C23—N24—C24A—C25	61.92 (18)
C115—N14—C14A—C15	112.47 (15)	C215—N24—C24A—C25	-113.89 (13)
C13—N14—C14A—C18A	118.52 (14)	C23—N24—C24A—C28A	-115.71 (14)
C115—N14—C14A—C18A	-68.89 (17)	C215—N24—C24A—C28A	68.49 (16)
C18A—C14A—C15—C16	1.2 (2)	C28A—C24A—C25—C26	0.3 (2)
N14—C14A—C15—C16	179.83 (13)	N24—C24A—C25—C26	-177.25 (12)
C14A—C15—C16—C17	0.7 (2)	C24A—C25—C26—C27	-1.0 (2)

C15—C16—C17—C18	-1.8 (2)	C25—C26—C27—C28	0.4 (2)
C16—C17—C18—C18A	1.0 (2)	C26—C27—C28—C28A	0.9 (2)
C17—C18—C18A—C14A	0.90 (19)	C27—C28—C28A—C24A	-1.56 (19)
C17—C18—C18A—C19	178.15 (13)	C27—C28—C28A—C29	178.65 (12)
C15—C14A—C18A—C18	-2.03 (19)	C25—C24A—C28A—C28	1.00 (19)
N14—C14A—C18A—C18	179.36 (12)	N24—C24A—C28A—C28	178.58 (11)
C15—C14A—C18A—C19	-179.46 (12)	C25—C24A—C28A—C29	-179.20 (12)
N14—C14A—C18A—C19	1.92 (17)	N24—C24A—C28A—C29	-1.62 (17)
C18—C18A—C19—C191	23.65 (17)	C28—C28A—C29—C29A	106.61 (13)
C14A—C18A—C19—C191	-159.10 (11)	C24A—C28A—C29—C29A	-73.18 (13)
C18—C18A—C19—C19A	-104.63 (14)	C28—C28A—C29—C291	-23.55 (17)
C14A—C18A—C19—C19A	72.62 (13)	C24A—C28A—C29—C291	156.66 (11)
C18A—C19—C19A—C110	113.42 (12)	C28A—C29—C29A—C210	-117.02 (12)
C191—C19—C19A—C110	-15.00 (16)	C291—C29—C29A—C210	12.01 (16)
C18A—C19—C19A—C114	-63.48 (14)	C28A—C29—C29A—C214	62.82 (13)
C191—C19—C19A—C114	168.09 (11)	C291—C29—C29A—C214	-168.16 (10)
C114—C19A—C110—C111	-1.25 (18)	C214—C29A—C210—C211	1.41 (17)
C19—C19A—C110—C111	-178.23 (11)	C29—C29A—C210—C211	-178.75 (11)
C19A—C110—C111—C112	0.21 (19)	C29A—C210—C211—C212	-2.46 (18)
C19A—C110—C111—C116	-179.52 (12)	C29A—C210—C211—C216	176.74 (11)
C110—C111—C112—C113	1.25 (19)	C210—C211—C212—C213	1.27 (18)
C116—C111—C112—C113	-179.02 (13)	C216—C211—C212—C213	-177.94 (12)
C111—C112—C113—C114	-1.7 (2)	C211—C212—C213—C214	0.92 (18)
C112—C113—C114—C19A	0.62 (19)	C212—C213—C214—C29A	-1.98 (18)
C112—C113—C114—C115	-176.88 (12)	C212—C213—C214—C215	174.33 (11)
C110—C19A—C114—C113	0.82 (18)	C210—C29A—C214—C213	0.81 (16)
C19—C19A—C114—C113	177.80 (11)	C29—C29A—C214—C213	-179.03 (11)
C110—C19A—C114—C115	178.04 (11)	C210—C29A—C214—C215	-175.05 (11)
C19—C19A—C114—C115	-4.98 (19)	C29—C29A—C214—C215	5.11 (17)
C13—N14—C115—C114	-150.37 (12)	C23—N24—C215—C214	146.00 (12)
C14A—N14—C115—C114	36.66 (18)	C24A—N24—C215—C214	-38.03 (17)
C13—N14—C115—S11	-28.03 (14)	C23—N24—C215—S21	24.98 (13)
C14A—N14—C115—S11	159.00 (11)	C24A—N24—C215—S21	-159.05 (10)
C113—C114—C115—N14	-158.47 (12)	C213—C214—C215—N24	161.04 (10)
C19A—C114—C115—N14	24.20 (18)	C29A—C214—C215—N24	-22.92 (17)
C113—C114—C115—S11	83.55 (12)	C213—C214—C215—S21	-81.06 (12)
C19A—C114—C115—S11	-93.77 (13)	C29A—C214—C215—S21	94.98 (12)
C12—S11—C115—N14	35.54 (9)	C22—S21—C215—N24	-30.96 (9)
C12—S11—C115—C114	162.03 (10)	C22—S21—C215—C214	-157.65 (10)
C18A—C19—C191—C192	66.55 (14)	C29A—C29—C291—C292	170.60 (10)
C19A—C19—C191—C192	-169.01 (11)	C28A—C29—C291—C292	-62.94 (13)

Hydrogen-bond geometry ( $\text{\AA}$ ,  $^\circ$ )

<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>
C115—H115 $\cdots$ O13 <sup>i</sup>	1.00	2.47	3.3084 (18)	141
C215—H215 $\cdots$ O23 <sup>ii</sup>	1.00	2.37	3.1681 (18)	136



C112—H112...Cg1 <sup>iii</sup>	0.95	2.81	3.6738 (13)	152
C113—H113...Cg2 <sup>iii</sup>	0.95	2.85	3.7676 (14)	163

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

**(II) (9*RS*,15*RS*)-9-Ethyl-7,12-dimethyl-9,13b-dihydrodibenzo[*c,f*]thiazolo[3,2-*a*]azepin-3(2*H*)-one**

*Crystal data*

C <sub>20</sub> H <sub>21</sub> NOS	$F(000) = 1376$
$M_r = 323.44$	$D_x = 1.297 \text{ Mg m}^{-3}$
Monoclinic, $C2/c$	Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$
$a = 18.1021 (12) \text{ \AA}$	Cell parameters from 3402 reflections
$b = 12.4436 (8) \text{ \AA}$	$\theta = 2.0\text{--}26.4^\circ$
$c = 14.8429 (9) \text{ \AA}$	$\mu = 0.20 \text{ mm}^{-1}$
$\beta = 97.645 (3)^\circ$	$T = 298 \text{ K}$
$V = 3313.7 (4) \text{ \AA}^3$	Block, colourless
$Z = 8$	$0.23 \times 0.22 \times 0.20 \text{ mm}$

*Data collection*

Bruker Kappa APEXII diffractometer	30952 measured reflections
Radiation source: high brilliance microfocus sealed tube	3402 independent reflections
$\varphi$ and $\omega$ scans	2307 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (SADABS; Bruker, 2006)	$R_{\text{int}} = 0.048$
$T_{\text{min}} = 0.818, T_{\text{max}} = 0.961$	$\theta_{\text{max}} = 26.4^\circ, \theta_{\text{min}} = 2.0^\circ$
	$h = -22 \rightarrow 22$
	$k = -15 \rightarrow 13$
	$l = -14 \rightarrow 18$

