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2,3-Dimethoxy-10-oxostrychnidinium 2-carboxy-4,5-dichlorobenzoate

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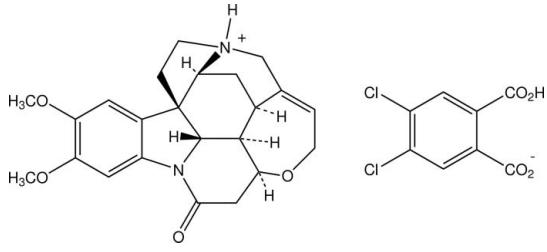
Key indicators: single-crystal X-ray study; T = 130 K; mean σ(C–C) = 0.003 Å; R factor = 0.036; wR factor = 0.087; data-to-parameter ratio = 12.4.

The structure of the title compound, $C_{23}H_{27}N_2O_4^+\cdot C_8H_3Cl_2O_4^-$, a 1:1 proton-transfer compound of brucine with 4,5-dichlorophthalic acid, has been determined at 130 K. The brucinium cations and the hydrogen phthalate anions associate through single N—H···$O_{carboxylate}$ hydrogen bonds [2.639 (3) Å], giving dimers which are extended *via* weak head-to-tail C—H···$O_{methoxy}$ associations into chains forming down the 2_1 screw axis of the unit cell. The carboxyl proton of the anion gives a short intramolecular O—H···$O_{carboxylate}$ hydrogen bond [2.441 (3) Å].

Related literature

Absolute configuration: (Peerdeman, 1956; Flack, 1983). Similar structures: (Oshikawa *et al.*, 2002; Smith *et al.*, 2005, 2006*a*,*b*; Bialońska & Ciunik, 2004*a*,*b*, 2006; Gould & Walkinshaw, 1984; Mallinson *et al.*, 2003; Bozkurt *et al.*, 2006).



Experimental

Crystal data

$C_{23}H_{27}N_2O_4^+\cdot C_8H_3Cl_2O_4^-$
M_r = 629.47
Monoclinic, $P2_1$
a = 9.5085 (10) Å
b = 12.9946 (13) Å
c = 11.5893 (12) Å
β = 104.110 (2)°
V = 1388.8 (2) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.29 mm^{-1}
T = 130 (2) K
0.45 × 0.20 × 0.15 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 1999)
T_{min} = 0.86, T_{max} = 0.96
8761 measured reflections
4923 independent reflections
4747 reflections with $I > 2\sigma(I)$
R_{int} = 0.024

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.036
$wR(F^2)$ = 0.087
S = 1.04
4923 reflections
396 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.28 e Å^{-3}
$\Delta\rho_{min}$ = −0.27 e Å^{-3}
Absolute structure: Flack (1983), 1604 Friedel pairs
Flack parameter: 0.07 (5)

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

D—H···A	D—H	H···A	D···A	D—H···A
N19—H19···O11*A*	0.90 (3)	1.75 (3)	2.639 (3)	170 (2)
O21*A*—H21*A*···O12*A*	0.84 (5)	1.61 (5)	2.441 (3)	170 (4)
C3*A*—H3*A*···O22*A*	0.93	2.31	2.667 (3)	103
C4—H4···O25	0.93	2.45	2.953 (3)	114
C6*A*—H6*A*···O11*A*	0.93	2.26	2.635 (3)	103
C16—H16···O12*A*	0.98	2.55	3.402 (3)	145
C17—H17*B*···O2^{i}	0.97	2.53	3.441 (3)	156
C18—H18*B*···O3^{i}	0.97	2.48	3.279 (3)	140
C22—H22···O22*A*^{ii}	0.93	2.45	3.316 (3)	155

Symmetry codes: (i) $-x+3, y+\frac{1}{2}, -z+1$; (ii) $x, y+1, z$.

Data collection: *SMART* (Bruker, 2000); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 1999); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *PLATON*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: FL2168).

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supplementary materials

Acta Cryst. (2007). E**63**, o4276-o4277 [**doi:10.1107/S1600536807047952**]

2,3-Dimethoxy-10-oxostrychnidinium 2-carboxy-4,5-dichlorobenzoate

G. Smith, U. D. Wermuth and J. M. White

Comment

Brucine (2,3-dimethoxystrychnidin-10-one) has shown a hit-or-miss ability to form crystalline salts with both chiral and achiral aromatic carboxylic acids. However, Oshikawa *et al.* (2002) observed the selectivity of brucine for *meta*-substituted benzoic acids and reported the structure of brucinium 3-nitrobenzoate. Since then, the brucinium salts of a number of other *meta*-substituted benzoic acids have been characterized, including 3,5-dinitrobenzoic acid (three solvent pseudopolymorphs) (Bialońska & Ciunik, 2006), 3-nitrophthalic acid (Smith *et al.*, 2005), 5-nitrosalicylic, 3,5-dinitrosalicylic and 5-sulfosalicylic acids (Smith, *et al.*, 2006*a*), and isophthalic acid (Smith,*et al.*, 2006*b*). We also obtained good crystals of the title compound from the 1:1 stoichiometric reaction of 4,5-dichlorophthalic acid with brucine in methanol and its structure is reported here.

