Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

9-(2-*tert*-Butylphenoxycarbonyl)-10-methylacridinium trifluoromethanesulfonate

Artur Sikorski,[a] Karol Krzymiński,[a] Piotr Malecha,[a] Tadeusz Lis[b] and Jerzy Błażejowski[a]*

[a]Faculty of Chemistry, University of Gdańsk, J. Sobieskiego 18, 80-952 Gdańsk, Poland, and [b]Faculty of Chemistry, University of Wrocław, F. Joliot-Curie 14, 50-383 Wrocław, Poland
Correspondence e-mail: bla@chem.univ.gda.pl

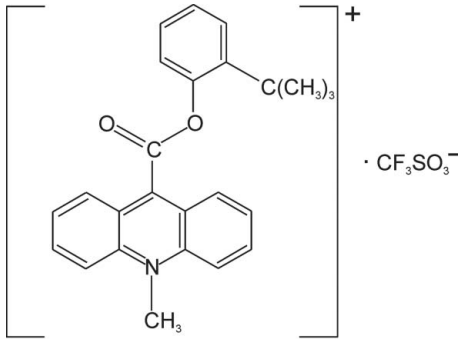
Received 1 October 2007; accepted 20 October 2007

Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.002 Å; R factor = 0.047; wR factor = 0.102; data-to-parameter ratio = 21.6.

The crystal structure of the title compound, $C_{25}H_{24}NO_2^+\cdot CF_3SO_3^-$, is stabilized by C—H···O, C—H···F and C—H···π hydrogen bonds, and by O···F [2.94 (1) Å] and O···N [2.87 (1) Å] interactions. In the packing of the molecules, acridine groups are either parallel or inclined at an angle of 5.4 (1)°. Similarly, the benzene rings are either parallel or lie at an angle of 72.4 (1)°.

Related literature

For related literature, see: Adamczyk *et al.* (2004); Allen *et al.* (1997); Bianchi *et al.* (2004); Dodeigne *et al.* (2000); Lee *et al.* (2003); Lyssenko & Antipin (2004); Meszko *et al.* (2002); Rak *et al.* (1999); Razawi & McCapra (2000); Roda *et al.* (2003); Sikorski *et al.* (2006*a*,*b*); Steiner (1999); Takahashi *et al.* (2001); Yang *et al.* (2002); Zomer & Jacquemijns (2001).



Experimental

Crystal data

$C_{25}H_{24}NO_2^+\cdot CF_3SO_3^-$
M_r = 519.52
Monoclinic, $P2_1/c$
a = 15.307 (4) Å
b = 13.480 (3) Å
c = 12.263 (3) Å
β = 102.56 (3)°
V = 2469.8 (10) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.19 mm^{-1}
T = 100 (2) K
0.50 × 0.40 × 0.07 mm

Data collection

Kuma KM4 CCD κ-geometry diffractometer
Absorption correction: none
29629 measured reflections
7120 independent reflections
4774 reflections with $I > 2\sigma(I)$
R_{int} = 0.056

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.102
S = 1.00
7120 reflections
329 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.39 e Å^{-3}
$\Delta\rho_{min}$ = −0.36 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C2—H2···O32	0.95	2.58	3.478 (2)	158
C4—H4···O31^{i}	0.95	2.35	3.285 (2)	168
C5—H5···O30^{ii}	0.95	2.45	3.330 (2)	155
C6—H6···O32^{ii}	0.95	2.42	3.265 (2)	148
C7—H7···F35^{iii}	0.95	2.46	3.264 (2)	143
C23—H23···F36^{iv}	0.95	2.52	3.208 (2)	130
C28—H28*A*···O32^{v}	0.98	2.42	3.251 (2)	142

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

Table 2
C—H···π interactions (Å,°).

X	H	J	H···J	X···J	X—H···J
C26	H26*B*	*Cg*4^{vi}	2.875	3.579 (2)	129

Symmetry code: (vi) $1-x, 1-y, 1-z$. *Cg*4 is the centroid of the C18–C23 ring.

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2003); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2003); data reduction: *CrysAlis RED*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPII* (Johnson, 1976); software used to prepare material for publication: *SHELXL97* and *PLATON* (Spek, 2003).

This study was financed by the State Funds for Scientific Research (grant No. BW/8000–5–0047–7).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: OM2169).

References

Adamczyk, M., Fino, J. R., Mattingly, P. G., Moore, J. A. & Pan, Y. (2004). *Bioorg. Med. Chem. Lett.* **14**, 2313–2317.
Allen, F. H., Lommerse, J. P. M., Hoy, V. J., Howard, J. A. K. & Desiraju, G. R. (1997). *Acta Cryst.* B**53**, 1006–1016.
Bianchi, R., Forni, A. & Pilati, T. (2004). *Acta Cryst.* B**60**, 559–568.
Dodeigne, C., Thunus, L. & Lejeune, R. (2000). *Talanta*, **51**, 415–439.
Johnson, C. K. (1976). *ORTEPII*. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.