*Refinement*

Refinement on $F^2$	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.060$	$w = 1/[\sigma^2(F_o^2) + (0.070P)^2 + 4.8478P]$
$wR(F^2) = 0.168$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.05$	$(\Delta/\sigma)_{\text{max}} < 0.001$
3402 reflections	$\Delta\rho_{\text{max}} = 0.57 \text{ e \AA}^{-3}$
211 parameters	$\Delta\rho_{\text{min}} = -0.45 \text{ e \AA}^{-3}$
0 restraints	

*Special details*

**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.

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ( $\text{\AA}^2$ )*

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
S1	0.40943 (5)	0.29645 (9)	0.65893 (6)	0.0801 (4)
C2	0.32187 (17)	0.2270 (3)	0.6390 (2)	0.0610 (8)
H2A	0.3186	0.1744	0.6866	0.073*
H2B	0.2811	0.2776	0.6391	0.073*
C3	0.31728 (16)	0.1719 (2)	0.5480 (2)	0.0524 (7)
O3	0.26206 (12)	0.12615 (19)	0.51154 (16)	0.0669 (6)

N4	0.38267 (12)	0.18081 (18)	0.51161 (15)	0.0466 (6)
C4A	0.38797 (15)	0.1538 (2)	0.41955 (18)	0.0453 (6)
C5	0.37768 (18)	0.0494 (2)	0.3880 (2)	0.0589 (8)
H5	0.3673	-0.0052	0.4272	0.071*
C6	0.38296 (18)	0.0273 (2)	0.2984 (2)	0.0612 (8)
H6	0.3754	-0.0429	0.2775	0.073*
C7	0.39919 (15)	0.1061 (2)	0.23845 (19)	0.0488 (7)
C8	0.40901 (14)	0.2104 (2)	0.27151 (17)	0.0421 (6)
H8	0.4194	0.2648	0.2320	0.051*
C8A	0.40380 (13)	0.23624 (19)	0.36146 (17)	0.0392 (6)
C9	0.41873 (14)	0.34878 (19)	0.40082 (17)	0.0403 (6)
H9	0.3821	0.3619	0.4427	0.048*
C9A	0.49525 (14)	0.34356 (19)	0.45798 (17)	0.0405 (6)
C10	0.55658 (16)	0.4003 (2)	0.43479 (19)	0.0486 (7)
H170	0.5497	0.4480	0.3862	0.058*
C11	0.62705 (16)	0.3875 (3)	0.4820 (2)	0.0571 (8)
H11	0.6664	0.4268	0.4645	0.069*
C12	0.64037 (16)	0.3175 (3)	0.5547 (2)	0.0548 (8)
C13	0.57916 (15)	0.2630 (2)	0.57933 (19)	0.0494 (7)
H13	0.5863	0.2162	0.6286	0.059*
C14	0.50769 (14)	0.2758 (2)	0.53333 (17)	0.0425 (6)
C15	0.44750 (15)	0.2154 (2)	0.57304 (18)	0.0484 (7)
H15	0.4703	0.1512	0.6034	0.058*
C71	0.40746 (18)	0.0815 (3)	0.1414 (2)	0.0613 (8)
H71A	0.3721	0.0272	0.1187	0.092*
H71B	0.4571	0.0560	0.1379	0.092*
H71C	0.3985	0.1455	0.1055	0.092*
C91	0.40972 (15)	0.4383 (2)	0.33031 (18)	0.0475 (7)
H91A	0.4249	0.5058	0.3598	0.057*
H91B	0.4426	0.4243	0.2851	0.057*
C92	0.33049 (18)	0.4491 (3)	0.2831 (2)	0.0630 (8)
H92A	0.3276	0.5085	0.2415	0.095*
H92B	0.2975	0.4614	0.3276	0.095*
H92C	0.3162	0.3842	0.2503	0.095*
C121	0.71688 (18)	0.3000 (3)	0.6059 (3)	0.0804 (11)
H12A	0.7308	0.2260	0.6009	0.121*
H12B	0.7166	0.3179	0.6687	0.121*
H12C	0.7521	0.3449	0.5807	0.121*

Atomic displacement parameters ( $\text{\AA}^2$ )

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
S1	0.0711 (6)	0.1163 (8)	0.0586 (5)	-0.0213 (5)	0.0302 (4)	-0.0193 (5)
C2	0.0511 (17)	0.081 (2)	0.0540 (17)	0.0063 (16)	0.0195 (14)	0.0199 (16)
C3	0.0453 (16)	0.0565 (17)	0.0574 (17)	0.0010 (14)	0.0140 (13)	0.0231 (14)
O3	0.0440 (12)	0.0749 (15)	0.0830 (15)	-0.0105 (11)	0.0124 (11)	0.0168 (12)
N4	0.0427 (13)	0.0499 (13)	0.0489 (13)	-0.0057 (10)	0.0120 (10)	0.0065 (10)
C4A	0.0408 (14)	0.0438 (14)	0.0523 (15)	-0.0048 (11)	0.0094 (12)	0.0030 (12)

C5	0.067 (2)	0.0446 (16)	0.0644 (19)	-0.0152 (14)	0.0072 (15)	0.0090 (14)
C6	0.073 (2)	0.0397 (15)	0.068 (2)	-0.0096 (14)	-0.0006 (16)	-0.0059 (14)
C7	0.0444 (15)	0.0468 (15)	0.0538 (16)	0.0003 (12)	0.0012 (12)	-0.0047 (13)
C8	0.0377 (14)	0.0407 (14)	0.0488 (15)	-0.0003 (11)	0.0090 (11)	0.0026 (12)
C8A	0.0317 (12)	0.0362 (13)	0.0509 (15)	-0.0005 (10)	0.0099 (11)	0.0021 (11)
C9	0.0430 (14)	0.0376 (13)	0.0441 (14)	0.0001 (11)	0.0192 (11)	-0.0010 (11)
C9A	0.0410 (14)	0.0358 (13)	0.0474 (15)	-0.0020 (11)	0.0159 (11)	-0.0077 (11)
C10	0.0544 (17)	0.0455 (15)	0.0497 (15)	-0.0087 (13)	0.0211 (13)	-0.0082 (12)
C11	0.0441 (17)	0.0680 (19)	0.0632 (19)	-0.0156 (14)	0.0218 (14)	-0.0191 (16)
C12	0.0428 (16)	0.0672 (19)	0.0559 (17)	-0.0022 (13)	0.0125 (13)	-0.0187 (15)
C13	0.0475 (16)	0.0523 (16)	0.0488 (15)	0.0031 (13)	0.0084 (13)	-0.0083 (13)
C14	0.0425 (15)	0.0416 (14)	0.0456 (14)	-0.0004 (11)	0.0145 (12)	-0.0058 (11)
C15	0.0471 (16)	0.0520 (16)	0.0470 (15)	-0.0037 (13)	0.0097 (12)	0.0064 (13)
C71	0.064 (2)	0.0570 (18)	0.0623 (19)	0.0020 (15)	0.0050 (15)	-0.0142 (15)
C91	0.0530 (16)	0.0403 (14)	0.0530 (16)	0.0013 (12)	0.0208 (13)	0.0024 (12)
C92	0.063 (2)	0.0562 (18)	0.071 (2)	0.0113 (15)	0.0147 (16)	0.0179 (15)
C121	0.0466 (19)	0.111 (3)	0.082 (2)	-0.0024 (19)	0.0051 (17)	-0.022 (2)

