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9-(2,6-Difluorophenoxycarbonyl)-10-methylacridinium trifluoromethanesulfonate and its precursor 2,6-difluorophenyl acridine-9-carboxylate: C—H···O, C—F···π, S—O···π and π–π stacking interactions

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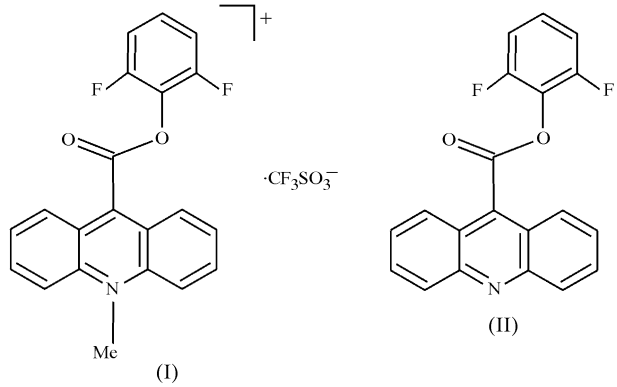
The title compounds, $C_{21}H_{14}F_2NO_2^+\cdot CF_3SO_3^-$, (I), and $C_{20}H_{11}F_2NO_2$, (II), form monoclinic and triclinic crystals, respectively. Adjacent cations of (I) are oriented in a 'head-to-tail' manner and are linked to one another *via* networks of C—H···O, C—F···π, S—O···π and multidirectional π–π interactions. Adjacent molecules of (II) are also arranged in a 'head-to-tail' manner and are linked *via* networks of C—H···O and multidirectional π–π interactions. The mean planes of the acridine moieties lie parallel in the lattices of both compounds. The benzene rings are also parallel. However, the acridine and difluorophenyl rings are mutually oriented at an angle of 17.3 (2)° in (I) and 5.8 (2)° in (II). This mutual orientation in various phenyl acridine-9-carboxylates and related compounds is strongly influenced by the nature of the substituents on the phenyl fragment.

Comment

Most commercially available immunoassay tests utilizing chemiluminescence employ derivatives of acridine-9-carboxylic acid (Weeks *et al.*, 1986; Rongen *et al.*, 1994; Razawi & McCapra, 2000*a*,*b*; Smith *et al.*, 2000). Sensitivities at the attomole level are available with this method, which makes acridine-based labels more profitable than standard radioisotopic techniques (*e.g.* ^{125}I or ^{3}H) (Zomer & Jacquemijns, 2001). Among the most frequently used of these derivatives are the phenyl esters of the 10-methylacridinium-9-carboxylic acid cation (Dodeigne *et al.*, 2000), although other compounds, like hydroxamic or sulfohydroxamic esters, have been tested in order to develop new assay options (Renotte *et al.*, 2000). These compounds react with H_2O_2 in alkaline media to produce molecules of electronically excited 10-methyl-9-acridinone (Rak *et al.*, 1999), which emits light. The intensity of the light is related directly to the concentration of the entity assayed and this is the foundation for the analytical application of chemiluminescence. Nevertheless, the use of acridinium esters for labelling biomolecules entails certain disadvantages. Although their chemiluminescence efficiency in aqueous solutions is relatively high (up to 10%), they are not very stable (Rak *et al.*, 1999; Razawi & McCapra, 2000*a*,*b*); they can react relatively fast with OH^-, which attacks the C atom in position 9. Many attempts have been made to enhance their resistance to hydrolysis, since this reaction competes with the chemiluminescence pathway in alkaline media, yielding a non-luminescent product, namely the non-excited 10-methyl-9-acridinone (Hammond *et al.*, 1991). Since the phenyl fragment is removed during oxidation of phenyl 10-methylacridinium-9-carboxylates, it is thought that the phenyl ring substituents exert the greatest influence on the ability to chemiluminesce and on the properties of this group of compounds (Sato, 1996; Rak *et al.*, 1999). Wilson *et al.* (2001) noted that reduction of 9-(2,6-difluorophenoxycarbonyl)-10-methylacridinium (the title cation) yielded the corresponding ester of acridan, which is not susceptible to nucleophilic substitution (and thus hydrolysis). In this case, chemiluminescence was triggered by the cathodic oxidation of the acridan, which regenerates to the original acridinium salt and decomposes to the electronically excited 10-methyl-9-acridinone (a light emitter). It can thus be expected that the presence of F atoms in the phenyl ring will improve the resistance of such compounds to alkaline hydrolysis and enhance their susceptibility to oxidation and their chemiluminescence ability. These were the premises for undertaking investigations on the title chemiluminogens, namely 9-(2,6-difluorophenoxycarbonyl)-10-methylacridinium trifluoromethanesulfonate, (I), and 2,6-difluorophenyl acridine-9-carboxylate, (II).