  doi:10.1107/S1600536807052002

Lee, C.-R., Tang, T.-H., Chen, L. & Wang, Y. (2003). *Chem. Eur. J.* **9**, 3112–3121.
Lyssenko, K. A. & Antipin, M. Y. (2004). *Russ. Chem. Bull. Int. Ed.* **53**, 10–17.
Meszko, J., Krzymiński, K., Konitz, A. & Błażejowski, J. (2002). *Acta Cryst.* C**58**, o157–o158.
Oxford Diffraction (2003). *KM4 CCD Software.* Version 1.171. Oxford Diffraction Poland, Wrocław, Poland.
Rak, J., Skurski, P. & Błażejowski, J. (1999). *J. Org. Chem.* **64**, 3002–3008.
Razawi, Z. & McCapra, F. (2000). *Luminescence*, **15**, 239–245, 245–249.
Roda, A., Guardigli, M., Michelini, E., Mirasoli, M. & Pasini, P. (2003). *Anal. Chem.* **75**, 462A–470A.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Sikorski, A., Krzymiński, K., Białońska, A., Lis, T. & Błażejowski, J. (2006*a*). *Acta Cryst.* E**62**, o555–o558.
Sikorski, A., Krzymiński, K., Białońska, A., Lis, T. & Błażejowski, J. (2006*b*). *Acta Cryst.* E**62**, o822–o824.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.
Steiner, T. (1999). *Chem. Commun.* pp. 313–314.
Takahashi, O., Kohno, Y., Iwasaki, S., Saito, K., Iwaoka, M., Tomada, S., Umezawa, Y., Tsuboyama, S. & Nishio, M. (2001). *Bull. Chem. Soc. Jpn*, **74**, 2421–2430.
Yang, M., Liu, C., Hu, H., He, P. & Fang, Y. (2002). *Anal. Chim. Acta*, **461**, 141–146.
Zomer, G. & Jacquemijns, M. (2001). *Chemiluminescence in Analytical Chemistry*, edited by A. M. Garcia-Campana & W. R. G. Baeyens, pp. 529–549. New York: Marcel Dekker.

supplementary materials

Acta Cryst. (2007). E**63**, o4484-o4485 [**doi:10.1107/S1600536807052002**]

9-(2-*tert*-Butylphenoxycarbonyl)-10-methylacridinium trifluoromethanesulfonate

A. Sikorski, K. Krzyminski, P. Malecha, T. Lis and J. Blazejowski

Comment

Phenyl 10-alkylacridinium-9-carboxylates have been successfully applied as chemiluminescent indicators or chemiluminogenic fragments of chemiluminescent labels in assays of biologically and environmentally important entities (Yang *et al.*, 2002; Adamczyk *et al.*, 2004). The reactions of the above-mentioned cations with hydrogen peroxide in alkaline media produce light, and determination of its intensity enables labeled entities or entities present in the medium to be assayed quantitatively at the attomole level (Roda *et al.*, 2003). Our own investigations (Rak *et al.*, 1999) and those of others (Dodeigne *et al.*, 2000; Razawi & McCapra, 2000; Zomer & Jacquemijns, 2001) have revealed that oxidation of these compounds is accompanied by the removal of the phenyl fragment and conversion of the rest of the molecule to electronically excited, light-emitting 10-alkyl-9-acridinones. It may thus be expected that the efficiency of chemiluminescence is affected by changes in the constitution of the phenyl ester fragment. In order to find out whether this is indeed the case investigations were undertaken on phenyl 10-methylacridinium-9-carboxylates differently substituted in the phenyl fragment. Alkyl-substituted representatives of this group of compounds were selected for these investigations principally because it is relatively easy to synthesize them and to influence their structure and features. The structure of 9-(2-methylphenoxycarbonyl-acridinium trifluoromethanesulfonate was described in an earlier report (Sikorski *et al.*, 2006*b*). Here the crystal structure of the title compound is presented.

Parameters characterizing the geometries of the central acridine ring and the ester fragment are typical of acridine-based derivatives (Meszko *et al.*, 2002; Sikorski *et al.*, 2006*a*,b). With respective average deviations from planarity of 0.009 and 0.002 Å, the acridine and benzene ring systems in the cation are oriented at 53.7 (1)° to each other (Fig. 1). The carboxyl group is twisted at an angle of 60.6 (1)° relative to the acridine skeleton. The mean planes of the acridine moieties lie either parallel or are inclined at an angle of 5.4 (1)° in the lattice. The benzene rings are either parallel or inclined at an angle of 72.4 (1)°.

All the O atoms and two F atoms of the trifluoromethanesulfonate anions are respectively involved in weak C–H···O and C–H···F hydrogen bonds with cations (Fig. 3). Adjacent cations are linked through C—H···π (phenyl) interactions (Figs. 2 and 3) and N···O (carbonyl) contacts [N10···O17 = 2.87 (1) Å (symmetry code: (vii) x, 1/2 − y, z − 1/2); Fig. 3]. Adjacent anions are linked through O···F contacts [O32···F34 = 2.94 (1) Å (symmetry code: (viii) x, 3/2 − y, z − 1/2); Fig. 2].

All interactions demonstrated were found by *PLATON* (Spek, 2003). The C–H···O (Bianchi *et al.*, 2004; Steiner, 1999), C–H···F (Bianchi *et al.*, 2004; Lyssenko & Antipin, 2004) and C–H···π (Takahashi *et al.*, 2001) interactions exhibit a hydrogen-bond-type nature. The C–F···O interactions between anions (Allen *et al.*, 1997; Lyssenko & Antipin, 2004) identified as O···F contacts, and also the N···O (carbonyl) contacts between cations (Lee *et al.*, 2003), should be of an attractive nature.

The crystal structure is stabilized by a network of the aforementioned short-range interactions, as well as by long-range electrostatic interactions between ions.