*Geometric parameters (Å, °)*

S1—C2	1.795 (3)	C9A—C10	1.397 (4)
S1—C15	1.831 (3)	C10—C11	1.381 (4)
C2—C3	1.508 (4)	C10—H170	0.9300
C2—H2A	0.9700	C11—C12	1.382 (4)
C2—H2B	0.9700	C11—H11	0.9300
C3—O3	1.214 (4)	C12—C13	1.389 (4)
C3—N4	1.369 (3)	C12—C121	1.504 (4)
N4—C4A	1.423 (3)	C13—C14	1.389 (4)
N4—C15	1.452 (3)	C13—H13	0.9300
C4A—C5	1.385 (4)	C14—C15	1.507 (4)
C4A—C8A	1.394 (3)	C15—H15	0.9800
C5—C6	1.374 (4)	C71—H71A	0.9600
C5—H5	0.9300	C71—H71B	0.9600
C6—C7	1.382 (4)	C71—H71C	0.9600
C6—H6	0.9300	C91—C92	1.516 (4)
C7—C8	1.391 (4)	C91—H91A	0.9700
C7—C71	1.499 (4)	C91—H91B	0.9700
C8—C8A	1.389 (3)	C92—H92A	0.9600
C8—H8	0.9300	C92—H92B	0.9600
C8A—C9	1.528 (3)	C92—H92C	0.9600
C9—C91	1.522 (3)	C121—H12A	0.9600
C9—C9A	1.527 (4)	C121—H12B	0.9600
C9—H9	0.9800	C121—H12C	0.9600
C9A—C14	1.395 (4)		
C2—S1—C15	91.50 (14)	C10—C11—C12	121.5 (3)
C3—C2—S1	107.97 (19)	C10—C11—H11	119.3
C3—C2—H2A	110.1	C12—C11—H11	119.3

S1—C2—H2A	110.1	C11—C12—C13	116.8 (3)
C3—C2—H2B	110.1	C11—C12—C121	122.6 (3)
S1—C2—H2B	110.1	C13—C12—C121	120.6 (3)
H2A—C2—H2B	108.4	C12—C13—C14	122.6 (3)
O3—C3—N4	124.5 (3)	C12—C13—H13	118.7
O3—C3—C2	123.8 (3)	C14—C13—H13	118.7
N4—C3—C2	111.7 (3)	C13—C14—C9A	120.2 (2)
C3—N4—C4A	122.1 (2)	C13—C14—C15	115.2 (2)
C3—N4—C15	116.4 (2)	C9A—C14—C15	124.6 (2)
C4A—N4—C15	121.4 (2)	N4—C15—C14	117.9 (2)
C5—C4A—C8A	120.7 (3)	N4—C15—S1	104.64 (18)
C5—C4A—N4	121.5 (2)	C14—C15—S1	110.88 (19)
C8A—C4A—N4	117.8 (2)	N4—C15—H15	107.7
C6—C5—C4A	119.5 (3)	C14—C15—H15	107.7
C6—C5—H5	120.3	S1—C15—H15	107.7
C4A—C5—H5	120.3	C7—C71—H71A	109.5
C5—C6—C7	121.9 (3)	C7—C71—H71B	109.5
C5—C6—H6	119.0	H71A—C71—H71B	109.5
C7—C6—H6	119.0	C7—C71—H71C	109.5
C6—C7—C8	117.6 (3)	H71A—C71—H71C	109.5
C6—C7—C71	122.0 (3)	H71B—C71—H71C	109.5
C8—C7—C71	120.3 (3)	C92—C91—C9	113.0 (2)
C8A—C8—C7	122.2 (2)	C92—C91—H91A	109.0
C8A—C8—H8	118.9	C9—C91—H91A	109.0
C7—C8—H8	118.9	C92—C91—H91B	109.0
C8—C8A—C4A	118.1 (2)	C9—C91—H91B	109.0
C8—C8A—C9	123.3 (2)	H91A—C91—H91B	107.8
C4A—C8A—C9	118.6 (2)	C91—C92—H92A	109.5
C91—C9—C9A	114.8 (2)	C91—C92—H92B	109.5
C91—C9—C8A	114.2 (2)	H92A—C92—H92B	109.5
C9A—C9—C8A	106.0 (2)	C91—C92—H92C	109.5
C91—C9—H9	107.1	H92A—C92—H92C	109.5
C9A—C9—H9	107.1	H92B—C92—H92C	109.5
C8A—C9—H9	107.1	C12—C121—H12A	109.5
C14—C9A—C10	117.0 (2)	C12—C121—H12B	109.5
C14—C9A—C9	120.6 (2)	H12A—C121—H12B	109.5
C10—C9A—C9	122.3 (2)	C12—C121—H12C	109.5
C11—C10—C9A	121.9 (3)	H12A—C121—H12C	109.5
C11—C10—H170	119.1	H12B—C121—H12C	109.5
C9A—C10—H170	119.1		
C15—S1—C2—C3	-18.5 (2)	C8A—C9—C9A—C14	-62.2 (3)
S1—C2—C3—O3	-173.3 (2)	C91—C9—C9A—C10	-13.3 (3)
S1—C2—C3—N4	6.3 (3)	C8A—C9—C9A—C10	113.8 (2)
O3—C3—N4—C4A	11.8 (4)	C14—C9A—C10—C11	2.2 (4)
C2—C3—N4—C4A	-167.8 (2)	C9—C9A—C10—C11	-173.9 (2)
O3—C3—N4—C15	-165.9 (3)	C9A—C10—C11—C12	0.1 (4)
C2—C3—N4—C15	14.6 (3)	C10—C11—C12—C13	-1.7 (4)

C3—N4—C4A—C5	-65.0 (4)	C10—C11—C12—C121	178.5 (3)
C15—N4—C4A—C5	112.5 (3)	C11—C12—C13—C14	0.9 (4)
C3—N4—C4A—C8A	114.9 (3)	C121—C12—C13—C14	-179.2 (3)
C15—N4—C4A—C8A	-67.6 (3)	C12—C13—C14—C9A	1.4 (4)
C8A—C4A—C5—C6	-0.2 (4)	C12—C13—C14—C15	-176.5 (2)
N4—C4A—C5—C6	179.7 (3)	C10—C9A—C14—C13	-2.9 (3)
C4A—C5—C6—C7	0.8 (5)	C9—C9A—C14—C13	173.3 (2)
C5—C6—C7—C8	-1.0 (4)	C10—C9A—C14—C15	174.8 (2)
C5—C6—C7—C71	178.0 (3)	C9—C9A—C14—C15	-9.0 (4)
C6—C7—C8—C8A	0.7 (4)	C3—N4—C15—C14	-151.3 (2)
C71—C7—C8—C8A	-178.3 (3)	C4A—N4—C15—C14	31.0 (4)
C7—C8—C8A—C4A	-0.2 (4)	C3—N4—C15—S1	-27.6 (3)
C7—C8—C8A—C9	176.4 (2)	C4A—N4—C15—S1	154.7 (2)
C5—C4A—C8A—C8	-0.1 (4)	C13—C14—C15—N4	-150.7 (2)
N4—C4A—C8A—C8	-180.0 (2)	C9A—C14—C15—N4	31.5 (4)
C5—C4A—C8A—C9	-176.8 (2)	C13—C14—C15—S1	88.7 (3)
N4—C4A—C8A—C9	3.3 (4)	C9A—C14—C15—S1	-89.0 (3)
C8—C8A—C9—C91	22.9 (3)	C2—S1—C15—N4	25.27 (19)
C4A—C8A—C9—C91	-160.6 (2)	C2—S1—C15—C14	153.4 (2)
C8—C8A—C9—C9A	-104.5 (3)	C9A—C9—C91—C92	-173.0 (2)
C4A—C8A—C9—C9A	72.0 (3)	C8A—C9—C91—C92	64.3 (3)
C91—C9—C9A—C14	170.8 (2)		