In (I), protonation occurs as expected at N19 of the brucine cage (Fig. 1), the invoked Peerdeman (1956) absolute configuration giving the overall Cahn-Ingold-Prelog stereochemistry of the cation molecule as C7(S), C8(S), C12(S), C13(*R*), C14(*R*), C16(S), N19(S). The cations and anions give a single *N*–*H*···$O_{carboxylate}$ hydrogen-bonding association resulting in dimers which are extended *via* weak head-to-tail *C*–*H*···$O_{methoxy}$ associations (Table 1) into chains which form down the 2_1 screw axis of the unit cell (Fig. 2). The brucinium cations give a variant of the previously described undulating sheet host substructure which is present in a significant number of brucine compounds (Gould & Walkinshaw, 1984; Bialońska & Ciunik, 2004*a*, 2004*b*; Smith,*et al.*, 2006*a*).

The hydrogen 4,5-dichlorophthalate anion is essentially planar [torsion angles C2A–C1A–C11A–O11A, −177.8 (2) °: C1A–C2A–C21A–O22A, −163.9 (2) °], having a strong intramolecular hydrogen bond [2.441 (3) Å] between the carboxylic acid and the *ortho*-related carboxylate group. This is similar to that found in other acid salts of 4,5-dichlorophthalic acid (Mallinson *et al.*, 2003; Bozkurt *et al.*, 2006).

Experimental

The title compound (I) was synthesized by heating 1 mmol quantities of brucine tetrahydrate and 4,5-dichlorophthalic acid in 50 ml of methanol for 10 min under reflux. After concentration to *ca*. 30 ml, partial room-temperature evaporation of the hot-filtered solution gave colourless prisms (m.p. 541–543 K).

Refinement

Hydrogen atoms potentially involved in hydrogen-bonding interactions were located by difference methods and their positional and isotropic displacement parameters were refined. Other H atoms were included in the refinement at calculated positions [C—H(aromatic) = 0.93 Å; C—H(aliphatic), 0.96–0.98 Å] and treated as riding models with U_{iso}(H) = 1.2U_{eq} (C). The absolute configuration determined for the parent strychnidin-10-one molecule (Peerdeman, 1956) was invoked and it agreed with that indicated byt the Flack parameter (Flack, 1983).

Figures

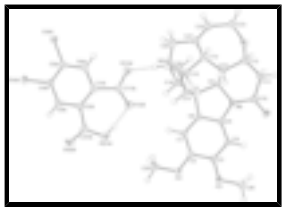

Fig. 1. Molecular configuration and atom numbering scheme for the brucinium cation and the hydrogen 4,5-dichlorophthalate anion in (I). Non-H atoms are shown as 50% probability displacement ellipsoids. Hydrogen bonds are shown as dashed lines.

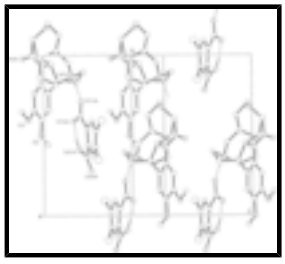

Fig. 2. The packing of the dimer units in (I) in the unit cell viewed perpendicular to the *b* axial direction. Non-interactive H atoms are omitted.

2,3-Dimethoxy-10-oxostrychnidinium 2-carboxy-4,5-dichlorobenzoate

Crystal data

$C_{23}H_{27}N_2O_4^+ \cdot C_8H_3Cl_2O_4^-$	F_{000} = 656
M_r = 629.47	D_x = 1.505 Mg m^{-3}
Monoclinic, $P2_1$	Melting point: 541–543 K
Hall symbol: P 2yb	Mo $K\alpha$ radiation λ = 0.71073 Å
a = 9.5085 (10) Å	Cell parameters from 3573 reflections
b = 12.9946 (13) Å	θ = 2.4–27.5º
c = 11.5893 (12) Å	μ = 0.29 mm^{-1}
β = 104.110 (2)º	T = 130 (2) K
V = 1388.8 (2) Å^3	Prism, colourless
Z = 2	0.45 × 0.20 × 0.15 mm

Data collection

Bruker SMART CCD area-detector diffractometer	4923 independent reflections
Radiation source: sealed tube	4747 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.024
T = 130(2) K	θ_{max} = 27.5º
π and ω scans	θ_{min} = 1.8º
Absorption correction: multi-scan (SADABS; Bruker, 1999)	h = −11→12
T_{min} = 0.86, T_{max} = 0.96	k = −13→16
8761 measured reflections	l = −14→14

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H atoms treated by a mixture of independent and constrained refinement
$R[F^2 > 2\sigma(F^2)] = 0.036$	$w = 1/[\sigma^2(F_o^2) + (0.0482P)^2 + 0.1824P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.087$	$(\Delta/\sigma)_{max} < 0.001$
$S = 1.04$	$\Delta\rho_{max} = 0.28$ e Å^{-3}
4923 reflections	$\Delta\rho_{min} = -0.27$ e Å^{-3}
396 parameters	Extinction correction: none
1 restraint	Absolute structure: Flack (1983)
Primary atom site location: structure-invariant direct methods	Flack parameter: 0.07 (5)
Secondary atom site location: difference Fourier map	