Experimental

9-(2-Tertbutylphenoxycarbonyl)-10-methylacridinium trifluoromethanesulfonate was synthesized by treatment of 2-tert-butylphenyl acridine-9-carboxylate [obtained in the same way as described elsewhere (Sikorski *et al.*, 2006*a*)], dissolved in anhydrous dichloromethane, with a fivefold molar excess of methyl trifluoromethanesulfonate, dissolved in the same solvent, under an Ar atmosphere at room temperature for 3 h. The crude salt was purified by repeated precipitation from ethanol-diethyl ether (1/20 *v*/*v*) solution (yield 63%). Pale-yellow crystals suitable for X-ray investigations were grown from absolute ethanol (m.p. = 502–504 K).

Refinement

All H atoms were positioned geometrically and refined using a riding model, with C—H distances of 0.95 Å and with U_{iso}(H) = 1.2U_{eq}(C), or C—H = 0.98 Å and U_{iso}(H) = 1.5U_{eq}(C) for the methyl group.

Figures

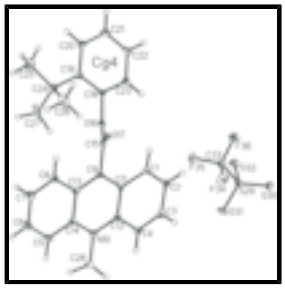


Fig. 1. The molecular structure of the title compound showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 25% probability level and H atoms are shown as small spheres of arbitrary radii. *Cg*4 denotes the ring centroid.

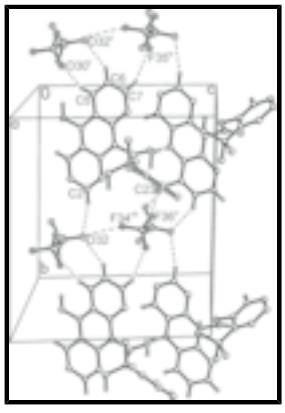


Fig. 2. The arrangement of the ions in the unit cell, viewed approximately along the *a* axis. The C—H···O and C—H···F interactions, as well as O···F contacts are represented by dashed lines. H atoms not involved in interactions have been omitted. [Symmetry codes: (ii) *x*, *y* − 1, *z*; (iii) *x*, 1/2 − *y*, *z* + 1/2; (iv) *x*, 3/2 − *y*, *z* + 1/2; (viii) *x*, 3/2 − *y*, *z* − 1/2.]

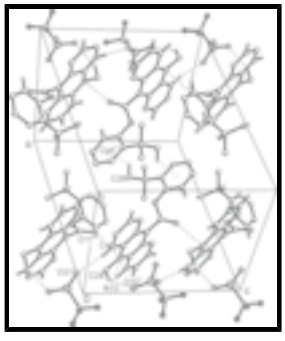


Fig. 3. The arrangement of the ions of in the unit cell, viewed approximately along the *b* axis. The C—H···O interactions and N···O contacts are represented by dashed lines, and C—H···π interactions by dotted lines. H atoms not involved in interactions have been omitted. [Symmetry codes: (i) −*x*, 1 − *y*, −*z*; (v) −*x*, *y* − 1/2, 1/2 − *z*; (vi) 1 − *x*,1 − *y*,1 − *z*; (vii) *x*, 1/2 − *y*, *z* − 1/2.]

9-(2-*tert*-Butylphenoxycarbonyl)-10-methylacridinium trifluoromethanesulfonate

Crystal data

$C_{25}H_{24}NO_2^+ \cdot CF_3O_3S^-$	$F_{000} = 1080$
$M_r = 519.52$	$D_x = 1.397$ Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
Hall symbol: -P 2ybc	Cell parameters from 29629 reflections
$a = 15.307$ (4) Å	$\theta = 3.1$–30.0º
$b = 13.480$ (3) Å	$\mu = 0.19$ mm^{-1}
$c = 12.263$ (3) Å	$T = 100$ (2) K
$\beta = 102.56$ (3)º	Plate, pale-yellow
$V = 2469.8$ (10) Å^3	0.50 × 0.40 × 0.07 mm
$Z = 4$	

Data collection

Kuma KM4 CCD κ-geometry diffractometer	4774 reflections with $I > 2\sigma(I)$
Radiation source: fine-focus sealed tube	$R_{int} = 0.056$
Monochromator: graphite	$\theta_{max} = 30.0$º
$T = 100$(2) K	$\theta_{min} = 3.1$º
ω scans	$h = -20 \rightarrow 21$
Absorption correction: none	$k = -18 \rightarrow 15$
29629 measured reflections	$l = -17 \rightarrow 16$
7120 independent reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.047$	H-atom parameters constrained
$wR(F^2) = 0.102$	$w = 1/[\sigma^2(F_o^2) + (0.0485P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.00$	$(\Delta/\sigma)_{max} = 0.001$
7120 reflections	$\Delta\rho_{max} = 0.39$ e Å^{-3}
329 parameters	$\Delta\rho_{min} = -0.36$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

