

Rhodamine 6G hexachloridostannate(IV) acetonitrile disolvate

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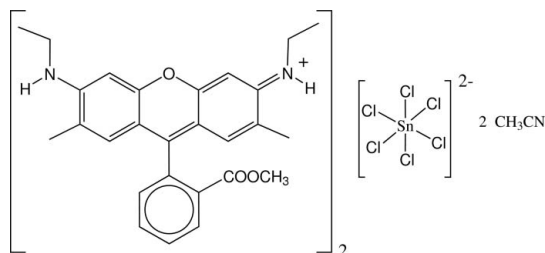
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Key indicators: single-crystal X-ray study; $T = 90$ K; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å; R factor = 0.029; wR factor = 0.073; data-to-parameter ratio = 39.8.

In the title compound, bis([6-ethylamino-10-[2-(methoxycarbonyl)phenyl]-2,7-dimethylxanthen-3-ylidene]ethanaminium) hexachloridotin(IV) acetonitrile disolvate, $(\text{C}_{27}\text{H}_{29}\text{N}_2\text{O}_3)_2[\text{SnCl}_6] \cdot 2\text{C}_2\text{H}_3\text{N}$, the octahedral SnCl_6^{2-} anion lies on an inversion center. The xanthen ring system is essentially planar, with an average deviation of 0.020 Å, and the substituent benzene ring forms a dihedral angle of 85.89 (2)° with it. The Sn—Cl distances are in the range 2.4237 (3)–2.4454 (3) Å. There are N—H...Cl hydrogen bonds between SnCl_6^{2-} ions and rhodamine 6G cations as well as π – π stacking interactions between rhodamine 6G cations (interplanar distance of 3.827 Å).

Related literature

For related literature, see: Bhagavathy *et al.* (1993); Fun *et al.* (1997); Herz (1974); Johnson & McGrane (1993); Liu *et al.* (1998); Nguyen & Meyer (1992); Wang *et al.* (1997). For the structure of the analogous ethyl ester as the chloride salt hydrate, see: Adhikesavalu *et al.* (2001).



Experimental

Crystal data

$(\text{C}_{27}\text{H}_{29}\text{N}_2\text{O}_3)_2[\text{SnCl}_6] \cdot 2\text{C}_2\text{H}_3\text{N}$

$M_r = 1272.54$

Triclinic, $P\bar{1}$

$a = 9.7871$ (10) Å

$b = 11.7827$ (11) Å

$c = 13.2893$ (12) Å

$\alpha = 80.583$ (4)°

$\beta = 77.309$ (4)°

$\gamma = 82.467$ (4)°

$V = 1467.7$ (2) Å³

$Z = 1$

Mo $K\alpha$ radiation

$\mu = 0.76$ mm⁻¹

$T = 90.0$ (5) K

$0.28 \times 0.27 \times 0.25$ mm

Data collection

Nonius KappaCCD diffractometer with Oxford Cryostream

Absorption correction: multi-scan (*DENZO* and *SCALEPACK*; Otwinowski & Minor, 1997)

$T_{\min} = 0.815$, $T_{\max} = 0.832$

54250 measured reflections

14412 independent reflections

12969 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.024$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.029$

$wR(F^2) = 0.073$

$S = 1.03$

14412 reflections

362 parameters

H atoms treated by a mixture of independent and constrained refinement

$\Delta\rho_{\text{max}} = 0.74$ e Å⁻³

$\Delta\rho_{\text{min}} = -1.24$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{N1}-\text{H1N}\cdots\text{Cl2}$	0.81 (2)	2.61 (2)	3.3644 (10)	156 (2)
$\text{N2}-\text{H2N}\cdots\text{Cl1}^{\dagger}$	0.86 (2)	2.75 (2)	3.5603 (10)	159 (2)

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

Data collection: *COLLECT* (Nonius, 2000); cell refinement: *DENZO* and *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO* and *SCALEPACK*; program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: PV2056).

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supplementary materials

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Rhodamine 6G hexachloridostannate(IV) acetonitrile disolvate

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Comment

The excellent photo-physical properties of rhodamines are well known (Herz, 1974; Johnson & McGrane, 1993; Nguyen & Meyer, 1992), and recently metal complexes of rhodamine 6 G have been reported by several authors (Bhagavathy *et al.*, 1993; Fun *et al.*, 1997; Wang *et al.*, 1997; Liu *et al.*, 1998). The aggregative properties of cationic species of the dye were observed to be dependent on the anionic environment created by the metal ions. We have synthesized a rhodamine 6 G derivative, 9-[2-methoxycarbonyl]phenyl]-3,6-bis(ethylamino)-2,7-dimethylxanthylium hexachlorotin(IV) diacetonitrile solvate, (I), the structure of which is presented in this paper.