Special details

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

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit S are based on F^2, conventional *R*-factors *R* are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2$sigma(F^2) is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on F, and R– factors based on ALL data will be even larger.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
O2	1.54757 (16)	0.49720 (12)	0.37798 (14)	0.0206 (5)
O3	1.79347 (15)	0.59526 (13)	0.45159 (14)	0.0207 (5)
O24	1.32796 (17)	1.17615 (14)	0.26352 (15)	0.0245 (5)
O25	1.75444 (16)	0.97830 (15)	0.38024 (15)	0.0270 (5)
N9	1.52499 (19)	0.92448 (15)	0.37457 (16)	0.0169 (5)
N19	1.03164 (19)	0.86493 (16)	0.25567 (17)	0.0185 (6)
C1	1.4104 (2)	0.65792 (19)	0.34534 (19)	0.0173 (6)
C2	1.5393 (2)	0.60267 (19)	0.37881 (18)	0.0168 (6)
C3	1.6727 (2)	0.65612 (18)	0.41809 (18)	0.0163 (6)
C4	1.6775 (2)	0.76311 (19)	0.41932 (19)	0.0177 (7)
C5	1.5471 (2)	0.81670 (18)	0.38124 (19)	0.0169 (6)
C6	1.4158 (2)	0.76555 (18)	0.34721 (18)	0.0155 (6)
C7	1.2924 (2)	0.84016 (19)	0.33033 (18)	0.0160 (6)
C8	1.3679 (2)	0.94622 (17)	0.32578 (19)	0.0151 (6)
C10	1.6246 (2)	0.99634 (18)	0.3600 (2)	0.0196 (7)
C11	1.5609 (2)	1.10200 (19)	0.3217 (2)	0.0211 (7)

C12	1.4182 (2)	1.10386 (19)	0.2230 (2)	0.0194 (7)
C13	1.3554 (2)	0.99410 (17)	0.20308 (19)	0.0169 (6)
C14	1.2047 (2)	0.9798 (2)	0.11891 (19)	0.0197 (7)
C15	1.1814 (2)	0.86306 (19)	0.1049 (2)	0.0207 (7)
C16	1.1650 (2)	0.81878 (18)	0.2216 (2)	0.0177 (6)
C17	1.2167 (2)	0.83530 (19)	0.43341 (19)	0.0176 (6)
C18	1.0774 (2)	0.89449 (19)	0.38545 (19)	0.0187 (6)
C20	0.9651 (2)	0.95409 (19)	0.1787 (2)	0.0223 (7)
C21	1.0808 (2)	1.0274 (2)	0.1633 (2)	0.0205 (7)
C22	1.0756 (3)	1.1263 (2)	0.1875 (2)	0.0236 (7)
C23	1.1939 (3)	1.2006 (2)	0.1784 (2)	0.0282 (8)
C25	1.4138 (2)	0.4424 (2)	0.3378 (2)	0.0218 (7)
C26	1.9270 (2)	0.6490 (2)	0.5014 (2)	0.0236 (7)
Cl4A	0.37189 (6)	0.34813 (5)	−0.00859 (6)	0.0289 (2)
Cl5A	0.34137 (6)	0.59206 (5)	−0.00357 (5)	0.0273 (2)
O11A	0.84305 (18)	0.71344 (14)	0.19623 (16)	0.0284 (6)
O12A	1.00497 (17)	0.58983 (16)	0.25332 (17)	0.0318 (6)
O21A	1.0085 (2)	0.40751 (18)	0.3045 (2)	0.0428 (7)
O22A	0.8526 (2)	0.28055 (15)	0.28601 (18)	0.0345 (6)
C1A	0.7527 (2)	0.54385 (19)	0.17814 (19)	0.0185 (6)
C2A	0.7584 (2)	0.43600 (19)	0.1910 (2)	0.0191 (7)
C3A	0.6375 (2)	0.37818 (18)	0.1349 (2)	0.0192 (7)
C4A	0.5121 (2)	0.42411 (19)	0.0697 (2)	0.0189 (7)
C5A	0.5011 (2)	0.5306 (2)	0.0667 (2)	0.0179 (6)
C6A	0.6206 (2)	0.58916 (19)	0.12083 (19)	0.0177 (6)
C11A	0.8761 (2)	0.6212 (2)	0.2133 (2)	0.0222 (7)
C21A	0.8802 (3)	0.3690 (2)	0.2654 (2)	0.0261 (8)
H1	1.32200	0.62380	0.32210	0.0210*
H4	1.76500	0.79800	0.44480	0.0210*
H8	1.33450	0.99550	0.37730	0.0180*
H11A	1.63290	1.14170	0.29450	0.0250*
H11B	1.54380	1.13660	0.39120	0.0250*
H12	1.43830	1.12910	0.14900	0.0230*
H13	1.42240	0.95520	0.16740	0.0200*
H14	1.20490	1.00960	0.04130	0.0240*
H15A	1.09510	0.84890	0.04250	0.0250*
H15B	1.26360	0.83160	0.08280	0.0250*
H16	1.15170	0.74420	0.21260	0.0210*
H17A	1.19680	0.76470	0.45160	0.0210*
H17B	1.27520	0.86770	0.50440	0.0210*
H18A	1.00410	0.87500	0.42660	0.0220*
H18B	1.09410	0.96800	0.39430	0.0220*
H19	0.966 (3)	0.814 (2)	0.244 (2)	0.015 (6)*
H20A	0.91220	0.92890	0.10150	0.0270*
H20B	0.89720	0.98950	0.21520	0.0270*
H22	0.99540	1.15120	0.21120	0.0280*
H23A	1.20910	1.19800	0.09870	0.0340*
H23B	1.16440	1.26990	0.19260	0.0340*
H25A	1.43260	0.36980	0.34120	0.0330*