Special details

Experimental. no

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.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.18044 (10)	0.44204 (12)	0.37019 (13)	0.0225 (3)
H1	0.2153	0.4694	0.4371	0.027*
C2	0.14022 (10)	0.50362 (12)	0.28609 (14)	0.0257 (4)
H2	0.1476	0.5734	0.2937	0.031*
C3	0.08736 (11)	0.46260 (13)	0.18723 (14)	0.0263 (4)
H3	0.0602	0.5060	0.1286	0.032*
C4	0.07408 (10)	0.36328 (12)	0.17317 (13)	0.0240 (3)
H4	0.0373	0.3383	0.1063	0.029*
C5	0.13276 (11)	0.02677 (12)	0.31435 (15)	0.0275 (4)
H5	0.0960	0.0010	0.2479	0.033*
C6	0.17425 (12)	−0.03608 (13)	0.39641 (16)	0.0322 (4)
H6	0.1658	−0.1056	0.3863	0.039*
C7	0.22935 (12)	−0.00065 (13)	0.49580 (15)	0.0307 (4)
H7	0.2576	−0.0461	0.5516	0.037*
C8	0.24223 (11)	0.09821 (12)	0.51229 (14)	0.0254 (4)
H8	0.2796	0.1215	0.5796	0.030*
C9	0.21201 (9)	0.27061 (11)	0.44236 (12)	0.0180 (3)
N10	0.10445 (8)	0.19637 (9)	0.24688 (11)	0.0203 (3)
C11	0.17080 (10)	0.33657 (11)	0.35902 (12)	0.0190 (3)
C12	0.11555 (10)	0.29719 (11)	0.25893 (13)	0.0199 (3)
C13	0.20006 (10)	0.16768 (11)	0.42931 (13)	0.0190 (3)
C14	0.14451 (10)	0.13085 (11)	0.32812 (13)	0.0200 (3)
C15	0.26928 (9)	0.30738 (11)	0.55043 (13)	0.0184 (3)
O16	0.34079 (7)	0.35813 (7)	0.53231 (8)	0.0187 (2)
O17	0.25354 (7)	0.29088 (8)	0.64040 (9)	0.0262 (3)
C18	0.40531 (10)	0.39120 (11)	0.62741 (13)	0.0191 (3)
C19	0.49459 (10)	0.36474 (11)	0.63384 (13)	0.0228 (3)
C20	0.55491 (11)	0.40454 (13)	0.72586 (15)	0.0308 (4)
H20	0.6168	0.3898	0.7350	0.037*
C21	0.52788 (12)	0.46475 (13)	0.80441 (15)	0.0337 (4)
H21	0.5711	0.4899	0.8658	0.040*
C22	0.43907 (12)	0.48808 (12)	0.79388 (14)	0.0290 (4)
H22	0.4206	0.5291	0.8477	0.035*
C23	0.37686 (11)	0.45117 (11)	0.70407 (14)	0.0234 (3)
H23	0.3152	0.4669	0.6952	0.028*
C24	0.52464 (11)	0.29761 (12)	0.54776 (15)	0.0285 (4)
C25	0.62422 (13)	0.27135 (19)	0.5846 (2)	0.0582 (7)
H25C	0.6411	0.2266	0.5296	0.087*
H25B	0.6351	0.2385	0.6576	0.087*
H25A	0.6601	0.3321	0.5900	0.087*

C26	0.50849 (15)	0.34945 (15)	0.43347 (17)	0.0466 (5)
H26C	0.5263	0.3051	0.3788	0.070*
H26B	0.5440	0.4105	0.4398	0.070*
H26A	0.4448	0.3658	0.4090	0.070*
C27	0.47260 (12)	0.19901 (12)	0.53535 (16)	0.0303 (4)
H27C	0.5035	0.1502	0.4979	0.045*
H27B	0.4120	0.2098	0.4908	0.045*
H27A	0.4692	0.1742	0.6094	0.045*
C28	0.04879 (11)	0.15863 (13)	0.14064 (14)	0.0286 (4)
H28C	0.0551	0.0864	0.1374	0.043*
H28B	0.0686	0.1890	0.0775	0.043*
H28A	−0.0141	0.1755	0.1368	0.043*
S29	0.08377 (3)	0.77767 (3)	0.14174 (3)	0.02144 (10)
O30	0.04026 (8)	0.87134 (8)	0.11081 (9)	0.0279 (3)
O31	0.04331 (8)	0.69409 (9)	0.07621 (10)	0.0339 (3)
O32	0.11069 (8)	0.75903 (9)	0.26006 (9)	0.0312 (3)
C33	0.19027 (11)	0.79272 (12)	0.10050 (14)	0.0257 (4)
F34	0.17898 (7)	0.80771 (9)	−0.00912 (9)	0.0457 (3)
F35	0.24265 (7)	0.71301 (8)	0.12630 (9)	0.0428 (3)
F36	0.23632 (7)	0.86934 (8)	0.15244 (10)	0.0414 (3)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0206 (8)	0.0202 (8)	0.0250 (8)	0.0011 (6)	0.0011 (6)	−0.0019 (6)
C2	0.0244 (8)	0.0203 (8)	0.0322 (9)	0.0024 (7)	0.0057 (7)	0.0031 (7)
C3	0.0228 (8)	0.0296 (9)	0.0262 (9)	0.0053 (7)	0.0051 (7)	0.0070 (7)
C4	0.0192 (8)	0.0330 (9)	0.0190 (8)	0.0018 (7)	0.0025 (6)	−0.0003 (7)
C5	0.0255 (9)	0.0235 (9)	0.0326 (10)	−0.0062 (7)	0.0047 (7)	−0.0090 (7)
C6	0.0364 (10)	0.0178 (8)	0.0422 (11)	−0.0036 (7)	0.0081 (8)	−0.0038 (8)
C7	0.0330 (10)	0.0200 (8)	0.0379 (10)	−0.0005 (7)	0.0046 (8)	0.0036 (8)
C8	0.0239 (8)	0.0218 (8)	0.0287 (9)	−0.0023 (7)	0.0018 (7)	0.0004 (7)
C9	0.0143 (7)	0.0209 (8)	0.0192 (7)	−0.0009 (6)	0.0044 (5)	−0.0016 (6)
N10	0.0160 (6)	0.0232 (7)	0.0212 (7)	−0.0008 (5)	0.0032 (5)	−0.0061 (5)
C11	0.0167 (7)	0.0202 (8)	0.0205 (8)	0.0010 (6)	0.0052 (6)	−0.0016 (6)
C12	0.0145 (7)	0.0244 (8)	0.0212 (8)	0.0014 (6)	0.0049 (6)	−0.0019 (6)
C13	0.0157 (7)	0.0194 (8)	0.0227 (8)	−0.0014 (6)	0.0057 (6)	−0.0013 (6)
C14	0.0152 (7)	0.0216 (8)	0.0244 (8)	−0.0017 (6)	0.0068 (6)	−0.0039 (7)
C15	0.0151 (7)	0.0159 (7)	0.0237 (8)	0.0006 (6)	0.0032 (6)	0.0001 (6)
O16	0.0175 (5)	0.0184 (5)	0.0193 (5)	−0.0034 (4)	0.0018 (4)	0.0000 (4)
O17	0.0259 (6)	0.0309 (6)	0.0227 (6)	−0.0077 (5)	0.0068 (5)	−0.0016 (5)
C18	0.0198 (8)	0.0140 (7)	0.0210 (8)	−0.0048 (6)	−0.0013 (6)	0.0024 (6)
C19	0.0208 (8)	0.0201 (8)	0.0271 (8)	−0.0020 (6)	0.0037 (6)	0.0032 (7)
C20	0.0207 (8)	0.0281 (9)	0.0396 (10)	−0.0019 (7)	−0.0023 (7)	0.0015 (8)
C21	0.0331 (10)	0.0282 (10)	0.0328 (10)	−0.0073 (8)	−0.0081 (8)	−0.0024 (8)
C22	0.0367 (10)	0.0216 (9)	0.0268 (9)	−0.0041 (7)	0.0029 (7)	−0.0042 (7)
C23	0.0224 (8)	0.0178 (8)	0.0294 (9)	−0.0008 (6)	0.0045 (7)	0.0004 (7)
C24	0.0239 (8)	0.0280 (9)	0.0354 (10)	−0.0007 (7)	0.0107 (7)	−0.0005 (7)