The structure of (I) consists of discrete SnCl_6^{2-} anions lying on inversion centers, rhodamine 6 G cations and acetonitrile solvent molecules (Fig. 1). The xanthe ring of the cation is planar within an average deviation of 0.020 Å (maximum deviation is 0.045 (1) Å for C4), and the phenyl ring forms a dihedral angle of 85.89 (2)° with it. The C—N distances N1—C24 and N2—C26 are normal for this type of single bond, whereas C3—N1 and C11—N2 are much shorter, showing partial double-bond character; details are in the Table. A similar trend is observed in the other rhodamine 6 G cations (Wang *et al.*, 1997; Liu *et al.*, 1998).

Both hydrogen bonding between cations and anions and π - π stacking interactions between rhodamine 6 G cations exist. Parallel rhodamine ions related by the inversion center have an interplanar distance of 3.827 Å (Fig. 2), and are slipped such that their O1—C13 bonds exactly overlap.

Experimental

Diphenyl tin dichloride (0.344 g, 1 mmol) was dissolved in 20 ml methanol, and then 20 ml of methanol solution of rhodamine 6 G (0.479 g, 1 mmol) was added. The bright red solution was refluxed for 1 hr, whereafter red brown solid were obtained on cooling. Suitable size crystals were obtained by the recrystallization at room temperature from acetonitrile (yield ca 85%).

Refinement

H atoms were placed in idealized positions with C—H distances at 0.99, 0.98 and 0.95 Å for CH₂, CH₃ and aromatic CH groups, respectively using a riding model. U_{iso} for H was assigned as 1.2 times U_{eq} of the attached C atoms (1.5 for methyl); a torsional parameter was refined for each methyl group. The largest positive and negative residual density peaks were located within 1 Å of the Sn1 position.

Figures

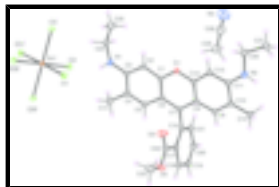


Fig. 1. Numbering scheme and ellipsoids at the 50% level. ($i = 1 - x, -y, -z$).

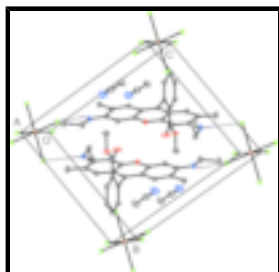


Fig. 2. The unit cell, showing stacking of rhodamine cations and hydrogen bonding.

bis({6-ethylamino-10-[2-(methoxycarbonyl)phenyl]-2,7-dimethylxanthen-3-ylidene}ethanaminium) hexachloridotin(IV) acetonitrile disolvate

Crystal data

$(C_{27}H_{29}N_2O_3)_2[SnCl_6] \cdot 2C_2H_3N$

$M_r = 1272.54$

Triclinic, $P\bar{1}$

Hall symbol: -P 1

$a = 9.7871$ (10) Å

$b = 11.7827$ (11) Å

$c = 13.2893$ (12) Å

$\alpha = 80.583$ (4)°

$\beta = 77.309$ (4)°

$\gamma = 82.467$ (4)°

$V = 1467.7$ (2) Å³

$Z = 1$

$F_{000} = 654$

$D_x = 1.440$ Mg m⁻³

Mo $K\alpha$ radiation

$\lambda = 0.71073$ Å

Cell parameters from 13141 reflections

$\theta = 2.5$ – 36.8 °

$\mu = 0.76$ mm⁻¹

$T = 90.0$ (5) K

Fragment, red-orange

$0.28 \times 0.27 \times 0.25$ mm

Data collection

Nonius KappaCCD
diffractometer with Oxford Cryostream

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 90.0$ (5) K

ω scans with κ offsets

Absorption correction: multi-scan

(DENZO and SCALEPACK; Otwinowski & Minor, 1997)