H25B	1.36890	0.46190	0.25740	0.0330*
H25C	1.35020	0.45890	0.38800	0.0330*
H26A	2.00510	0.60040	0.52210	0.0350*
H26B	1.91870	0.68570	0.57130	0.0350*
H26C	1.94620	0.69680	0.44390	0.0350*
H3A	0.64130	0.30690	0.14160	0.0230*
H6A	0.61280	0.66050	0.11910	0.0210*
H21A	1.015 (4)	0.469 (4)	0.283 (3)	0.063 (12)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O2	0.0162 (7)	0.0148 (9)	0.0297 (9)	0.0011 (6)	0.0036 (6)	−0.0004 (7)
O3	0.0136 (7)	0.0170 (9)	0.0295 (8)	0.0008 (7)	0.0017 (6)	−0.0017 (7)
O24	0.0223 (8)	0.0174 (9)	0.0337 (9)	−0.0020 (7)	0.0069 (7)	0.0016 (7)
O25	0.0138 (8)	0.0241 (10)	0.0428 (9)	0.0025 (7)	0.0063 (6)	−0.0014 (9)
N9	0.0147 (9)	0.0170 (10)	0.0195 (9)	0.0005 (7)	0.0049 (7)	0.0006 (8)
N19	0.0119 (9)	0.0186 (11)	0.0236 (10)	0.0010 (8)	0.0017 (7)	−0.0007 (8)
C1	0.0135 (10)	0.0191 (12)	0.0188 (11)	0.0038 (8)	0.0031 (8)	0.0019 (9)
C2	0.0187 (10)	0.0168 (12)	0.0155 (10)	0.0003 (9)	0.0055 (8)	−0.0013 (9)
C3	0.0163 (10)	0.0172 (12)	0.0153 (10)	−0.0009 (8)	0.0037 (8)	−0.0021 (9)
C4	0.0123 (10)	0.0211 (13)	0.0196 (11)	0.0025 (9)	0.0037 (8)	−0.0016 (10)
C5	0.0177 (10)	0.0172 (12)	0.0171 (11)	0.0015 (8)	0.0070 (8)	0.0006 (9)
C6	0.0133 (10)	0.0180 (12)	0.0146 (10)	0.0004 (8)	0.0020 (8)	−0.0005 (9)
C7	0.0152 (10)	0.0144 (11)	0.0177 (10)	0.0019 (9)	0.0029 (8)	−0.0007 (9)
C8	0.0117 (10)	0.0150 (12)	0.0184 (10)	0.0004 (8)	0.0031 (7)	0.0007 (8)
C10	0.0211 (11)	0.0187 (13)	0.0204 (11)	0.0023 (9)	0.0078 (8)	0.0031 (9)
C11	0.0201 (11)	0.0167 (13)	0.0274 (12)	0.0069 (9)	0.0073 (9)	0.0012 (10)
C12	0.0209 (11)	0.0178 (12)	0.0214 (11)	0.0010 (9)	0.0086 (8)	−0.0030 (10)
C13	0.0162 (10)	0.0140 (12)	0.0212 (11)	−0.0004 (8)	0.0060 (8)	0.0009 (9)
C14	0.0204 (11)	0.0224 (13)	0.0158 (10)	0.0030 (10)	0.0032 (8)	−0.0022 (10)
C15	0.0199 (11)	0.0221 (13)	0.0180 (10)	0.0010 (9)	0.0007 (8)	0.0038 (10)
C16	0.0145 (10)	0.0144 (12)	0.0232 (11)	0.0011 (8)	0.0028 (8)	0.0036 (9)
C17	0.0159 (10)	0.0170 (12)	0.0209 (11)	0.0010 (9)	0.0064 (8)	−0.0019 (9)
C18	0.0156 (10)	0.0179 (12)	0.0234 (11)	0.0019 (9)	0.0063 (8)	0.0009 (9)
C20	0.0177 (11)	0.0214 (14)	0.0255 (11)	−0.0028 (9)	0.0008 (8)	−0.0012 (10)
C21	0.0178 (11)	0.0224 (13)	0.0191 (11)	−0.0024 (9)	0.0002 (8)	−0.0034 (9)
C22	0.0201 (11)	0.0260 (13)	0.0242 (12)	−0.0040 (10)	0.0045 (9)	−0.0041 (10)
C23	0.0252 (12)	0.0208 (14)	0.0380 (14)	−0.0037 (10)	0.0063 (10)	−0.0037 (12)
C25	0.0212 (11)	0.0188 (13)	0.0253 (12)	0.0050 (9)	0.0054 (9)	0.0006 (9)
C26	0.0157 (11)	0.0228 (13)	0.0300 (13)	0.0009 (9)	0.0011 (9)	−0.0029 (11)
Cl4A	0.0224 (3)	0.0194 (3)	0.0403 (4)	−0.0056 (2)	−0.0011 (2)	−0.0055 (3)
Cl5A	0.0187 (3)	0.0217 (3)	0.0365 (3)	0.0013 (2)	−0.0030 (2)	0.0022 (3)
O11A	0.0234 (9)	0.0205 (10)	0.0379 (10)	−0.0064 (7)	0.0010 (7)	−0.0012 (8)
O12A	0.0167 (8)	0.0300 (11)	0.0460 (11)	−0.0033 (8)	0.0022 (7)	−0.0011 (10)
O21A	0.0235 (10)	0.0288 (13)	0.0670 (15)	0.0038 (9)	−0.0066 (9)	0.0050 (11)
O22A	0.0332 (10)	0.0233 (11)	0.0454 (11)	0.0070 (8)	0.0064 (8)	0.0099 (9)
C1A	0.0196 (11)	0.0202 (12)	0.0164 (10)	−0.0012 (9)	0.0059 (8)	−0.0025 (9)