*Hydrogen-bond geometry (Å, °)*

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
C2—H2 <i>B</i> ...O3 <sup>i</sup>	0.97	2.57	3.120 (4)	116
C91—H91 <i>A</i> ...C <i>g</i> 3 <sup>ii</sup>	0.97	2.82	3.736 (3)	157

Symmetry codes: (i)  $-x+1/2, -y+1/2, -z+1$ ; (ii)  $-x+1, -y+1, -z+1$ .**(III) Ethyl 2-chloro-13-ethyl-4-oxo-8,13-dihydro-4*H*-benzo[5,6]azepino[3,2,1-*ij*]quinoline-5-carboxylate***Crystal data*C<sub>22</sub>H<sub>20</sub>ClNO<sub>3</sub>*M<sub>r</sub>* = 381.84Triclinic, *P*1*a* = 6.8533 (16) Å*b* = 10.612 (5) Å*c* = 13.561 (3) Å $\alpha$  = 72.45 (4)° $\beta$  = 75.840 (19)° $\gamma$  = 82.46 (2)°*V* = 910.0 (6) Å<sup>3</sup>*Z* = 2*F*(000) = 400*D<sub>x</sub>* = 1.394 Mg m<sup>-3</sup>Mo *K*α radiation,  $\lambda$  = 0.71073 Å

Cell parameters from 4176 reflections

 $\theta$  = 2.9–27.5° $\mu$  = 0.23 mm<sup>-1</sup>*T* = 120 K

Needle, colourless

0.26 × 0.15 × 0.13 mm

*Data collection*

Nonius KappaCCD

diffractometer

Radiation source: fine focus sealed tube

Graphite monochromator

 $\varphi$  and  $\omega$  scans

Absorption correction: multi-scan

(SADABS; Sheldrick, 2003)

*T<sub>min</sub>* = 0.845, *T<sub>max</sub>* = 0.970

20183 measured reflections

3776 independent reflections

2195 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.154$   
 $\theta_{\text{max}} = 26.6^\circ$ ,  $\theta_{\text{min}} = 3.8^\circ$

$h = -8 \rightarrow 8$   
 $k = -13 \rightarrow 13$   
 $l = -16 \rightarrow 17$

*Refinement*

Refinement on  $F^2$   
 Least-squares matrix: full  
 $R[F^2 > 2\sigma(F^2)] = 0.065$   
 $wR(F^2) = 0.118$   
 $S = 1.06$   
 3776 reflections  
 322 parameters  
 74 restraints

Hydrogen site location: inferred from neighbouring sites  
 H-atom parameters constrained  
 $w = 1/[\sigma^2(F_o^2) + (0.0192P)^2 + 1.1423P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\text{max}} < 0.001$   
 $\Delta\rho_{\text{max}} = 0.29 \text{ e } \text{Å}^{-3}$   
 $\Delta\rho_{\text{min}} = -0.31 \text{ e } \text{Å}^{-3}$

*Special details*

**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.

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ( $\text{Å}^2$ )*

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$	Occ. (<1)
C11	0.8032 (8)	0.3061 (4)	0.6534 (3)	0.0131 (8)	0.900 (6)
H11	0.8219	0.2338	0.7126	0.016*	0.900 (6)
C12	0.8306 (11)	0.2830 (4)	0.5549 (3)	0.0131 (8)	0.900 (6)
Cl12	0.9006 (7)	0.1222 (2)	0.5455 (3)	0.0224 (4)	0.900 (6)
C13	0.8071 (8)	0.3838 (4)	0.4674 (3)	0.0134 (10)	0.900 (6)
H13	0.8313	0.3677	0.4001	0.016*	0.900 (6)
C13A	0.7467 (12)	0.5117 (5)	0.4780 (3)	0.0138 (10)	0.900 (6)
C14	0.7273 (10)	0.6182 (5)	0.3794 (3)	0.0128 (11)	0.900 (6)
O14	0.750 (2)	0.5900 (6)	0.2952 (3)	0.0201 (10)	0.900 (6)
C15	0.675 (2)	0.7488 (5)	0.3946 (4)	0.0154 (9)	0.900 (6)
C16	0.623 (2)	0.7619 (4)	0.4956 (4)	0.0146 (12)	0.900 (6)
H16	0.5786	0.8476	0.5036	0.018*	0.900 (6)
N17	0.6312 (15)	0.6629 (4)	0.5838 (3)	0.0129 (7)	0.900 (6)
C18	0.5458 (9)	0.6900 (5)	0.6874 (4)	0.0151 (8)	0.900 (6)
H18A	0.4500	0.6222	0.7318	0.018*	0.900 (6)
H18B	0.4708	0.7777	0.6761	0.018*	0.900 (6)
C18A	0.7098 (10)	0.6883 (4)	0.7436 (5)	0.0147 (9)	0.900 (6)
C19	0.7676 (9)	0.8073 (4)	0.7489 (6)	0.0198 (14)	0.900 (6)
H19	0.6997	0.8888	0.7191	0.024*	0.900 (6)
C110	0.9232 (11)	0.8066 (5)	0.7972 (7)	0.0250 (13)	0.900 (6)
H110	0.9668	0.8877	0.7979	0.030*	0.900 (6)
C111	1.0156 (8)	0.6872 (5)	0.8449 (5)	0.0244 (12)	0.900 (6)
H111	1.1171	0.6862	0.8819	0.029*	0.900 (6)
C112	0.9603 (7)	0.5682 (5)	0.8387 (4)	0.0201 (11)	0.900 (6)
H112	1.0274	0.4871	0.8697	0.024*	0.900 (6)
C12A	0.8079 (6)	0.5675 (4)	0.7876 (3)	0.0155 (10)	0.900 (6)
C113	0.7482 (7)	0.4385 (4)	0.7797 (3)	0.0164 (9)	0.900 (6)