In this paper, we present the results of our crystal structure investigations, which were paralleled by laboratory studies on the relationship between the structural and chemiluminogenic properties of this group of compounds.

Parameters characterizing the geometry of the central ring of the acridine moiety and the carboxyl fragment in (I) are given in Table 1. The acridine moiety, with an average deviation from planarity for its constituent atoms of 0.015 Å, and

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the phenyl ring in (I) are mutually oriented at an angle of 17.3 (2)° (denoted by δ, this is the angle between the mean planes delineated by all the non-H atoms of the acridine and phenyl moieties) (Fig. 1). The carboxyl group is twisted at an angle of 67.6 (2)° relative to the acridine skeleton (denoted by ε, this is the angle between the mean planes delineated by all the non-H atoms of the acridine moiety and atoms C15, O16 and O17). The H atoms of the methyl group are disordered over two orientations, twisted through 60° with respect to one another, each with an occupancy of 0.5.

In the crystalline phase, two adjacent cations of (I), arranged in a 'head-to-tail' manner and related by a centre of symmetry, are linked *via* two C—H···O interactions to yield dimers, which are, in turn, linked to anions *via* C—H···O interactions to form strips (Fig. 2 and Table 2). These strips are linked by networks of two types of C—H···O, C—F···π, S—O···π and π–π transverse interactions involving F atoms, O atoms from the anion, and the acridine or phenyl rings (Fig. 2, and Tables 3 and 4). This variety of interactions, rare in other acridine derivatives, accounts for the stability of the crystalline phase of (I).

Parameters characterizing the geometry of the central ring of the acridine moiety and the carboxyl fragment in (II) are given in Table 5. Angle δ between the acridine (average deviation from planarity for its constituent atoms = 0.003 Å) and phenyl moieties in (II) is 5.8 (2)° (Fig. 3). The carboxyl group in (II) is twisted at an angle ε = 78.8 (2)° relative to the acridine skeleton. Comparison of the δ and ε angles for (I) and (II) yields an inverse relationship.

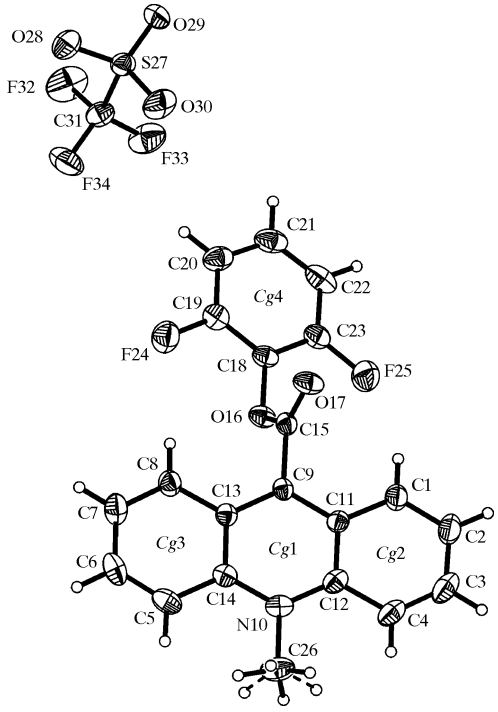

Figure 1
The molecular structure of (I), showing the atom-labelling scheme and 25% probability displacement ellipsoids. H atoms are shown as small spheres of arbitrary radii.