C2A	0.0179 (11)	0.0213 (13)	0.0189 (11)	0.0020 (9)	0.0063 (8)	0.0011 (9)
C3A	0.0241 (11)	0.0119 (12)	0.0230 (11)	0.0020 (9)	0.0085 (9)	0.0013 (9)
C4A	0.0175 (11)	0.0159 (12)	0.0227 (11)	−0.0053 (9)	0.0040 (8)	−0.0037 (9)
C5A	0.0164 (10)	0.0173 (12)	0.0192 (11)	0.0012 (9)	0.0029 (8)	0.0015 (9)
C6A	0.0185 (10)	0.0142 (11)	0.0201 (10)	−0.0008 (9)	0.0043 (8)	0.0008 (9)
C11A	0.0184 (11)	0.0252 (14)	0.0231 (12)	−0.0039 (9)	0.0053 (9)	−0.0021 (10)
C21A	0.0226 (12)	0.0265 (15)	0.0282 (13)	0.0069 (10)	0.0043 (9)	0.0006 (11)

Geometric parameters (Å, °)

Cl4A—C4A	1.728 (2)	C20—C21	1.499 (3)
Cl5A—C5A	1.733 (2)	C21—C22	1.319 (4)
O2—C2	1.373 (3)	C22—C23	1.506 (4)
O2—C25	1.433 (3)	C1—H1	0.9300
O3—C3	1.370 (3)	C4—H4	0.9300
O3—C26	1.441 (3)	C8—H8	0.9800
O24—C23	1.444 (3)	C11—H11A	0.9700
O24—C12	1.427 (3)	C11—H11B	0.9700
O25—C10	1.222 (3)	C12—H12	0.9800
O11A—C11A	1.243 (3)	C13—H13	0.9800
O12A—C11A	1.267 (3)	C14—H14	0.9800
O21A—C21A	1.294 (3)	C15—H15A	0.9700
O22A—C21A	1.216 (3)	C15—H15B	0.9700
O21A—H21A	0.84 (5)	C16—H16	0.9800
N9—C8	1.490 (3)	C17—H17B	0.9700
N9—C10	1.370 (3)	C17—H17A	0.9700
N9—C5	1.416 (3)	C18—H18A	0.9700
N19—C18	1.510 (3)	C18—H18B	0.9700
N19—C20	1.504 (3)	C20—H20B	0.9700
N19—C16	1.539 (3)	C20—H20A	0.9700
N19—H19	0.90 (3)	C22—H22	0.9300
C1—C6	1.400 (3)	C23—H23A	0.9700
C1—C2	1.392 (3)	C23—H23B	0.9700
C2—C3	1.420 (3)	C25—H25A	0.9600
C3—C4	1.391 (3)	C25—H25B	0.9600
C4—C5	1.397 (3)	C25—H25C	0.9600
C5—C6	1.384 (3)	C26—H26A	0.9600
C6—C7	1.498 (3)	C26—H26B	0.9600
C7—C8	1.561 (3)	C26—H26C	0.9600
C7—C17	1.539 (3)	C1A—C2A	1.409 (3)
C7—C16	1.545 (3)	C1A—C11A	1.523 (3)
C8—C13	1.530 (3)	C1A—C6A	1.400 (3)
C10—C11	1.523 (3)	C2A—C3A	1.394 (3)
C11—C12	1.547 (3)	C2A—C21A	1.535 (3)
C12—C13	1.542 (3)	C3A—C4A	1.382 (3)
C13—C14	1.536 (3)	C4A—C5A	1.388 (4)
C14—C15	1.536 (4)	C5A—C6A	1.384 (3)
C14—C21	1.527 (3)	C3A—H3A	0.9300
C15—C16	1.512 (3)	C6A—H6A	0.9300