$T_{\min} = 0.815$, $T_{\max} = 0.832$

54250 measured reflections

14412 independent reflections

12969 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.024$

$\theta_{\text{max}} = 36.8$ °

$\theta_{\text{min}} = 2.7$ °

$h = -16 \rightarrow 16$

$k = -19 \rightarrow 19$

$l = -22 \rightarrow 21$

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
$R[F^2 > 2\sigma(F^2)] = 0.029$	$w = 1/[\sigma^2(F_o^2) + (0.0303P)^2 + 0.821P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.073$	$(\Delta/\sigma)_{\max} < 0.001$
$S = 1.03$	$\Delta\rho_{\max} = 0.74 \text{ e } \text{\AA}^{-3}$
14412 reflections	$\Delta\rho_{\min} = -1.24 \text{ e } \text{\AA}^{-3}$
362 parameters	Extinction correction: SHELXL, $F_c^* = kFc[1 + 0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Primary atom site location: structure-invariant direct methods	Extinction coefficient: 0.0053 (5)
Secondary atom site location: difference Fourier map	

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

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

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

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
Sn1	0.5000	0.0000	0.0000	0.00926 (3)
Cl1	0.53900 (3)	0.04133 (2)	0.164917 (19)	0.01502 (5)
Cl2	0.51522 (3)	0.20275 (2)	-0.07554 (2)	0.01362 (4)
Cl3	0.24802 (3)	0.04038 (2)	0.05732 (2)	0.01381 (4)
O1	0.40633 (8)	0.58975 (7)	0.38427 (6)	0.01354 (13)
O2	1.06811 (9)	0.46854 (8)	0.36352 (7)	0.01853 (15)
O3	0.83375 (10)	0.47563 (9)	0.39044 (9)	0.0272 (2)
N1	0.42673 (10)	0.33594 (9)	0.13842 (8)	0.01642 (17)
H1N	0.4731 (19)	0.3059 (16)	0.0897 (14)	0.020*
N2	0.35159 (10)	0.83221 (9)	0.64389 (7)	0.01542 (16)
H2N	0.3892 (19)	0.8743 (16)	0.6750 (14)	0.019*
C1	0.48174 (10)	0.54161 (9)	0.30004 (8)	0.01118 (15)
C2	0.41464 (10)	0.46614 (9)	0.26289 (8)	0.01275 (16)
H2	0.3206	0.4516	0.2948	0.015*
C3	0.48671 (11)	0.41085 (9)	0.17721 (8)	0.01291 (16)

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C4	0.63032 (11)	0.43418 (9)	0.12992 (8)	0.01360 (16)
C5	0.69115 (11)	0.51151 (9)	0.16815 (8)	0.01310 (16)
H5	0.7846	0.5275	0.1359	0.016*
C6	0.62049 (10)	0.56913 (9)	0.25423 (8)	0.01122 (15)
C7	0.68061 (10)	0.64746 (8)	0.29612 (8)	0.01058 (15)
C8	0.60111 (10)	0.69685 (9)	0.38325 (8)	0.01108 (15)
C9	0.65144 (10)	0.77664 (9)	0.43315 (8)	0.01170 (15)
H9	0.7429	0.8005	0.4054	0.014*
C10	0.57244 (11)	0.82019 (9)	0.51976 (8)	0.01222 (15)
C11	0.43234 (11)	0.78594 (9)	0.56098 (8)	0.01258 (16)
C12	0.38037 (11)	0.70714 (9)	0.51364 (8)	0.01331 (16)
H12	0.2889	0.6832	0.5408	0.016*
C13	0.46414 (10)	0.66475 (9)	0.42693 (8)	0.01159 (15)
C14	0.70941 (13)	0.37222 (11)	0.04109 (10)	0.0197 (2)
H14A	0.8074	0.3907	0.0232	0.030*
H14B	0.6655	0.3969	-0.0195	0.030*
H14C	0.7069	0.2886	0.0616	0.030*
C15	0.62856 (12)	0.90417 (10)	0.57043 (9)	0.01553 (17)
H15A	0.7239	0.9186	0.5326	0.023*
H15B	0.6310	0.8716	0.6428	0.023*
H15C	0.5674	0.9770	0.5689	0.023*
C16	0.82316 (10)	0.68456 (8)	0.24669 (8)	0.01064 (15)
C17	0.94862 (10)	0.62467 (9)	0.27097 (8)	0.01147 (15)
C18	1.07793 (11)	0.66730 (9)	0.22341 (8)	0.01360 (16)
H18	1.1625	0.6273	0.2402	0.016*
C19	1.08333 (11)	0.76787 (9)	0.15174 (8)	0.01417 (17)
H19	1.1713	0.7963	0.1196	0.017*
C20	0.95942 (12)	0.82636 (10)	0.12744 (9)	0.01498 (17)
H20	0.9629	0.8948	0.0784	0.018*
C21	0.82987 (11)	0.78526 (9)	0.17463 (8)	0.01412 (17)
H21	0.7457	0.8260	0.1576	0.017*
C22	0.94161 (11)	0.51676 (9)	0.34679 (9)	0.01400 (17)
C23	1.06484 (13)	0.36159 (11)	0.43498 (10)	0.0211 (2)
H23A	0.9925	0.3717	0.4976	0.032*
H23B	1.1569	0.3406	0.4543	0.032*
H23C	1.0429	0.3000	0.4014	0.032*
C24	0.28833 (12)	0.29543 (10)	0.18084 (9)	0.01633 (18)
H24A	0.2628	0.3019	0.2562	0.020*
H24B	0.2931	0.2127	0.1733	0.020*
C25	0.17375 (14)	0.36257 (13)	0.12788 (12)	0.0258 (2)
H25A	0.1665	0.4443	0.1367	0.039*
H25B	0.0836	0.3314	0.1593	0.039*
H25C	0.1973	0.3554	0.0535	0.039*
C26	0.20472 (12)	0.81093 (11)	0.68565 (9)	0.0185 (2)
H26A	0.2000	0.7296	0.7190	0.022*
H26B	0.1527	0.8234	0.6281	0.022*
C27	0.13662 (13)	0.89136 (12)	0.76511 (10)	0.0232 (2)
H27A	0.1871	0.8779	0.8227	0.035*
H27B	0.0382	0.8763	0.7920	0.035*