C25	0.0235 (10)	0.0716 (16)	0.0808 (17)	0.0045 (10)	0.0139 (10)	−0.0276 (14)
C26	0.0683 (15)	0.0342 (11)	0.0479 (13)	−0.0014 (10)	0.0361 (11)	0.0058 (9)
C27	0.0342 (10)	0.0215 (9)	0.0373 (10)	0.0054 (7)	0.0124 (8)	0.0002 (7)
C28	0.0270 (9)	0.0296 (9)	0.0258 (9)	−0.0018 (7)	−0.0014 (7)	−0.0090 (7)
S29	0.02347 (19)	0.0218 (2)	0.01792 (19)	−0.00431 (16)	0.00205 (14)	−0.00007 (16)
O30	0.0275 (6)	0.0292 (6)	0.0258 (6)	0.0059 (5)	0.0033 (5)	−0.0017 (5)
O31	0.0408 (7)	0.0270 (6)	0.0284 (7)	−0.0141 (5)	−0.0043 (5)	−0.0001 (5)
O32	0.0380 (7)	0.0351 (7)	0.0190 (6)	−0.0063 (5)	0.0028 (5)	0.0036 (5)
C33	0.0258 (8)	0.0270 (9)	0.0234 (8)	0.0018 (7)	0.0033 (6)	−0.0023 (7)
F34	0.0346 (6)	0.0779 (9)	0.0271 (6)	0.0028 (6)	0.0124 (5)	0.0057 (6)
F35	0.0349 (6)	0.0407 (6)	0.0488 (7)	0.0174 (5)	0.0001 (5)	−0.0094 (5)
F36	0.0302 (6)	0.0375 (6)	0.0564 (7)	−0.0148 (5)	0.0092 (5)	−0.0106 (5)

Geometric parameters (Å, °)

C1—C2	1.362 (2)	C19—C20	1.400 (2)
C1—C11	1.433 (2)	C19—C24	1.535 (2)
C1—H1	0.9500	C20—C21	1.390 (3)
C2—C3	1.416 (2)	C20—H20	0.9500
C2—H2	0.9500	C21—C22	1.374 (3)
C3—C4	1.359 (2)	C21—H21	0.9500
C3—H3	0.9500	C22—C23	1.383 (2)
C4—C12	1.418 (2)	C22—H22	0.9500
C4—H4	0.9500	C23—H23	0.9500
C5—C6	1.362 (2)	C24—C25	1.534 (3)
C5—C14	1.420 (2)	C24—C26	1.537 (3)
C5—H5	0.9500	C24—C27	1.540 (2)
C6—C7	1.406 (3)	C25—H25C	0.9800
C6—H6	0.9500	C25—H25B	0.9800
C7—C8	1.356 (2)	C25—H25A	0.9800
C7—H7	0.9500	C26—H26C	0.9800
C8—C13	1.430 (2)	C26—H26B	0.9800
C8—H8	0.9500	C26—H26A	0.9800
C9—C11	1.397 (2)	C27—H27C	0.9800
C9—C13	1.404 (2)	C27—H27B	0.9800
C9—C15	1.505 (2)	C27—H27A	0.9800
N10—C14	1.372 (2)	C28—H28C	0.9800
N10—C12	1.374 (2)	C28—H28B	0.9800
N10—C28	1.483 (2)	C28—H28A	0.9800
C11—C12	1.433 (2)	S29—O30	1.4395 (12)
C13—C14	1.432 (2)	S29—O32	1.4416 (12)
C15—O16	1.3496 (18)	S29—O31	1.4431 (12)
C15—O17	1.2001 (18)	S29—C33	1.8202 (17)
O16—C18	1.4259 (18)	C33—F36	1.3319 (19)
C18—C23	1.380 (2)	C33—F34	1.3330 (19)
C18—C19	1.398 (2)	C33—F35	1.3369 (19)
C2—C1—C11	120.80 (15)	C21—C20—H20	118.7
C2—C1—H1	119.6	C19—C20—H20	118.7
C11—C1—H1	119.6	C22—C21—C20	120.42 (16)