Arranged in a 'head-to-tail' manner and related by a centre of symmetry, two adjacent molecules of (II) are linked *via* two C—H···O interactions [as in (I)] to produce dimers (Fig. 4 and Table 6). These are further linked by a network of multidirectional π–π interactions involving the central acridine ring, one of the lateral acridine rings and the phenyl ring (Fig. 4 and Table 7), all of which makes for a stable molecular crystal lattice.

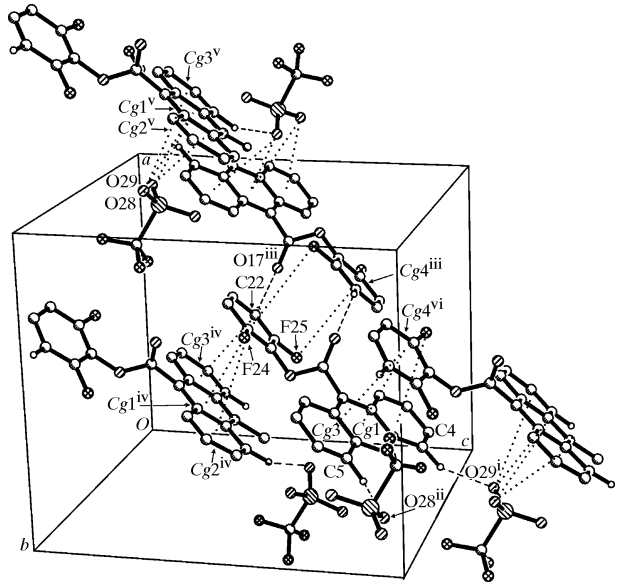

Figure 2
The arrangement of the ions of (I) in the unit cell. The C—H···O interactions are represented by dashed lines, and the Y—X···π and π–π interactions by dotted lines. H atoms not involved in C—H···O interactions have been omitted. [Symmetry codes: (i) $x-1, y, 1+z$; (ii) $1-x, 1-y, 1-z$; (iii) $1-x, -y, 1-z$; (iv) $x, \frac{1}{2}-y, z-\frac{1}{2}$; (v) $1+x, \frac{1}{2}-y, z-\frac{1}{2}$; (vi) $x, \frac{1}{2}-y, z-\frac{1}{2}$.]

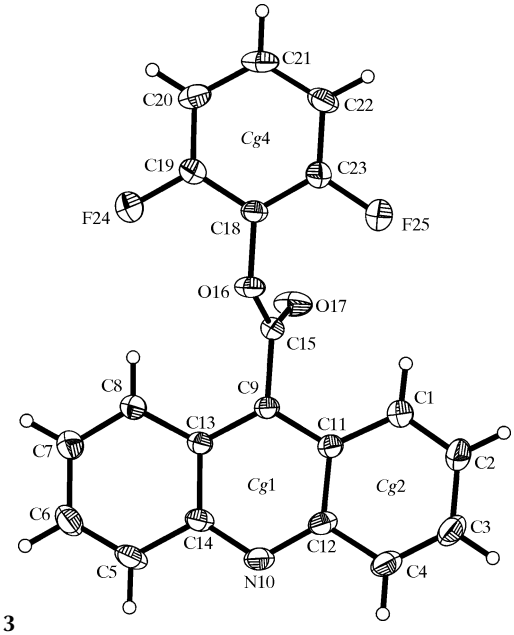

Figure 3
The molecular structure of (II), showing the atom-labelling scheme and 25% probability displacement ellipsoids. H atoms are shown as small spheres of arbitrary radii.