H113	0.8579	0.3720	0.8023	0.020*	0.900 (6)
C13C	0.7491 (7)	0.4319 (4)	0.6683 (3)	0.0132 (10)	0.900 (6)
C131	0.5547 (6)	0.3870 (4)	0.8619 (3)	0.0206 (10)	0.900 (6)
H13A	0.4468	0.4590	0.8571	0.025*	0.900 (6)
H13B	0.5095	0.3134	0.8439	0.025*	0.900 (6)
C132	0.5838 (8)	0.3386 (6)	0.9751 (3)	0.0223 (14)	0.900 (6)
H13C	0.6297	0.4106	0.9935	0.033*	0.900 (6)
H13D	0.6849	0.2638	0.9816	0.033*	0.900 (6)
H13E	0.4556	0.3101	1.0234	0.033*	0.900 (6)
C13B	0.7097 (12)	0.5356 (5)	0.5784 (3)	0.0137 (11)	0.900 (6)
C151	0.662 (2)	0.8739 (5)	0.3103 (4)	0.0181 (9)	0.900 (6)
O151	0.6227 (17)	0.9825 (6)	0.3249 (5)	0.0256 (15)	0.900 (6)
O152	0.707 (4)	0.8574 (8)	0.2120 (4)	0.0237 (10)	0.900 (6)
C152	0.6960 (10)	0.9782 (7)	0.1264 (4)	0.0234 (16)	0.900 (6)
H15A	0.7887	1.0424	0.1262	0.028*	0.900 (6)
H15B	0.5571	1.0197	0.1342	0.028*	0.900 (6)
C153	0.757 (3)	0.9364 (13)	0.0249 (5)	0.029 (2)	0.900 (6)
H15C	0.7525	1.0143	-0.0359	0.043*	0.900 (6)
H15D	0.8948	0.8953	0.0186	0.043*	0.900 (6)
H15E	0.6645	0.8727	0.0265	0.043*	0.900 (6)
C21	0.822 (8)	0.330 (3)	0.645 (2)	0.0131 (8)	0.100 (6)
H21	0.8866	0.2706	0.6970	0.016*	0.100 (6)
C22	0.844 (11)	0.307 (2)	0.546 (2)	0.0131 (8)	0.100 (6)
C122	0.889 (7)	0.146 (2)	0.536 (3)	0.0224 (4)	0.100 (6)
C23	0.830 (9)	0.410 (3)	0.459 (2)	0.0134 (10)	0.100 (6)
H23	0.8925	0.4039	0.3896	0.016*	0.100 (6)
C23A	0.720 (12)	0.527 (3)	0.474 (2)	0.0138 (10)	0.100 (6)
C24	0.694 (12)	0.634 (4)	0.377 (2)	0.0128 (11)	0.100 (6)
O24	0.73 (2)	0.607 (5)	0.291 (3)	0.0201 (10)	0.100 (6)
C25	0.68 (2)	0.766 (3)	0.390 (3)	0.0154 (9)	0.100 (6)
C26	0.64 (2)	0.780 (3)	0.490 (3)	0.0146 (12)	0.100 (6)
H26	0.6103	0.8675	0.4974	0.018*	0.100 (6)
N27	0.630 (14)	0.680 (3)	0.579 (2)	0.0129 (7)	0.100 (6)
C28	0.536 (7)	0.710 (4)	0.681 (3)	0.0151 (8)	0.100 (6)
H28A	0.4242	0.6517	0.7199	0.018*	0.100 (6)
H28B	0.4789	0.8031	0.6667	0.018*	0.100 (6)
C28A	0.689 (9)	0.691 (2)	0.748 (5)	0.0147 (9)	0.100 (6)
C29	0.800 (10)	0.797 (3)	0.739 (6)	0.0198 (14)	0.100 (6)
H29	0.7762	0.8816	0.6922	0.024*	0.100 (6)
C210	0.945 (11)	0.778 (4)	0.798 (7)	0.0250 (13)	0.100 (6)
H210	0.9976	0.8513	0.8064	0.030*	0.100 (6)
C211	1.013 (8)	0.650 (5)	0.844 (6)	0.0244 (12)	0.100 (6)
H211	1.1323	0.6345	0.8710	0.029*	0.100 (6)
C212	0.908 (7)	0.543 (4)	0.850 (4)	0.0201 (11)	0.100 (6)
H212	0.9427	0.4563	0.8910	0.024*	0.100 (6)
C22A	0.752 (6)	0.561 (2)	0.798 (3)	0.0155 (10)	0.100 (6)
C213	0.664 (5)	0.446 (2)	0.7812 (17)	0.0164 (9)	0.100 (6)
H223	0.5160	0.4694	0.7970	0.020*	0.100 (6)

C23C	0.704 (8)	0.441 (3)	0.6666 (17)	0.0132 (10)	0.100 (6)
C231	0.684 (5)	0.314 (2)	0.8666 (19)	0.0206 (10)	0.100 (6)
H23A	0.5920	0.2518	0.8633	0.025*	0.100 (6)
H23B	0.8238	0.2748	0.8512	0.025*	0.100 (6)
C232	0.636 (11)	0.330 (5)	0.9781 (19)	0.0223 (14)	0.100 (6)
H23C	0.6340	0.2425	1.0298	0.033*	0.100 (6)
H23D	0.5043	0.3775	0.9910	0.033*	0.100 (6)
H23E	0.7398	0.3803	0.9851	0.033*	0.100 (6)
C23B	0.679 (13)	0.549 (3)	0.576 (2)	0.0137 (11)	0.100 (6)
C251	0.66 (2)	0.891 (4)	0.305 (3)	0.0181 (9)	0.100 (6)
O251	0.592 (18)	0.996 (5)	0.320 (5)	0.0256 (15)	0.100 (6)
O252	0.70 (4)	0.871 (7)	0.208 (4)	0.0237 (10)	0.100 (6)
C252	0.649 (14)	0.985 (7)	0.124 (4)	0.0234 (16)	0.100 (6)
H25A	0.7156	1.0637	0.1225	0.028*	0.100 (6)
H25B	0.5016	1.0068	0.1354	0.028*	0.100 (6)
C253	0.73 (3)	0.947 (12)	0.022 (4)	0.029 (2)	0.100 (6)
H25C	0.7002	1.0204	-0.0378	0.043*	0.100 (6)
H25D	0.8737	0.9256	0.0125	0.043*	0.100 (6)
H25E	0.6609	0.8689	0.0254	0.043*	0.100 (6)

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

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
C11	0.0130 (16)	0.0069 (15)	0.0188 (14)	-0.0051 (16)	-0.0023 (12)	-0.0017 (11)
C12	0.0130 (16)	0.0069 (15)	0.0188 (14)	-0.0051 (16)	-0.0023 (12)	-0.0017 (11)
Cl12	0.0303 (8)	0.0107 (10)	0.0255 (9)	0.0016 (10)	-0.0043 (6)	-0.0069 (9)
C13	0.008 (2)	0.017 (2)	0.0168 (19)	-0.0060 (19)	0.0014 (15)	-0.0073 (16)
C13A	0.010 (3)	0.016 (2)	0.0157 (18)	-0.0007 (16)	-0.0053 (14)	-0.0035 (15)
C14	0.008 (4)	0.018 (2)	0.0136 (17)	0.0013 (16)	-0.0052 (14)	-0.0046 (15)
O14	0.024 (3)	0.021 (2)	0.0164 (13)	0.003 (3)	-0.0057 (12)	-0.0078 (13)
C15	0.0154 (19)	0.017 (2)	0.0115 (17)	0.000 (3)	-0.0043 (16)	-0.0001 (15)
C16	0.015 (3)	0.012 (2)	0.0172 (18)	-0.001 (3)	-0.0053 (17)	-0.0032 (16)
N17	0.0182 (16)	0.0090 (18)	0.0106 (14)	0.0009 (19)	-0.0040 (13)	-0.0014 (12)
C18	0.0159 (19)	0.017 (2)	0.0121 (18)	0.0032 (17)	-0.0020 (14)	-0.0060 (17)
C18A	0.014 (2)	0.0192 (18)	0.0096 (17)	0.0003 (14)	0.0009 (17)	-0.0049 (14)
C19	0.026 (3)	0.0171 (19)	0.015 (3)	-0.0041 (18)	0.001 (2)	-0.0049 (17)
C110	0.028 (3)	0.026 (3)	0.024 (2)	-0.010 (3)	-0.006 (2)	-0.009 (3)
C111	0.020 (2)	0.036 (3)	0.025 (2)	-0.007 (2)	-0.0062 (17)	-0.018 (3)
C112	0.013 (3)	0.027 (3)	0.018 (2)	0.0031 (19)	-0.002 (2)	-0.0055 (19)
C12A	0.015 (3)	0.0191 (18)	0.0097 (18)	-0.0027 (16)	0.0046 (19)	-0.0055 (15)
C113	0.020 (2)	0.0177 (19)	0.0114 (18)	0.0014 (17)	-0.0055 (17)	-0.0029 (14)
C13C	0.012 (3)	0.0152 (18)	0.0138 (17)	-0.0004 (15)	-0.0044 (15)	-0.0042 (14)
C131	0.026 (2)	0.018 (2)	0.017 (2)	-0.0053 (17)	-0.0030 (17)	-0.0037 (17)
C132	0.027 (4)	0.022 (2)	0.0172 (19)	0.000 (2)	-0.0055 (18)	-0.0029 (16)
C13B	0.008 (4)	0.0157 (19)	0.0180 (17)	-0.0012 (15)	-0.0036 (15)	-0.0046 (15)
C151	0.0148 (18)	0.021 (2)	0.0184 (19)	-0.004 (3)	-0.0043 (17)	-0.0037 (17)
O151	0.040 (5)	0.0151 (18)	0.0198 (15)	0.0079 (17)	-0.0102 (16)	-0.0028 (14)
O152	0.039 (4)	0.018 (2)	0.0124 (13)	-0.001 (3)	-0.0069 (15)	-0.0001 (13)