C1—C2—C3	119.37 (15)	C22—C21—H21	119.8
C1—C2—H2	120.3	C20—C21—H21	119.8
C3—C2—H2	120.3	C21—C22—C23	119.25 (16)
C4—C3—C2	122.35 (15)	C21—C22—H22	120.4
C4—C3—H3	118.8	C23—C22—H22	120.4
C2—C3—H3	118.8	C18—C23—C22	119.25 (15)
C3—C4—C12	119.61 (15)	C18—C23—H23	120.4
C3—C4—H4	120.2	C22—C23—H23	120.4
C12—C4—H4	120.2	C25—C24—C19	111.16 (15)
C6—C5—C14	120.00 (16)	C25—C24—C26	109.00 (17)
C6—C5—H5	120.0	C19—C24—C26	110.26 (14)
C14—C5—H5	120.0	C25—C24—C27	106.82 (15)
C5—C6—C7	121.63 (16)	C19—C24—C27	110.54 (13)
C5—C6—H6	119.2	C26—C24—C27	108.97 (15)
C7—C6—H6	119.2	C24—C25—H25C	109.5
C8—C7—C6	120.23 (16)	C24—C25—H25B	109.5
C8—C7—H7	119.9	H25C—C25—H25B	109.5
C6—C7—H7	119.9	C24—C25—H25A	109.5
C7—C8—C13	120.61 (16)	H25C—C25—H25A	109.5
C7—C8—H8	119.7	H25B—C25—H25A	109.5
C13—C8—H8	119.7	C24—C26—H26C	109.5
C11—C9—C13	121.23 (14)	C24—C26—H26B	109.5
C11—C9—C15	121.22 (13)	H26C—C26—H26B	109.5
C13—C9—C15	117.54 (13)	C24—C26—H26A	109.5
C14—N10—C12	122.24 (13)	H26C—C26—H26A	109.5
C14—N10—C28	119.84 (13)	H26B—C26—H26A	109.5
C12—N10—C28	117.90 (13)	C24—C27—H27C	109.5
C9—C11—C12	118.65 (14)	C24—C27—H27B	109.5
C9—C11—C1	122.73 (14)	H27C—C27—H27B	109.5
C12—C11—C1	118.61 (14)	C24—C27—H27A	109.5
N10—C12—C4	121.11 (14)	H27C—C27—H27A	109.5
N10—C12—C11	119.66 (14)	H27B—C27—H27A	109.5
C4—C12—C11	119.22 (14)	N10—C28—H28C	109.5
C9—C13—C8	122.60 (14)	N10—C28—H28B	109.5
C9—C13—C14	118.65 (14)	H28C—C28—H28B	109.5
C8—C13—C14	118.74 (14)	N10—C28—H28A	109.5
N10—C14—C5	121.64 (14)	H28C—C28—H28A	109.5
N10—C14—C13	119.56 (13)	H28B—C28—H28A	109.5
C5—C14—C13	118.80 (14)	O30—S29—O32	115.64 (7)
O17—C15—O16	125.27 (14)	O30—S29—O31	114.84 (7)
C9—C15—O16	111.10 (13)	O32—S29—O31	114.74 (7)
C9—C15—O17	123.61 (13)	O30—S29—C33	102.71 (7)
C15—O16—C18	117.78 (11)	O32—S29—C33	102.82 (8)
C23—C18—C19	124.09 (14)	O31—S29—C33	103.54 (8)
C23—C18—O16	118.38 (13)	F36—C33—F34	107.68 (14)
C19—C18—O16	117.45 (14)	F36—C33—F35	106.40 (13)
C18—C19—C20	114.39 (15)	F34—C33—F35	107.46 (13)
C18—C19—C24	123.17 (14)	F36—C33—S29	111.49 (11)
C20—C19—C24	122.44 (15)	F34—C33—S29	111.69 (11)