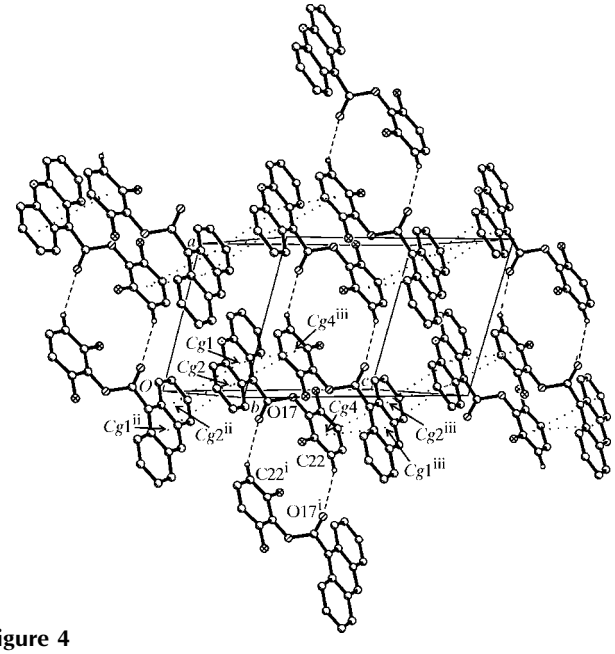

Figure 4
The arrangement of the molecules of (II) in the unit cell. The C—H···O interactions are represented by dashed lines and the π–π interactions by dotted lines. H atoms not involved in C—H···O interactions have been omitted. [Symmetry codes: (i) $-1-x, 1-y, 1-z$; (ii) $-x, 1-y, -z$; (iii) $-x, 1-y, 1-z$.]

The geometry of the molecules in the crystalline phase is the product of intramolecular forces and intermolecular interactions. If some fragments are rigid, as with the acridine, phenyl and carboxyl (O═C—O—) moieties in compounds (I) and (II), the molecules can exist in a variety of structures as a result of rotation around the single bonds, in the present case C9—C15 and O16—C18. This provides the opportunity to investigate the influence of the structure of the molecular fragments on their mutual arrangement and on the structure of the whole molecules. Table 8 lists the angles δ, reflecting the mutual arrangement of the acridine and phenyl rings, and the angles ε, representing the relative arrangement of the acridine ring and the carboxyl (or vinyl) group, for a series of phenyl acridine-9-carboxylates, their relevant 10-methylacridinium cations and related compounds. The angles δ are the largest in two known structures of styrylacridines. They are quite large if bulky groups (Et) or atoms (Br, I) are *ortho*-substituted in the phenyl ring of phenyl acridine-9-carboxylates. As far as the halogen-disubstituted compounds are concerned, the angle δ in the fluoro derivatives of phenyl acridine-9-carboxylates and their 10-methylacridinium cations is relatively small and increases with the size of the halogen atom. All of the angles ε listed in Table 8 are larger than 50° and are not correlated with either the angles δ or the size and number of the substituents in the phenyl fragment. The angles ε are most probably the outcome of the most efficient crystal packing. A property which does not arise directly from the δ values listed in Table 8, but which emerges from a meticulous analysis of crystal phase structures, is that there is a relatively large number and variety of intermolecular interactions in the difluoro derivatives, which may be a consequence of the nearly parallel mutual orientation of the acridine and phenyl rings.

Experimental

Compound (II) was prepared by heating anhydrous acridine-9-carboxylic acid with a tenfold molar excess of thionyl chloride, followed by esterification of the resulting acid chloride with an equimolar quantity of 2,6-difluorophenol (Sato, 1996). The reaction was carried out in anhydrous dichloromethane in the presence of triethylamine (1.5 molar excess) and a catalytic quantity of 4-(dimethylamino)pyridine. The crude product was subsequently washed with dilute HCl, $NaHCO_3$ and saturated saline, and then purified chromatographically with silica gel as the stationary phase and cyclohexane–ethyl acetate (1:1 *v*/*v*) as the mobile phase (yield 79%). Analysis calculated for $C_{22}H_{14}F_5NO_5S$: C 71.6, H 3.3, N 4.2%; found: C 71.5, H 3.3, N 25.1%. Yellow crystals of (II) suitable for X-ray analysis were grown from cyclohexane (m.p. 454–455 K). Compound (I) was synthesized by treating compound (II) 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 4 h). The crude salt was purified by repeated precipitation from an ethanol–diethyl ether (1:20 *v*/*v*) solution (yield 86%). Pale-yellow crystals of (I) suitable for X-ray investigations were grown from absolute ethanol (m.p. 504–506 K).