H27C	0.1406	0.9718	0.7319	0.035*
N3	-0.01218 (17)	0.87440 (15)	0.42927 (13)	0.0429 (4)
C28	0.09893 (16)	0.88240 (12)	0.37996 (11)	0.0259 (2)
C29	0.23901 (15)	0.89178 (14)	0.31671 (11)	0.0267 (3)
H29A	0.2723	0.8203	0.2862	0.040*
H29B	0.3031	0.9042	0.3603	0.040*
H29C	0.2364	0.9571	0.2609	0.040*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Sn1	0.01002 (4)	0.00916 (4)	0.00947 (4)	-0.00130 (3)	-0.00229 (3)	-0.00309 (3)
Cl1	0.01755 (10)	0.01736 (11)	0.01246 (10)	-0.00091 (8)	-0.00530 (8)	-0.00629 (8)
Cl2	0.01605 (10)	0.01023 (9)	0.01489 (10)	-0.00222 (7)	-0.00319 (8)	-0.00189 (7)
Cl3	0.01070 (9)	0.01542 (10)	0.01489 (10)	-0.00084 (7)	-0.00195 (7)	-0.00214 (8)
O1	0.0112 (3)	0.0151 (3)	0.0155 (3)	-0.0041 (2)	0.0009 (2)	-0.0078 (3)
O2	0.0122 (3)	0.0194 (4)	0.0205 (4)	0.0012 (3)	-0.0034 (3)	0.0050 (3)
O3	0.0140 (4)	0.0235 (4)	0.0401 (6)	-0.0072 (3)	-0.0080 (4)	0.0150 (4)
N1	0.0148 (4)	0.0163 (4)	0.0198 (4)	-0.0041 (3)	0.0003 (3)	-0.0103 (3)
N2	0.0155 (4)	0.0181 (4)	0.0135 (4)	-0.0043 (3)	0.0004 (3)	-0.0073 (3)
C1	0.0097 (3)	0.0108 (4)	0.0131 (4)	-0.0009 (3)	-0.0008 (3)	-0.0041 (3)
C2	0.0104 (4)	0.0126 (4)	0.0162 (4)	-0.0023 (3)	-0.0012 (3)	-0.0058 (3)
C3	0.0126 (4)	0.0116 (4)	0.0155 (4)	-0.0019 (3)	-0.0024 (3)	-0.0047 (3)
C4	0.0120 (4)	0.0142 (4)	0.0150 (4)	-0.0018 (3)	-0.0005 (3)	-0.0058 (3)
C5	0.0109 (4)	0.0143 (4)	0.0143 (4)	-0.0017 (3)	-0.0002 (3)	-0.0054 (3)
C6	0.0094 (3)	0.0113 (4)	0.0132 (4)	-0.0014 (3)	-0.0015 (3)	-0.0034 (3)
C7	0.0092 (3)	0.0106 (4)	0.0119 (4)	-0.0011 (3)	-0.0016 (3)	-0.0022 (3)
C8	0.0106 (4)	0.0112 (4)	0.0118 (4)	-0.0019 (3)	-0.0017 (3)	-0.0026 (3)
C9	0.0116 (4)	0.0114 (4)	0.0128 (4)	-0.0022 (3)	-0.0026 (3)	-0.0026 (3)
C10	0.0133 (4)	0.0121 (4)	0.0123 (4)	-0.0023 (3)	-0.0031 (3)	-0.0030 (3)
C11	0.0136 (4)	0.0126 (4)	0.0115 (4)	-0.0019 (3)	-0.0010 (3)	-0.0032 (3)
C12	0.0129 (4)	0.0138 (4)	0.0135 (4)	-0.0039 (3)	0.0007 (3)	-0.0052 (3)
C13	0.0112 (4)	0.0115 (4)	0.0127 (4)	-0.0025 (3)	-0.0015 (3)	-0.0038 (3)
C14	0.0171 (5)	0.0222 (5)	0.0207 (5)	-0.0045 (4)	0.0030 (4)	-0.0129 (4)
C15	0.0174 (4)	0.0168 (4)	0.0149 (4)	-0.0039 (3)	-0.0038 (3)	-0.0067 (3)
C16	0.0095 (3)	0.0114 (4)	0.0114 (4)	-0.0020 (3)	-0.0014 (3)	-0.0028 (3)
C17	0.0099 (4)	0.0109 (4)	0.0135 (4)	-0.0015 (3)	-0.0016 (3)	-0.0020 (3)
C18	0.0097 (4)	0.0142 (4)	0.0168 (4)	-0.0018 (3)	-0.0015 (3)	-0.0029 (3)
C19	0.0122 (4)	0.0149 (4)	0.0153 (4)	-0.0037 (3)	-0.0001 (3)	-0.0034 (3)
C20	0.0152 (4)	0.0147 (4)	0.0144 (4)	-0.0045 (3)	-0.0019 (3)	0.0005 (3)
C21	0.0121 (4)	0.0140 (4)	0.0158 (4)	-0.0018 (3)	-0.0031 (3)	-0.0002 (3)
C22	0.0124 (4)	0.0128 (4)	0.0167 (4)	-0.0012 (3)	-0.0042 (3)	-0.0002 (3)
C23	0.0200 (5)	0.0197 (5)	0.0199 (5)	0.0023 (4)	-0.0043 (4)	0.0049 (4)
C24	0.0158 (4)	0.0139 (4)	0.0206 (5)	-0.0046 (3)	-0.0018 (4)	-0.0065 (4)
C25	0.0188 (5)	0.0259 (6)	0.0337 (7)	-0.0029 (4)	-0.0078 (5)	-0.0030 (5)
C26	0.0152 (4)	0.0217 (5)	0.0186 (5)	-0.0044 (4)	0.0023 (4)	-0.0088 (4)
C27	0.0178 (5)	0.0290 (6)	0.0230 (5)	-0.0010 (4)	0.0017 (4)	-0.0134 (5)
N3	0.0356 (7)	0.0410 (8)	0.0433 (8)	-0.0075 (6)	0.0048 (6)	0.0053 (7)