C21—C20—C19	122.61 (16)	F35—C33—S29	111.84 (12)
C11—C1—C2—C3	0.8 (2)	C9—C13—C14—C5	179.59 (14)
C1—C2—C3—C4	0.8 (2)	C8—C13—C14—C5	0.5 (2)
C2—C3—C4—C12	−1.2 (2)	C11—C9—C15—O17	119.85 (17)
C14—C5—C6—C7	−0.1 (3)	C13—C9—C15—O17	−58.9 (2)
C5—C6—C7—C8	0.2 (3)	C11—C9—C15—O16	−61.87 (17)
C6—C7—C8—C13	0.1 (3)	C13—C9—C15—O16	119.39 (14)
C13—C9—C11—C12	0.0 (2)	O17—C15—O16—C18	3.0 (2)
C15—C9—C11—C12	−178.70 (13)	C9—C15—O16—C18	−175.23 (12)
C13—C9—C11—C1	179.05 (14)	C15—O16—C18—C23	−56.98 (18)
C15—C9—C11—C1	0.4 (2)	C15—O16—C18—C19	126.28 (15)
C2—C1—C11—C9	178.95 (15)	C23—C18—C19—C20	−0.1 (2)
C2—C1—C11—C12	−2.0 (2)	O16—C18—C19—C20	176.40 (13)
C14—N10—C12—C4	179.37 (14)	C23—C18—C19—C24	179.78 (15)
C28—N10—C12—C4	0.9 (2)	O16—C18—C19—C24	−3.7 (2)
C14—N10—C12—C11	0.2 (2)	C18—C19—C20—C21	0.4 (2)
C28—N10—C12—C11	−178.30 (13)	C24—C19—C20—C21	−179.52 (16)
C3—C4—C12—N10	−179.19 (14)	C19—C20—C21—C22	−0.3 (3)
C3—C4—C12—C11	0.0 (2)	C20—C21—C22—C23	−0.2 (3)
C9—C11—C12—N10	−0.1 (2)	C19—C18—C23—C22	−0.3 (2)
C1—C11—C12—N10	−179.23 (13)	O16—C18—C23—C22	−176.77 (13)
C9—C11—C12—C4	−179.34 (13)	C21—C22—C23—C18	0.4 (2)
C1—C11—C12—C4	1.6 (2)	C18—C19—C24—C25	−172.91 (17)
C11—C9—C13—C8	179.12 (14)	C20—C19—C24—C25	7.0 (2)
C15—C9—C13—C8	−2.1 (2)	C18—C19—C24—C26	66.1 (2)
C11—C9—C13—C14	0.1 (2)	C20—C19—C24—C26	−113.99 (19)
C15—C9—C13—C14	178.85 (13)	C18—C19—C24—C27	−54.5 (2)
C7—C8—C13—C9	−179.48 (15)	C20—C19—C24—C27	125.45 (17)
C7—C8—C13—C14	−0.5 (2)	O30—S29—C33—F36	58.93 (13)
C12—N10—C14—C5	−179.72 (14)	O32—S29—C33—F36	−61.49 (13)
C28—N10—C14—C5	−1.3 (2)	O31—S29—C33—F36	178.76 (11)
C12—N10—C14—C13	−0.1 (2)	O30—S29—C33—F34	−61.59 (13)
C28—N10—C14—C13	178.38 (13)	O32—S29—C33—F34	177.99 (12)
C6—C5—C14—N10	179.41 (15)	O31—S29—C33—F34	58.23 (13)
C6—C5—C14—C13	−0.2 (2)	O30—S29—C33—F35	177.92 (11)
C9—C13—C14—N10	−0.1 (2)	O32—S29—C33—F35	57.51 (12)
C8—C13—C14—N10	−179.12 (13)	O31—S29—C33—F35	−62.25 (13)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C2—H2···O32	0.95	2.58	3.478 (2)	158
C4—H4···O31^{i}	0.95	2.35	3.285 (2)	168
C5—H5···O30^{ii}	0.95	2.45	3.330 (2)	155
C6—H6···O32^{ii}	0.95	2.42	3.265 (2)	148
C7—H7···F35^{iii}	0.95	2.46	3.264 (2)	143
C23—H23···F36^{iv}	0.95	2.52	3.208 (2)	130
C28—H28A···O32^{v}	0.98	2.42	3.251 (2)	142

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

C—H···π interactions (Å, °).

X	H	J	H···J	*X*···J	X-H···J
C26	H26B	*Cg*4^{vi}	2.875 (2)	3.579 (2)	129.46

Symmetry codes: (vi) 1 − *x*, 1 − *y*, 1 − *z*.
Notes: *Cg*4 is the centroid of the C19–C23 ring.