Compound (I)

Crystal data

$C_{21}H_{14}F_2NO_2^+\cdot CF_3O_3S^-$
M_r = 499.40
Monoclinic, $P2_1/c$
a = 11.382 (2) Å
b = 14.048 (3) Å
c = 13.162 (3) Å
β = 91.16 (3)°
V = 2104.1 (8) Å^3
Z = 4
D_x = 1.576 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 50 reflections
θ = 2.1–25.0°
μ = 0.23 mm^{-1}
T = 290 (2) K
Prism, yellow
0.5 × 0.3 × 0.2 mm

Data collection

Kuma KM-4 diffractometer
$\theta/2\theta$ scans
3874 measured reflections
3700 independent reflections
1760 reflections with $I > 2\sigma(I)$
R_{int} = 0.029
θ_{max} = 25.0°
$h = -13 \rightarrow 13$
$k = 0 \rightarrow 16$
$l = 0 \rightarrow 15$
3 standard reflections every 200 reflections
intensity decay: 4.5%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.048
$wR(F^2)$ = 0.144
S = 0.97
3700 reflections
309 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0693P)^2 + 0.9523P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.27 e Å^{-3}
$\Delta\rho_{min}$ = −0.32 e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 1997)
Extinction coefficient: 0.0096 (9)

Table 1
Selected geometric parameters (Å, °) for (I).

N10—C12	1.367 (5)	O16—C18	1.401 (4)
N10—C14	1.363 (5)	C19—F24	1.336 (5)
C15—O16	1.355 (4)	C23—F25	1.352 (4)
C15—O17	1.179 (4)		
C9—C15—O16	111.9 (3)	C9—C15—O17	124.6 (3)
C11—C9—C15—O17	−64.2 (5)	C15—O16—C18—C19	−96.3 (4)

Table 2
Hydrogen-bond geometry (Å, °) for (I).

D—H···A	D—H	H···A	D···A	D—H···A
C4—H4···O29^{i}	0.93	2.54	3.225 (5)	131
C5—H5···O28^{ii}	0.93	2.59	3.417 (5)	148
C22—H22···O17^{iii}	0.93	2.52	3.316 (5)	144

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

Table 3
Y—X···π interactions in (I) (Å, °).

*Cg*1 is the centroid of ring C9/C11/C12/N10/C14/C13, *Cg*2 of ring C1–C4/C12/C11, *Cg*3 of ring C5–C8/C13/C14 and *Cg*4 of ring C18–C23.

Y—X	Cg	X···Cg	Y···Cg	Y—X···Cg
C19—F24	2^{iv}	3.874 (3)	4.109 (4)	90.5 (2)
C23—F25	4^{iii}	3.977 (3)	4.383 (4)	98.4 (2)
S27—O28	1^{v}	3.511 (3)	3.708 (2)	86.74 (13)
S27—O28	2^{v}	3.374 (3)	4.080 (2)	110.17 (14)
S27—O29	1^{v}	3.569 (4)	3.708 (2)	84.22 (14)
S27—O29	3^{v}	3.835 (4)	4.740 (2)	121.64 (16)

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

Table 4
π–π interactions in (I) (Å, °).

The centroids are as in Table 3. The dihedral angle is between the *CgI* and *CgJ* planes and the interplanar distance is the perpendicular distance of *CgI* from ring *J* and the offset is the perpendicular distance of ring *I* from ring *J*.