C17—C18	1.515 (3)		
C2—O2—C25	116.78 (17)	C13—C12—H12	109.00
C3—O3—C26	115.44 (18)	C11—C12—H12	109.00
C12—O24—C23	114.93 (18)	C8—C13—H13	106.00
C21A—O21A—H21A	113 (3)	C12—C13—H13	106.00
C5—N9—C10	125.69 (18)	C14—C13—H13	106.00
C8—N9—C10	118.92 (18)	C13—C14—H14	109.00
C5—N9—C8	109.28 (17)	C15—C14—H14	109.00
C16—N19—C20	113.69 (17)	C21—C14—H14	109.00
C18—N19—C20	111.85 (18)	C14—C15—H15B	110.00
C16—N19—C18	107.42 (16)	C16—C15—H15A	110.00
C20—N19—H19	107.6 (16)	C16—C15—H15B	110.00
C16—N19—H19	105.1 (18)	H15A—C15—H15B	108.00
C18—N19—H19	111.0 (15)	C14—C15—H15A	110.00
C2—C1—C6	119.0 (2)	N19—C16—H16	109.00
C1—C2—C3	119.6 (2)	C15—C16—H16	109.00
O2—C2—C1	124.14 (19)	C7—C16—H16	109.00
O2—C2—C3	116.25 (18)	C7—C17—H17A	111.00
C2—C3—C4	121.10 (19)	C18—C17—H17A	111.00
O3—C3—C4	123.42 (18)	C18—C17—H17B	111.00
O3—C3—C2	115.5 (2)	H17A—C17—H17B	109.00
C3—C4—C5	118.09 (19)	C7—C17—H17B	111.00
N9—C5—C6	110.36 (18)	N19—C18—H18B	111.00
C4—C5—C6	121.4 (2)	C17—C18—H18A	111.00
N9—C5—C4	128.3 (2)	N19—C18—H18A	111.00
C5—C6—C7	110.5 (2)	H18A—C18—H18B	109.00
C1—C6—C7	128.34 (19)	C17—C18—H18B	111.00
C1—C6—C5	120.76 (19)	N19—C20—H20B	110.00
C6—C7—C8	102.94 (16)	C21—C20—H20A	110.00
C6—C7—C16	115.06 (19)	N19—C20—H20A	110.00
C16—C7—C17	101.75 (16)	H20A—C20—H20B	108.00
C8—C7—C17	111.62 (18)	C21—C20—H20B	110.00
C6—C7—C17	111.83 (18)	C21—C22—H22	119.00
C8—C7—C16	114.00 (18)	C23—C22—H22	119.00
N9—C8—C13	105.87 (16)	O24—C23—H23B	109.00
C7—C8—C13	117.28 (17)	C22—C23—H23A	109.00
N9—C8—C7	104.28 (17)	O24—C23—H23A	109.00
O25—C10—N9	122.8 (2)	H23A—C23—H23B	108.00
O25—C10—C11	122.7 (2)	C22—C23—H23B	109.00
N9—C10—C11	114.39 (17)	O2—C25—H25A	109.00
C10—C11—C12	116.39 (19)	O2—C25—H25C	109.00
C11—C12—C13	109.52 (19)	H25A—C25—H25B	110.00
O24—C12—C13	114.60 (17)	H25A—C25—H25C	109.00
O24—C12—C11	104.85 (18)	H25B—C25—H25C	109.00
C8—C13—C12	107.31 (17)	O2—C25—H25B	110.00
C12—C13—C14	118.33 (19)	O3—C26—H26A	110.00
C8—C13—C14	112.97 (17)	O3—C26—H26C	109.00
C15—C14—C21	109.42 (18)	H26A—C26—H26B	110.00
C13—C14—C15	105.96 (18)	O3—C26—H26B	109.00