C152	0.030 (5)	0.021 (2)	0.0154 (18)	0.000 (3)	-0.008 (2)	0.0024 (16)
C153	0.039 (7)	0.030 (3)	0.0157 (19)	-0.016 (3)	-0.0037 (18)	0.0014 (18)
C21	0.0130 (16)	0.0069 (15)	0.0188 (14)	-0.0051 (16)	-0.0023 (12)	-0.0017 (11)
C22	0.0130 (16)	0.0069 (15)	0.0188 (14)	-0.0051 (16)	-0.0023 (12)	-0.0017 (11)
Cl22	0.0303 (8)	0.0107 (10)	0.0255 (9)	0.0016 (10)	-0.0043 (6)	-0.0069 (9)
C23	0.008 (2)	0.017 (2)	0.0168 (19)	-0.0060 (19)	0.0014 (15)	-0.0073 (16)
C23A	0.010 (3)	0.016 (2)	0.0157 (18)	-0.0007 (16)	-0.0053 (14)	-0.0035 (15)
C24	0.008 (4)	0.018 (2)	0.0136 (17)	0.0013 (16)	-0.0052 (14)	-0.0046 (15)
O24	0.024 (3)	0.021 (2)	0.0164 (13)	0.003 (3)	-0.0057 (12)	-0.0078 (13)
C25	0.0154 (19)	0.017 (2)	0.0115 (17)	0.000 (3)	-0.0043 (16)	-0.0001 (15)
C26	0.015 (3)	0.012 (2)	0.0172 (18)	-0.001 (3)	-0.0053 (17)	-0.0032 (16)
N27	0.0182 (16)	0.0090 (18)	0.0106 (14)	0.0009 (19)	-0.0040 (13)	-0.0014 (12)
C28	0.0159 (19)	0.017 (2)	0.0121 (18)	0.0032 (17)	-0.0020 (14)	-0.0060 (17)
C28A	0.014 (2)	0.0192 (18)	0.0096 (17)	0.0003 (14)	0.0009 (17)	-0.0049 (14)
C29	0.026 (3)	0.0171 (19)	0.015 (3)	-0.0041 (18)	0.001 (2)	-0.0049 (17)
C210	0.028 (3)	0.026 (3)	0.024 (2)	-0.010 (3)	-0.006 (2)	-0.009 (3)
C211	0.020 (2)	0.036 (3)	0.025 (2)	-0.007 (2)	-0.0062 (17)	-0.018 (3)
C212	0.013 (3)	0.027 (3)	0.018 (2)	0.0031 (19)	-0.002 (2)	-0.0055 (19)
C22A	0.015 (3)	0.0191 (18)	0.0097 (18)	-0.0027 (16)	0.0046 (19)	-0.0055 (15)
C213	0.020 (2)	0.0177 (19)	0.0114 (18)	0.0014 (17)	-0.0055 (17)	-0.0029 (14)
C23C	0.012 (3)	0.0152 (18)	0.0138 (17)	-0.0004 (15)	-0.0044 (15)	-0.0042 (14)
C231	0.026 (2)	0.018 (2)	0.017 (2)	-0.0053 (17)	-0.0030 (17)	-0.0037 (17)
C232	0.027 (4)	0.022 (2)	0.0172 (19)	0.000 (2)	-0.0055 (18)	-0.0029 (16)
C23B	0.008 (4)	0.0157 (19)	0.0180 (17)	-0.0012 (15)	-0.0036 (15)	-0.0046 (15)
C251	0.0148 (18)	0.021 (2)	0.0184 (19)	-0.004 (3)	-0.0043 (17)	-0.0037 (17)
O251	0.040 (5)	0.0151 (18)	0.0198 (15)	0.0079 (17)	-0.0102 (16)	-0.0028 (14)
O252	0.039 (4)	0.018 (2)	0.0124 (13)	-0.001 (3)	-0.0069 (15)	-0.0001 (13)
C252	0.030 (5)	0.021 (2)	0.0154 (18)	0.000 (3)	-0.008 (2)	0.0024 (16)
C253	0.039 (7)	0.030 (3)	0.0157 (19)	-0.016 (3)	-0.0037 (18)	0.0014 (18)

*Geometric parameters (Å, °)*

C11—C12	1.393 (5)	C21—C23C	1.401 (11)
C11—C13C	1.399 (5)	C21—C22	1.402 (10)
C11—H11	0.9500	C21—H21	0.9500
C12—C13	1.364 (5)	C22—C23	1.364 (10)
C12—Cl12	1.746 (4)	C22—Cl22	1.740 (10)
C13—C13A	1.405 (5)	C23—C23A	1.406 (11)
C13—H13	0.9500	C23—H23	0.9500
C13A—C13B	1.416 (5)	C23A—C23B	1.417 (9)
C13A—C14	1.489 (5)	C23A—C24	1.490 (10)
C14—O14	1.236 (4)	C24—O24	1.239 (10)
C14—C15	1.448 (5)	C24—C25	1.450 (11)
C15—C16	1.374 (5)	C25—C26	1.374 (10)
C15—C151	1.477 (5)	C25—C251	1.478 (10)
C16—N17	1.340 (5)	C26—N27	1.341 (9)
C16—H16	0.9500	C26—H26	0.9500
N17—C13B	1.403 (4)	N27—C23B	1.403 (10)