Fig. 1

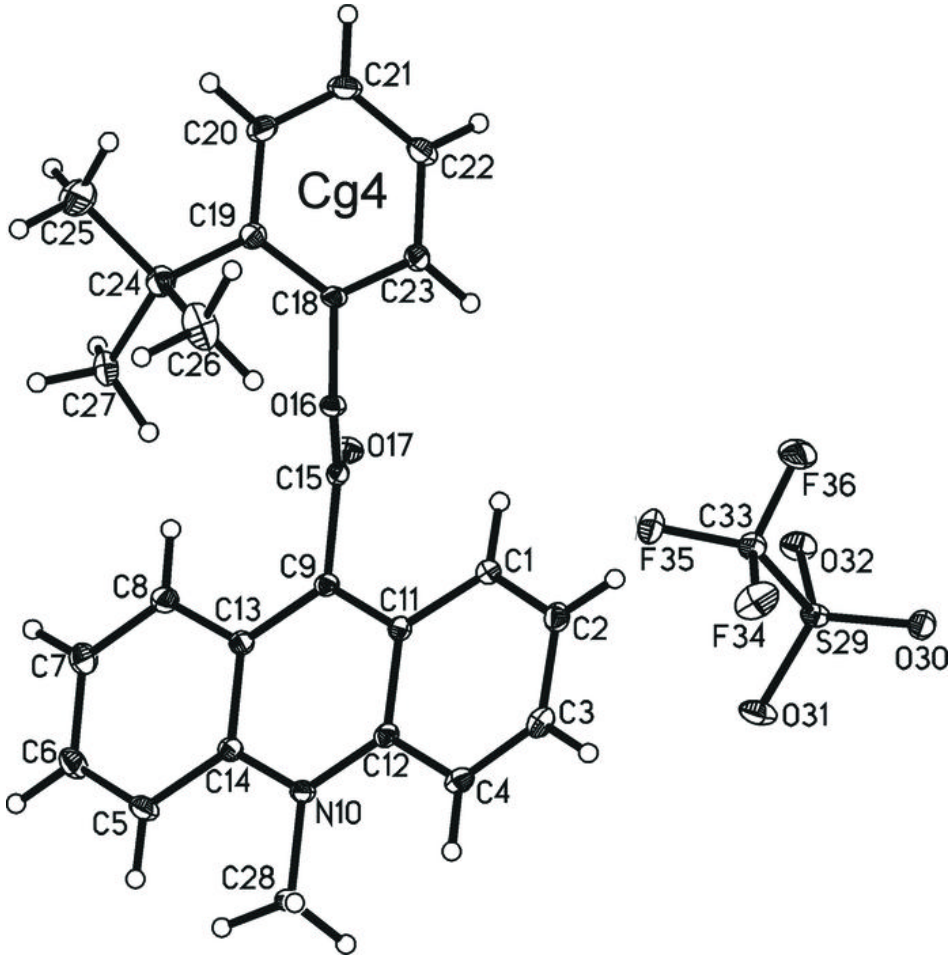

C21
C20
C22
Cg4
C19
C25
C24
C18
C23
C26
C27
O16
O17
C15
F36
C33
F35
O32
C9
C1
C8
C13
C11
C2
F34
S29
O30
C7
C3
O31
C6
C14
C12
C4
C5
N10
C28

Fig. 2

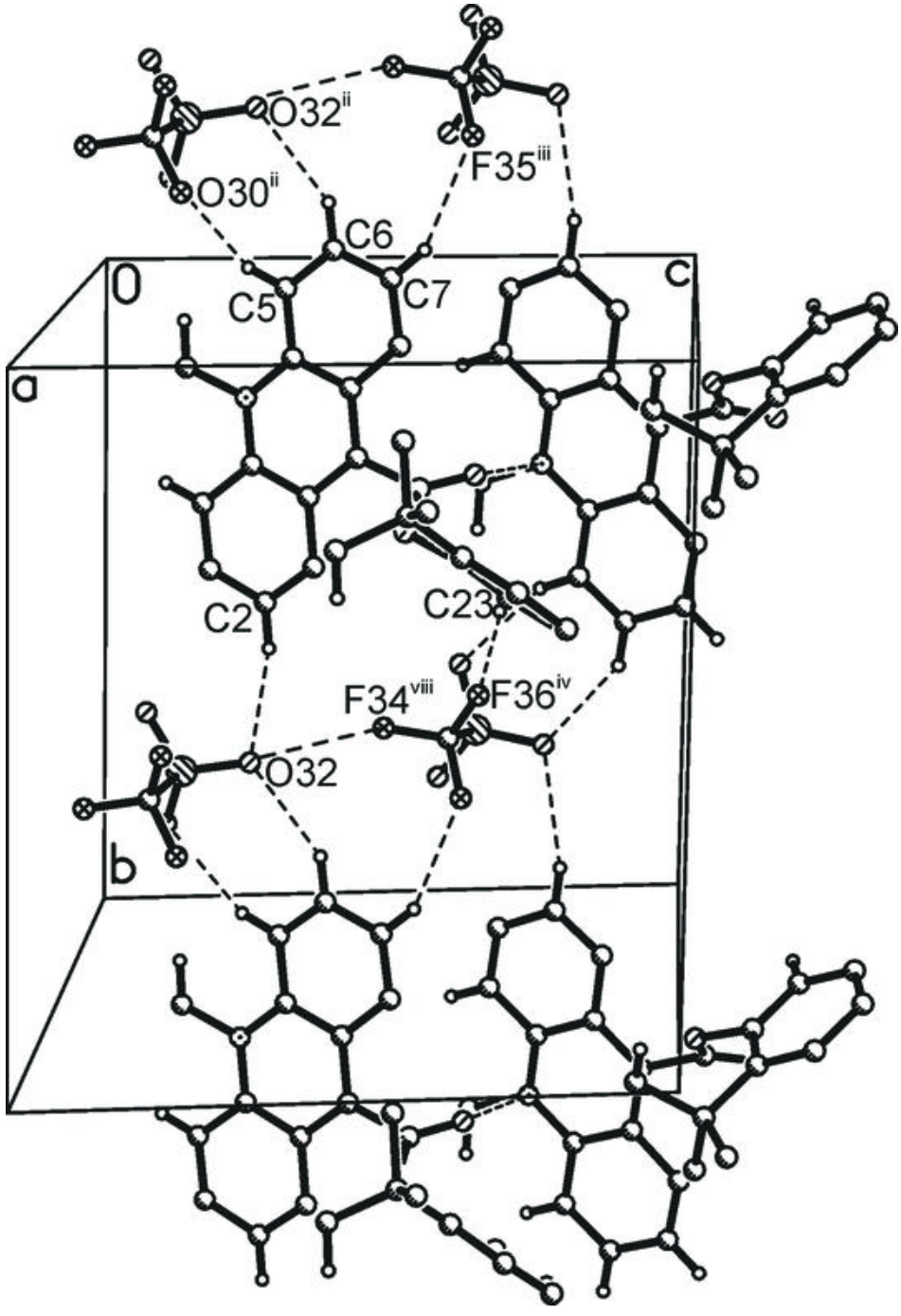


Fig. 3