supplementary materials

C28	0.0311 (6)	0.0223 (6)	0.0233 (6)	-0.0063 (5)	-0.0045 (5)	0.0009 (4)
C29	0.0252 (6)	0.0370 (7)	0.0190 (5)	-0.0098 (5)	-0.0050 (4)	-0.0010 (5)

Geometric parameters (Å, °)

Sn1—Cl3	2.4237 (3)	C14—H14A	0.9800
Sn1—Cl3 ⁱ	2.4237 (3)	C14—H14B	0.9800
Sn1—Cl1 ⁱ	2.4396 (3)	C14—H14C	0.9800
Sn1—Cl1	2.4396 (3)	C15—H15A	0.9800
Sn1—Cl2 ⁱ	2.4454 (3)	C15—H15B	0.9800
Sn1—Cl2	2.4454 (3)	C15—H15C	0.9800
O1—C1	1.3618 (12)	C16—C21	1.3966 (15)
O1—C13	1.3632 (12)	C16—C17	1.4080 (14)
O2—C22	1.3417 (13)	C17—C18	1.4017 (14)
O2—C23	1.4479 (15)	C17—C22	1.4865 (15)
O3—C22	1.2078 (14)	C18—C19	1.3925 (15)
N1—C3	1.3443 (13)	C18—H18	0.9500
N1—C24	1.4583 (15)	C19—C20	1.3893 (16)
N1—H1N	0.81 (2)	C19—H19	0.9500
N2—C11	1.3553 (13)	C20—C21	1.3955 (15)
N2—C26	1.4613 (15)	C20—H20	0.9500
N2—H2N	0.86 (2)	C21—H21	0.9500
C1—C2	1.3802 (14)	C23—H23A	0.9800
C1—C6	1.4169 (14)	C23—H23B	0.9800
C2—C3	1.4099 (14)	C23—H23C	0.9800
C2—H2	0.9500	C24—C25	1.5201 (18)
C3—C4	1.4500 (15)	C24—H24A	0.9900
C4—C5	1.3673 (14)	C24—H24B	0.9900
C4—C14	1.5027 (15)	C25—H25A	0.9800
C5—C6	1.4252 (14)	C25—H25B	0.9800
C5—H5	0.9500	C25—H25C	0.9800
C6—C7	1.4008 (14)	C26—C27	1.5198 (16)
C7—C8	1.4109 (14)	C26—H26A	0.9900
C7—C16	1.4941 (14)	C26—H26B	0.9900
C8—C13	1.4128 (14)	C27—H27A	0.9800
C8—C9	1.4266 (14)	C27—H27B	0.9800
C9—C10	1.3727 (14)	C27—H27C	0.9800
C9—H9	0.9500	N3—C28	1.146 (2)
C10—C11	1.4442 (15)	C28—C29	1.450 (2)
C10—C15	1.5024 (14)	C29—H29A	0.9800
C11—C12	1.4043 (14)	C29—H29B	0.9800
C12—C13	1.3839 (14)	C29—H29C	0.9800
C12—H12	0.9500		
Cl3—Sn1—Cl3 ⁱ	180.0	H14A—C14—H14C	109.5
Cl3—Sn1—Cl1 ⁱ	89.363 (10)	H14B—C14—H14C	109.5
Cl3 ⁱ —Sn1—Cl1 ⁱ	90.638 (10)	C10—C15—H15A	109.5
Cl3—Sn1—Cl1	90.638 (10)	C10—C15—H15B	109.5

C13 ⁱ —Sn1—C11	89.362 (10)	H15A—C15—H15B	109.5
C11 ⁱ —Sn1—C11	180.0	C10—C15—H15C	109.5
C13—Sn1—C12 ⁱ	90.291 (10)	H15A—C15—H15C	109.5