C13—C14—C21	114.47 (18)	H26B—C26—H26C	109.00
C14—C15—C16	108.96 (18)	H26A—C26—H26C	109.00
N19—C16—C15	110.63 (18)	C2A—C1A—C6A	118.4 (2)
C7—C16—C15	115.44 (17)	C2A—C1A—C11A	128.38 (19)
N19—C16—C7	104.59 (17)	C6A—C1A—C11A	113.1 (2)
C7—C17—C18	103.11 (17)	C1A—C2A—C3A	118.7 (2)
N19—C18—C17	104.48 (17)	C1A—C2A—C21A	128.9 (2)
N19—C20—C21	110.21 (17)	C3A—C2A—C21A	112.3 (2)
C14—C21—C22	122.6 (2)	C2A—C3A—C4A	121.7 (2)
C20—C21—C22	121.8 (2)	Cl4A—C4A—C5A	120.86 (16)
C14—C21—C20	115.6 (2)	C3A—C4A—C5A	119.6 (2)
C21—C22—C23	122.9 (2)	Cl4A—C4A—C3A	119.52 (19)
O24—C23—C22	111.2 (2)	Cl5A—C5A—C6A	119.21 (19)
C2—C1—H1	120.00	C4A—C5A—C6A	119.2 (2)
C6—C1—H1	121.00	Cl5A—C5A—C4A	121.56 (17)
C5—C4—H4	121.00	C1A—C6A—C5A	121.8 (2)
C3—C4—H4	121.00	O11A—C11A—C1A	116.46 (19)
N9—C8—H8	110.00	O12A—C11A—C1A	119.9 (2)
C13—C8—H8	110.00	O11A—C11A—O12A	123.6 (2)
C7—C8—H8	110.00	O21A—C21A—C2A	119.7 (2)
C10—C11—H11B	108.00	O22A—C21A—C2A	118.5 (2)
C12—C11—H11A	108.00	O21A—C21A—O22A	121.8 (3)
C10—C11—H11A	108.00	C2A—C3A—H3A	119.00
H11A—C11—H11B	107.00	C4A—C3A—H3A	119.00
C12—C11—H11B	108.00	C1A—C6A—H6A	119.00
O24—C12—H12	109.00	C5A—C6A—H6A	119.00
C25—O2—C2—C1	1.1 (3)	C6—C7—C8—C13	100.84 (19)
C25—O2—C2—C3	−179.61 (18)	C6—C7—C8—N9	−15.8 (2)
C26—O3—C3—C2	−174.67 (17)	C17—C7—C16—N19	31.8 (2)
C26—O3—C3—C4	6.6 (3)	C17—C7—C16—C15	153.6 (2)
C23—O24—C12—C13	−67.2 (2)	C8—C7—C16—C15	33.3 (3)
C23—O24—C12—C11	172.68 (19)	C7—C8—C13—C14	39.7 (3)
C12—O24—C23—C22	87.8 (2)	N9—C8—C13—C14	155.46 (18)
C5—N9—C10—C11	163.85 (19)	C7—C8—C13—C12	171.88 (17)
C5—N9—C10—O25	−18.7 (3)	N9—C8—C13—C12	−72.3 (2)
C10—N9—C5—C6	−155.4 (2)	N9—C10—C11—C12	−43.7 (3)
C8—N9—C5—C6	−3.5 (2)	O25—C10—C11—C12	138.9 (2)
C10—N9—C5—C4	26.7 (3)	C10—C11—C12—C13	11.6 (2)
C8—N9—C10—O25	−168.2 (2)	C10—C11—C12—O24	135.00 (19)
C8—N9—C5—C4	178.6 (2)	C11—C12—C13—C14	173.12 (17)
C5—N9—C8—C7	12.4 (2)	C11—C12—C13—C8	43.9 (2)
C10—N9—C8—C13	42.1 (2)	O24—C12—C13—C8	−73.6 (2)
C8—N9—C10—C11	14.4 (3)	O24—C12—C13—C14	55.6 (3)
C5—N9—C8—C13	−111.97 (19)	C8—C13—C14—C21	60.0 (3)
C10—N9—C8—C7	166.43 (18)	C12—C13—C14—C15	172.82 (17)
C16—N19—C18—C17	−17.7 (2)	C8—C13—C14—C15	−60.6 (2)
C20—N19—C16—C15	−9.8 (3)	C12—C13—C14—C21	−66.5 (3)
C18—N19—C16—C15	−134.06 (19)	C13—C14—C21—C20	−120.9 (2)
C18—N19—C20—C21	75.8 (2)	C13—C14—C21—C22	58.7 (3)