N17—C18	1.482 (4)	N27—C28	1.484 (10)
C18—C18A	1.500 (5)	C28—C28A	1.502 (10)
C18—H18A	0.9900	C28—H28A	0.9900
C18—H18B	0.9900	C28—H28B	0.9900
C18A—C19	1.399 (5)	C28A—C29	1.400 (11)
C18A—C12A	1.400 (5)	C28A—C22A	1.405 (9)
C19—C110	1.380 (5)	C29—C210	1.381 (10)
C19—H19	0.9500	C29—H29	0.9500
C110—C111	1.386 (6)	C210—C211	1.387 (11)
C110—H110	0.9500	C210—H210	0.9500
C111—C112	1.396 (6)	C211—C212	1.396 (11)
C111—H111	0.9500	C211—H211	0.9500
C112—C12A	1.389 (5)	C212—C22A	1.387 (10)
C112—H112	0.9500	C212—H212	0.9500
C12A—C113	1.519 (5)	C22A—C213	1.521 (10)
C113—C13C	1.531 (5)	C213—C23C	1.527 (9)
C113—C131	1.550 (5)	C213—C231	1.540 (10)
C113—H113	1.0000	C213—H223	1.0000
C13C—C13B	1.430 (5)	C23C—C23B	1.438 (9)
C131—C132	1.519 (5)	C231—C232	1.523 (11)
C131—H13A	0.9900	C231—H23A	0.9900
C131—H13B	0.9900	C231—H23B	0.9900
C132—H13C	0.9800	C232—H23C	0.9800
C132—H13D	0.9800	C232—H23D	0.9800
C132—H13E	0.9800	C232—H23E	0.9800
C151—O151	1.213 (5)	C251—O251	1.214 (11)
C151—O152	1.352 (4)	C251—O252	1.354 (11)
O152—C152	1.455 (6)	O252—C252	1.455 (12)
C152—C153	1.517 (5)	C252—C253	1.518 (11)
C152—H15A	0.9900	C252—H25A	0.9900
C152—H15B	0.9900	C252—H25B	0.9900
C153—H15C	0.9800	C253—H25C	0.9800
C153—H15D	0.9800	C253—H25D	0.9800
C153—H15E	0.9800	C253—H25E	0.9800
C12—C11—C13C	122.4 (3)	H15D—C153—H15E	109.5
C12—C11—H11	118.8	C23C—C21—C22	119.8 (13)
C13C—C11—H11	118.8	C23C—C21—H21	120.1
C13—C12—C11	121.0 (3)	C22—C21—H21	120.1
C13—C12—C112	120.6 (3)	C23—C22—C21	120.2 (12)
C11—C12—C112	118.4 (3)	C23—C22—C122	120.5 (12)
C12—C13—C13A	119.3 (3)	C21—C22—C122	119.3 (12)
C12—C13—H13	120.4	C22—C23—C23A	118.0 (14)
C13A—C13—H13	120.4	C22—C23—H23	121.0
C13—C13A—C13B	120.4 (3)	C23A—C23—H23	121.0
C13—C13A—C14	116.9 (3)	C23—C23A—C23B	119.3 (16)
C13B—C13A—C14	122.7 (3)	C23—C23A—C24	116.5 (14)
O14—C14—C15	126.0 (3)	C23B—C23A—C24	122.7 (10)

O14—C14—C13A	119.8 (3)	O24—C24—C25	125 (2)
C15—C14—C13A	114.2 (3)	O24—C24—C23A	118.9 (17)
C16—C15—C14	119.3 (3)	C25—C24—C23A	113.5 (13)
C16—C15—C151	114.5 (3)	C26—C25—C24	118.8 (16)
C14—C15—C151	126.0 (3)	C26—C25—C251	114.7 (14)
N17—C16—C15	124.8 (4)	C24—C25—C251	125.3 (18)
N17—C16—H16	117.6	N27—C26—C25	124.9 (13)
C15—C16—H16	117.6	N27—C26—H26	117.5
C16—N17—C13B	121.1 (3)	C25—C26—H26	117.5
C16—N17—C18	118.0 (3)	C26—N27—C23B	121.3 (11)
C13B—N17—C18	120.8 (3)	C26—N27—C28	117.1 (14)
N17—C18—C18A	110.7 (3)	C23B—N27—C28	120.8 (16)
N17—C18—H18A	109.5	N27—C28—C28A	110.5 (15)
C18A—C18—H18A	109.5	N27—C28—H28A	109.5
N17—C18—H18B	109.5	C28A—C28—H28A	109.5
C18A—C18—H18B	109.5	N27—C28—H28B	109.5
H18A—C18—H18B	108.1	C28A—C28—H28B	109.5
C19—C18A—C12A	120.6 (3)	H28A—C28—H28B	108.1
C19—C18A—C18	119.9 (3)	C29—C28A—C22A	120.2 (14)
C12A—C18A—C18	119.5 (3)	C29—C28A—C28	119.8 (15)
C110—C19—C18A	120.1 (4)	C22A—C28A—C28	118.2 (12)
C110—C19—H19	120.0	C210—C29—C28A	119.7 (15)
C18A—C19—H19	120.0	C210—C29—H29	120.2
C19—C110—C111	119.8 (4)	C28A—C29—H29	120.2
C19—C110—H110	120.1	C29—C210—C211	119.0 (15)
C111—C110—H110	120.1	C29—C210—H210	120.5
C110—C111—C112	120.3 (4)	C211—C210—H210	120.5
C110—C111—H111	119.8	C210—C211—C212	120.0 (14)
C112—C111—H111	119.8	C210—C211—H211	120.0
C12A—C112—C111	120.6 (4)	C212—C211—H211	120.0
C12A—C112—H112	119.7	C22A—C212—C211	120.6 (13)
C111—C112—H112	119.7	C22A—C212—H212	119.7
C112—C12A—C18A	118.6 (3)	C211—C212—H212	119.7
C112—C12A—C113	120.8 (3)	C212—C22A—C28A	118.3 (11)
C18A—C12A—C113	120.6 (3)	C212—C22A—C213	122.4 (13)
C12A—C113—C13C	116.4 (3)	C28A—C22A—C213	118.9 (11)
C12A—C113—C131	113.0 (3)	C22A—C213—C23C	115.5 (14)
C13C—C113—C131	112.9 (3)	C22A—C213—C231	113.8 (13)
C12A—C113—H113	104.3	C23C—C213—C231	117.1 (12)
C13C—C113—H113	104.3	C22A—C213—H223	102.5
C131—C113—H113	104.3	C23C—C213—H223	102.5
C11—C13C—C13B	116.6 (3)	C231—C213—H223	102.5
C11—C13C—C113	114.0 (3)	C21—C23C—C23B	115.6 (14)
C13B—C13C—C113	129.4 (3)	C21—C23C—C213	115.6 (12)
C132—C131—C113	113.4 (3)	C23B—C23C—C213	126.8 (11)
C132—C131—H13A	108.9	C232—C231—C213	112.8 (15)
C113—C131—H13A	108.9	C232—C231—H23A	109.0
C132—C131—H13B	108.9	C213—C231—H23A	109.0