C13 ⁱ —Sn1—C12 ⁱ	89.709 (10)	H15B—C15—H15C	109.5
C11 ⁱ —Sn1—C12 ⁱ	90.829 (10)	C21—C16—C17	119.27 (9)
C11—Sn1—C12 ⁱ	89.170 (10)	C21—C16—C7	117.43 (9)
C13—Sn1—C12	89.709 (10)	C17—C16—C7	123.29 (9)
C13 ⁱ —Sn1—C12	90.290 (10)	C18—C17—C16	119.71 (9)
C11 ⁱ —Sn1—C12	89.171 (11)	C18—C17—C22	121.02 (9)
C11—Sn1—C12	90.830 (10)	C16—C17—C22	119.27 (9)
C12 ⁱ —Sn1—C12	180.0	C19—C18—C17	120.52 (10)
C1—O1—C13	120.30 (8)	C19—C18—H18	119.7
C22—O2—C23	114.57 (9)	C17—C18—H18	119.7
C3—N1—C24	126.24 (9)	C20—C19—C18	119.63 (10)
C3—N1—H1N	118.0 (13)	C20—C19—H19	120.2
C24—N1—H1N	115.6 (13)	C18—C19—H19	120.2
C11—N2—C26	123.53 (9)	C19—C20—C21	120.47 (10)
C11—N2—H2N	118.5 (12)	C19—C20—H20	119.8
C26—N2—H2N	118.0 (12)	C21—C20—H20	119.8
O1—C1—C2	116.05 (9)	C20—C21—C16	120.40 (10)
O1—C1—C6	121.03 (9)	C20—C21—H21	119.8
C2—C1—C6	122.93 (9)	C16—C21—H21	119.8
C1—C2—C3	119.50 (9)	O3—C22—O2	122.39 (10)
C1—C2—H2	120.3	O3—C22—C17	124.24 (10)
C3—C2—H2	120.3	O2—C22—C17	113.37 (9)
N1—C3—C2	121.95 (10)	O2—C23—H23A	109.5
N1—C3—C4	118.77 (9)	O2—C23—H23B	109.5
C2—C3—C4	119.29 (9)	H23A—C23—H23B	109.5
C5—C4—C3	119.00 (9)	O2—C23—H23C	109.5
C5—C4—C14	121.49 (10)	H23A—C23—H23C	109.5
C3—C4—C14	119.51 (9)	H23B—C23—H23C	109.5
C4—C5—C6	122.88 (9)	N1—C24—C25	113.19 (10)
C4—C5—H5	118.6	N1—C24—H24A	108.9
C6—C5—H5	118.6	C25—C24—H24A	108.9
C7—C6—C1	119.38 (9)	N1—C24—H24B	108.9
C7—C6—C5	124.23 (9)	C25—C24—H24B	108.9
C1—C6—C5	116.38 (9)	H24A—C24—H24B	107.8
C6—C7—C8	118.96 (9)	C24—C25—H25A	109.5
C6—C7—C16	121.31 (9)	C24—C25—H25B	109.5
C8—C7—C16	119.63 (9)	H25A—C25—H25B	109.5
C7—C8—C13	119.28 (9)	C24—C25—H25C	109.5
C7—C8—C9	124.03 (9)	H25A—C25—H25C	109.5
C13—C8—C9	116.67 (9)	H25B—C25—H25C	109.5
C10—C9—C8	122.33 (9)	N2—C26—C27	110.50 (10)
C10—C9—H9	118.8	N2—C26—H26A	109.5
C8—C9—H9	118.8	C27—C26—H26A	109.5
C9—C10—C11	119.04 (9)	N2—C26—H26B	109.5