C20—N19—C16—C7	115.2 (2)	C15—C14—C21—C22	177.4 (2)
C16—N19—C20—C21	−46.1 (2)	C13—C14—C15—C16	68.9 (2)
C18—N19—C16—C7	−9.2 (2)	C21—C14—C15—C16	−55.0 (2)
C20—N19—C18—C17	−143.14 (18)	C15—C14—C21—C20	−2.1 (3)
C2—C1—C6—C7	−171.5 (2)	C14—C15—C16—N19	61.7 (2)
C6—C1—C2—C3	2.2 (3)	C14—C15—C16—C7	−56.8 (2)
C2—C1—C6—C5	0.3 (3)	C7—C17—C18—N19	37.9 (2)
C6—C1—C2—O2	−178.52 (19)	N19—C20—C21—C14	53.0 (2)
O2—C2—C3—C4	178.15 (18)	N19—C20—C21—C22	−126.5 (2)
O2—C2—C3—O3	−0.7 (3)	C20—C21—C22—C23	177.0 (2)
C1—C2—C3—C4	−2.6 (3)	C14—C21—C22—C23	−2.5 (4)
C1—C2—C3—O3	178.65 (18)	C21—C22—C23—O24	−64.5 (3)
C2—C3—C4—C5	0.3 (3)	C6A—C1A—C2A—C3A	7.0 (3)
O3—C3—C4—C5	178.97 (19)	C6A—C1A—C2A—C21A	−169.7 (2)
C3—C4—C5—C6	2.3 (3)	C11A—C1A—C2A—C3A	−169.4 (2)
C3—C4—C5—N9	−180.0 (2)	C11A—C1A—C2A—C21A	13.9 (4)
N9—C5—C6—C1	179.27 (19)	C2A—C1A—C6A—C5A	−6.6 (3)
C4—C5—C6—C1	−2.6 (3)	C11A—C1A—C6A—C5A	170.4 (2)
C4—C5—C6—C7	170.48 (19)	C2A—C1A—C11A—O11A	−177.8 (2)
N9—C5—C6—C7	−7.6 (2)	C2A—C1A—C11A—O12A	4.8 (4)
C1—C6—C7—C8	−172.8 (2)	C6A—C1A—C11A—O11A	5.6 (3)
C5—C6—C7—C8	14.7 (2)	C6A—C1A—C11A—O12A	−171.8 (2)
C5—C6—C7—C17	−105.2 (2)	C1A—C2A—C3A—C4A	−1.5 (3)
C1—C6—C7—C16	−48.2 (3)	C21A—C2A—C3A—C4A	175.8 (2)
C5—C6—C7—C16	139.34 (19)	C1A—C2A—C21A—O21A	−16.0 (4)
C1—C6—C7—C17	67.2 (3)	C1A—C2A—C21A—O22A	163.9 (2)
C6—C7—C16—C15	−85.3 (2)	C3A—C2A—C21A—O21A	167.1 (2)
C8—C7—C17—C18	78.8 (2)	C3A—C2A—C21A—O22A	−13.0 (3)
C16—C7—C17—C18	−43.2 (2)	C2A—C3A—C4A—Cl4A	174.28 (17)
C16—C7—C8—C13	−24.5 (3)	C2A—C3A—C4A—C5A	−4.9 (3)
C17—C7—C8—C13	−139.07 (18)	Cl4A—C4A—C5A—Cl5A	6.1 (3)
C6—C7—C16—N19	152.91 (18)	Cl4A—C4A—C5A—C6A	−173.72 (17)
C16—C7—C8—N9	−141.13 (17)	C3A—C4A—C5A—Cl5A	−174.72 (17)
C8—C7—C16—N19	−88.5 (2)	C3A—C4A—C5A—C6A	5.5 (3)
C6—C7—C17—C18	−166.49 (18)	Cl5A—C5A—C6A—C1A	−179.53 (17)
C17—C7—C8—N9	104.28 (19)	C4A—C5A—C6A—C1A	0.3 (3)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N19—H19···O11A	0.90 (3)	1.75 (3)	2.639 (3)	170 (2)
O21A—H21A···O12A	0.84 (5)	1.61 (5)	2.441 (3)	170 (4)
C3A—H3A···O22A	0.93	2.31	2.667 (3)	103
C4—H4···O25	0.93	2.45	2.953 (3)	114
C6A—H6A···O11A	0.93	2.26	2.635 (3)	103
C16—H16···O12A	0.98	2.55	3.402 (3)	145
C17—H17B···O2^{i}	0.97	2.53	3.441 (3)	156
C18—H18B···O3^{i}	0.97	2.48	3.279 (3)	140

C22—H22···O22A^{ii}	0.93	2.45	3.316 (3)	155

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

Fig. 1

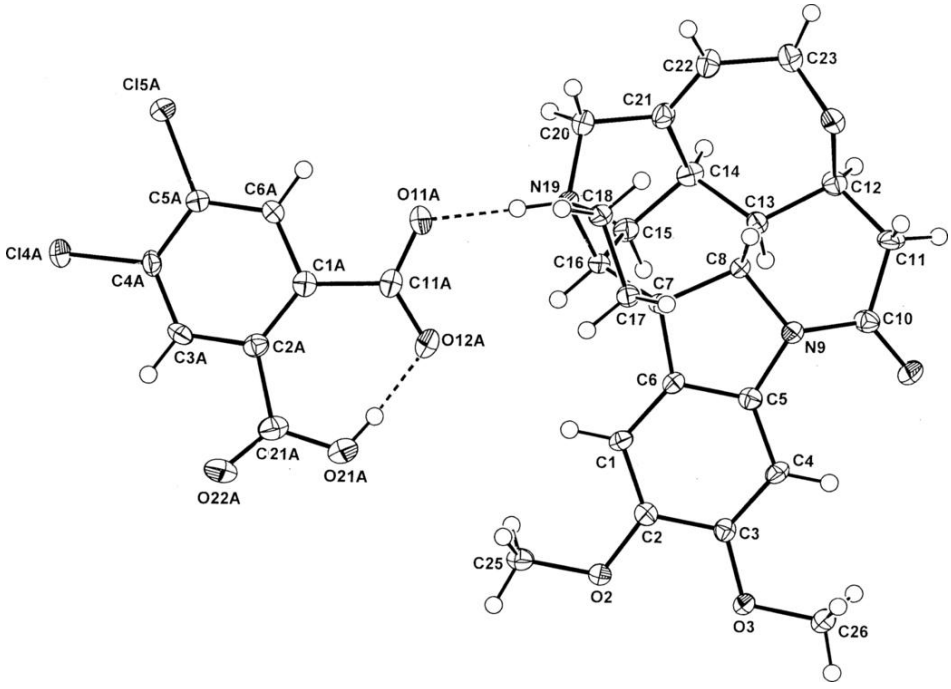

Fig. 2