C113—C131—H13B	108.9	C232—C231—H23B	109.0
H13A—C131—H13B	107.7	C213—C231—H23B	109.0
C131—C132—H13C	109.5	H23A—C231—H23B	107.8
C131—C132—H13D	109.5	C231—C232—H23C	109.5
H13C—C132—H13D	109.5	C231—C232—H23D	109.5
C131—C132—H13E	109.5	H23C—C232—H23D	109.5
H13C—C132—H13E	109.5	C231—C232—H23E	109.5
H13D—C132—H13E	109.5	H23C—C232—H23E	109.5
N17—C13B—C13A	116.7 (3)	H23D—C232—H23E	109.5
N17—C13B—C13C	123.2 (3)	N27—C23B—C23A	116.5 (11)
C13A—C13B—C13C	120.1 (3)	N27—C23B—C23C	123.4 (13)
O151—C151—O152	121.9 (4)	C23A—C23B—C23C	119.9 (10)
O151—C151—C15	124.7 (4)	O251—C251—O252	121 (2)
O152—C151—C15	113.3 (3)	O251—C251—C25	125 (2)
C151—O152—C152	115.2 (4)	O252—C251—C25	112.5 (15)
O152—C152—C153	105.8 (4)	C251—O252—C252	115.3 (17)
O152—C152—H15A	110.6	O252—C252—C253	105.7 (14)
C153—C152—H15A	110.6	O252—C252—H25A	110.6
O152—C152—H15B	110.6	C253—C252—H25A	110.6
C153—C152—H15B	110.6	H25A—C252—H25B	108.7
H15A—C152—H15B	108.7	C252—C253—H25C	109.5
C152—C153—H15C	109.5	C252—C253—H25D	109.5
C152—C153—H15D	109.5	H25C—C253—H25D	109.5
H15C—C153—H15D	109.5	C252—C253—H25E	109.5
C152—C153—H15E	109.5	H25D—C253—H25E	109.5
H15C—C153—H15E	109.5		
C13C—C11—C12—C13	0.6 (7)	C15—C151—O152—C152	179.9 (16)
C13C—C11—C12—C112	179.3 (5)	C151—O152—C152—C153	178.5 (16)
C11—C12—C13—C13A	-2.5 (8)	C23C—C21—C22—C23	28 (6)
C112—C12—C13—C13A	178.7 (6)	C23C—C21—C22—C122	-152 (6)
C12—C13—C13A—C13B	-0.3 (9)	C21—C22—C23—C23A	-24 (7)
C12—C13—C13A—C14	178.9 (6)	C122—C22—C23—C23A	155 (6)
C13—C13A—C14—O14	5.3 (11)	C22—C23—C23A—C23B	18 (8)
C13B—C13A—C14—O14	-175.6 (10)	C22—C23—C23A—C24	-175 (6)
C13—C13A—C14—C15	-176.8 (9)	C23—C23A—C24—O24	14 (11)
C13B—C13A—C14—C15	2.3 (9)	C23B—C23A—C24—O24	180 (9)
O14—C14—C15—C16	169.4 (12)	C23—C23A—C24—C25	-150 (8)
C13A—C14—C15—C16	-8.4 (13)	C23B—C23A—C24—C25	16 (8)
O14—C14—C15—C151	-7.4 (16)	O24—C24—C25—C26	-179 (12)
C13A—C14—C15—C151	174.8 (10)	C23A—C24—C25—C26	-17 (12)
C14—C15—C16—N17	5.4 (18)	O24—C24—C25—C251	14 (15)
C151—C15—C16—N17	-177.5 (11)	C23A—C24—C25—C251	176 (9)
C15—C16—N17—C13B	4.8 (16)	C251—C25—C26—N27	176 (11)
C15—C16—N17—C18	-172.5 (11)	C25—C26—N27—C23B	4 (16)
C16—N17—C18—C18A	-110.9 (9)	C25—C26—N27—C28	-166 (10)
C13B—N17—C18—C18A	71.9 (8)	C26—N27—C28—C28A	-116 (8)
N17—C18—C18A—C19	105.6 (7)	C23B—N27—C28—C28A	74 (6)

N17—C18—C18A—C12A	-72.5 (6)	N27—C28—C28A—C29	90 (7)
C12A—C18A—C19—C110	0.4 (10)	N27—C28—C28A—C22A	-74 (4)
C18—C18A—C19—C110	-177.7 (7)	C22A—C28A—C29—C210	-14 (10)
C18A—C19—C110—C111	-3.0 (11)	C28—C28A—C29—C210	-178 (7)
C19—C110—C111—C112	3.8 (11)	C28A—C29—C210—C211	16 (12)
C110—C111—C112—C12A	-2.0 (9)	C29—C210—C211—C212	-14 (12)
C111—C112—C12A—C18A	-0.7 (7)	C210—C211—C212—C22A	9 (10)
C111—C112—C12A—C113	179.4 (5)	C211—C212—C22A—C28A	-7 (8)
C19—C18A—C12A—C112	1.5 (8)	C211—C212—C22A—C213	166 (5)
C18—C18A—C12A—C112	179.6 (5)	C29—C28A—C22A—C212	9 (8)
C19—C18A—C12A—C113	-178.6 (5)	C28—C28A—C22A—C212	174 (5)
C18—C18A—C12A—C113	-0.5 (8)	C29—C28A—C22A—C213	-164 (5)
C112—C12A—C113—C13C	-126.3 (4)	C28—C28A—C22A—C213	1 (7)
C18A—C12A—C113—C13C	53.8 (6)	C212—C22A—C213—C23C	-112 (5)
C112—C12A—C113—C131	100.6 (4)	C28A—C22A—C213—C23C	60 (4)
C18A—C12A—C113—C131	-79.3 (6)	C212—C22A—C213—C231	27 (5)
C12—C11—C13C—C13B	4.0 (7)	C28A—C22A—C213—C231	-160 (4)
C12—C11—C13C—C113	-173.9 (4)	C22—C21—C23C—C23B	-23 (6)
C12A—C113—C13C—C11	146.8 (4)	C22—C21—C23C—C213	171 (3)
C131—C113—C13C—C11	-80.1 (5)	C22A—C213—C23C—C21	116 (4)
C12A—C113—C13C—C13B	-30.8 (7)	C231—C213—C23C—C21	-22 (5)
C131—C113—C13C—C13B	102.3 (6)	C22A—C213—C23C—C23B	-47 (6)
C12A—C113—C131—C132	-69.4 (5)	C231—C213—C23C—C23B	175 (5)
C13C—C113—C131—C132	155.8 (4)	C22A—C213—C231—C232	42 (5)
C16—N17—C13B—C13A	-10.6 (12)	C23C—C213—C231—C232	-179 (4)
C18—N17—C13B—C13A	166.5 (7)	C26—N27—C23B—C23A	-5 (11)
C16—N17—C13B—C13C	170.4 (10)	C28—N27—C23B—C23A	165 (6)
C18—N17—C13B—C13C	-12.4 (11)	C26—N27—C23B—C23C	170 (10)
C13—C13A—C13B—N17	-174.0 (8)	C28—N27—C23B—C23C	-20 (11)
C14—C13A—C13B—N17	6.9 (10)	C23—C23A—C23B—N27	160 (7)
C13—C13A—C13B—C13C	5.0 (10)	C24—C23A—C23B—N27	-6 (10)
C14—C13A—C13B—C13C	-174.1 (7)	C23—C23A—C23B—C23C	-15 (9)
C11—C13C—C13B—N17	172.2 (7)	C24—C23A—C23B—C23C	179 (7)
C113—C13C—C13B—N17	-10.2 (10)	C21—C23C—C23B—N27	-157 (7)
C11—C13C—C13B—C13A	-6.7 (9)	C213—C23C—C23B—N27	6 (10)
C113—C13C—C13B—C13A	170.9 (6)	C21—C23C—C23B—C23A	18 (8)
C16—C15—C151—O151	4.8 (16)	C213—C23C—C23B—C23A	-179 (5)
C14—C15—C151—O151	-178.3 (15)	C24—C25—C251—O251	157 (14)
C16—C15—C151—O152	-177.8 (16)	C26—C25—C251—O252	-177 (15)
C14—C15—C151—O152	-0.9 (16)	C25—C251—O252—C252	170 (16)
O151—C151—O152—C152	-3 (2)	C251—O252—C252—C253	171 (16)

Hydrogen-bond geometry ( $\text{\AA}$ ,  $^\circ$ )

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C18—H18B $\cdots$ O151 <sup>i</sup>	0.99	2.54	3.483 (9)	159
C19—H19 $\cdots$ O151 <sup>i</sup>	0.95	2.54	3.396 (12)	149

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C28—H28B···O251 <sup>i</sup>	0.99	2.10	3.13 (7)	159
C29—H29···O251 <sup>i</sup>	0.95	2.75	3.38 (13)	124

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