supplementary materials

C9—C10—C15	121.00 (9)	C27—C26—H26B	109.5
C11—C10—C15	119.95 (9)	H26A—C26—H26B	108.1
N2—C11—C12	120.82 (10)	C26—C27—H27A	109.5
N2—C11—C10	119.37 (9)	C26—C27—H27B	109.5
C12—C11—C10	119.80 (9)	H27A—C27—H27B	109.5
C13—C12—C11	119.16 (9)	C26—C27—H27C	109.5
C13—C12—H12	120.4	H27A—C27—H27C	109.5
C11—C12—H12	120.4	H27B—C27—H27C	109.5
O1—C13—C12	115.99 (9)	N3—C28—C29	179.32 (18)
O1—C13—C8	121.03 (9)	C28—C29—H29A	109.5
C12—C13—C8	122.98 (9)	C28—C29—H29B	109.5
C4—C14—H14A	109.5	H29A—C29—H29B	109.5
C4—C14—H14B	109.5	C28—C29—H29C	109.5
H14A—C14—H14B	109.5	H29A—C29—H29C	109.5
C4—C14—H14C	109.5	H29B—C29—H29C	109.5
C13—O1—C1—C2	179.54 (9)	C9—C10—C11—C12	1.40 (15)
C13—O1—C1—C6	-0.75 (15)	C15—C10—C11—C12	-179.79 (10)
O1—C1—C2—C3	-178.69 (9)	N2—C11—C12—C13	177.80 (10)
C6—C1—C2—C3	1.61 (16)	C10—C11—C12—C13	-0.92 (16)
C24—N1—C3—C2	-4.43 (18)	C1—O1—C13—C12	-178.66 (9)
C24—N1—C3—C4	174.88 (11)	C1—O1—C13—C8	1.91 (15)
C1—C2—C3—N1	179.50 (10)	C11—C12—C13—O1	-179.22 (9)
C1—C2—C3—C4	0.18 (16)	C11—C12—C13—C8	0.19 (16)
N1—C3—C4—C5	179.10 (11)	C7—C8—C13—O1	-2.03 (15)
C2—C3—C4—C5	-1.56 (16)	C9—C8—C13—O1	179.45 (9)
N1—C3—C4—C14	-1.55 (16)	C7—C8—C13—C12	178.58 (10)
C2—C3—C4—C14	177.78 (11)	C9—C8—C13—C12	0.06 (15)
C3—C4—C5—C6	1.23 (17)	C6—C7—C16—C21	93.07 (12)
C14—C4—C5—C6	-178.10 (11)	C8—C7—C16—C21	-83.31 (12)
O1—C1—C6—C7	-0.26 (15)	C6—C7—C16—C17	-88.32 (13)
C2—C1—C6—C7	179.42 (10)	C8—C7—C16—C17	95.30 (12)
O1—C1—C6—C5	178.39 (9)	C21—C16—C17—C18	0.58 (15)
C2—C1—C6—C5	-1.93 (15)	C7—C16—C17—C18	-178.01 (9)
C4—C5—C6—C7	179.04 (10)	C21—C16—C17—C22	-179.32 (9)
C4—C5—C6—C1	0.46 (16)	C7—C16—C17—C22	2.09 (15)
C1—C6—C7—C8	0.11 (15)	C16—C17—C18—C19	-0.52 (15)
C5—C6—C7—C8	-178.42 (10)	C22—C17—C18—C19	179.38 (10)
C1—C6—C7—C16	-176.29 (9)	C17—C18—C19—C20	0.11 (16)
C5—C6—C7—C16	5.18 (16)	C18—C19—C20—C21	0.25 (16)
C6—C7—C8—C13	1.00 (15)	C19—C20—C21—C16	-0.19 (16)
C16—C7—C8—C13	177.46 (9)	C17—C16—C21—C20	-0.23 (15)
C6—C7—C8—C9	179.40 (10)	C7—C16—C21—C20	178.44 (10)
C16—C7—C8—C9	-4.14 (15)	C23—O2—C22—O3	2.09 (17)
C7—C8—C9—C10	-177.98 (10)	C23—O2—C22—C17	-178.43 (10)
C13—C8—C9—C10	0.46 (15)	C18—C17—C22—O3	179.92 (12)
C8—C9—C10—C11	-1.18 (15)	C16—C17—C22—O3	-0.18 (17)
C8—C9—C10—C15	-179.97 (10)	C18—C17—C22—O2	0.46 (15)
C26—N2—C11—C12	-4.18 (17)	C16—C17—C22—O2	-179.64 (9)
C26—N2—C11—C10	174.54 (10)	C3—N1—C24—C25	96.39 (14)

C9—C10—C11—N2	-177.33 (10)	C11—N2—C26—C27	-169.80 (11)
C15—C10—C11—N2	1.48 (15)		

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

Hydrogen-bond geometry (Å, °)

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
N1—H1N \cdots Cl2	0.81 (2)	2.61 (2)	3.3644 (10)	156 (2)
N2—H2N \cdots C11 ⁱⁱ	0.86 (2)	2.75 (2)	3.5603 (10)	159 (2)

Symmetry codes: (ii) $-x+1, -y+1, -z+1$.

Fig. 1

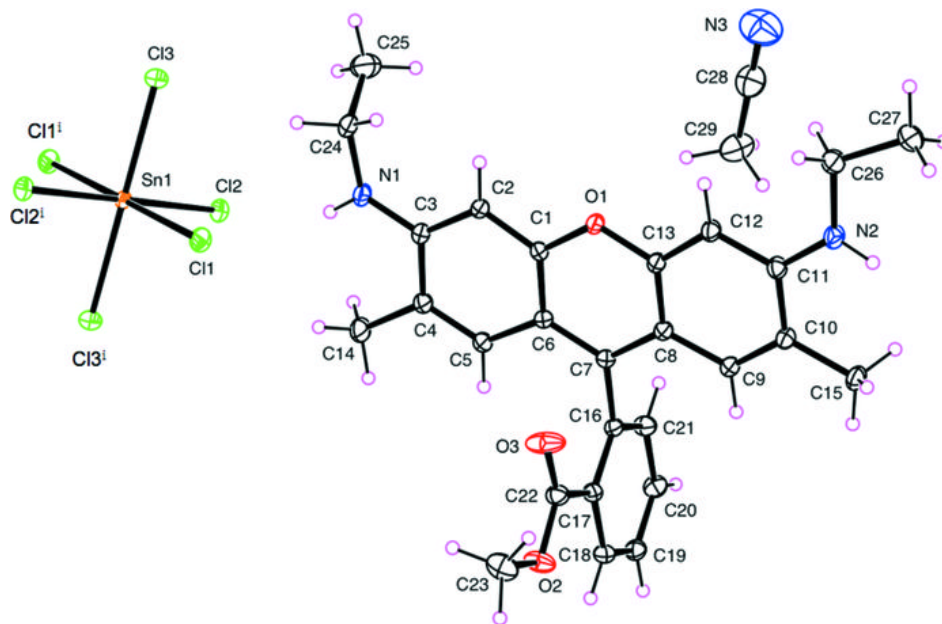


Fig. 2