CgI	*CgJ*	*Cg*···*Cg*	Dihedral angle	Interplanar distance	Offset
1	4^{vi}	3.620 (2)	7.8	3.424 (2)	1.841 (2)
3	4^{vi}	3.940 (3)	6.4	3.482 (3)	1.003 (3)
4	1^{iv}	3.620 (2)	7.8	3.530 (2)	1.037 (2)
4	3^{iv}	3.940 (3)	6.4	3.527 (3)	1.570 (3)

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

Compound (II)

Crystal data

$C_{20}H_{11}F_2NO_2$
M_r = 335.30
Triclinic, $P\overline{1}$
a = 8.306 (2) Å
b = 9.057 (2) Å
c = 11.423 (2) Å
α = 69.94 (3)°
β = 77.80 (3)°
γ = 72.16 (3)°
V = 762.9 (3) Å^3
Z = 2
D_x = 1.460 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 50 reflections
θ = 2.5–25.0°
μ = 0.11 mm^{-1}
T = 290 (2) K
Prism, yellow
0.4 × 0.3 × 0.2 mm

Data collection

Kuma KM-4 diffractometer
$\theta/2\theta$ scans
2875 measured reflections
2681 independent reflections
1314 reflections with $I > 2\sigma(I)$
R_{int} = 0.023; θ_{max} = 25.0°
$h = -9 \rightarrow 9$
$k = -10 \rightarrow 10$
$l = 0 \rightarrow 13$
3 standard reflections
every 200 reflections
intensity decay: 1.0%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.129
S = 1.01
2681 reflections
227 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0607P)^2 + 0.1152P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.18 e Å^{-3}
$\Delta\rho_{min}$ = −0.16 e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.0025 (4)

Table 5
Selected geometric parameters (Å, °) for (II).

N10—C12	1.339 (3)	O16—C18	1.395 (2)
N10—C14	1.337 (3)	C19—F24	1.342 (3)
C15—O16	1.352 (3)	C23—F25	1.347 (3)
C15—O17	1.180 (3)		
C9—C15—O16	110.6 (2)	C9—C15—O17	125.9 (2)
C11—C9—C15—O17	78.1 (3)	C15—O16—C18—C19	104.9 (3)

Table 6
Hydrogen-bond geometry (Å, °) for (II).

D—H···A	D—H	H···A	D···A	D—H···A
C22—H22···O17^{i}	0.93	2.55	3.395 (4)	151

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

Table 7
π–π interactions in (II) (Å, °).

The centroids are as in Table 3. The dihedral angle is between the *CgI* and *CgJ* planes and the interplanar distance is the perpendicular distance of *CgI* from ring *J* and the offset is the perpendicular distance of ring *I* from ring *J*.

CgI	*CgJ*	*Cg*···*Cg*	Dihedral angle	Interplanar distance	Offset
1	2^{ii}	3.912 (2)	1.1	3.459 (3)	1.472 (2)
1	4^{iii}	3.735 (2)	6.3	3.469 (3)	1.413 (2)
2	1^{ii}	3.912 (2)	1.1	3.427 (3)	1.740 (2)
2	2^{ii}	3.514 (2)	0.0	3.444 (3)	1.634 (2)
2	4^{iii}	3.787 (2)	6.3	3.406 (3)	1.472 (2)
4	1^{iii}	3.735 (2)	6.3	3.535 (3)	1.413 (2)
4	2^{iii}	3.787 (2)	6.3	3.527 (3)	1.740 (2)

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

Table 8
The angles between the acridine and phenyl rings (δ, °), and between the acridine ring and the carboxyl or vinyl fragment (ε, °), in substituted phenyl acridine-9-carboxylates, their relevant 10-methylacridinium cations and related compounds.

Compound	δ	ε	Reference
(I)	17.3 (2)	67.6 (2)	This work
(II)	5.8 (2)	78.8 (2)	This work
(III)	35.9 (2)	56.5 (2)	Meszko *et al.* (2002)
(IV)	30.2 (2)	57.9 (2)	To be published
(V)	62.1 (2)	67.3 (2)	Sikorski *et al.* (2005*a*)
(VI)	35.7 (2)	68.1 (2)	Sikorski *et al.* (2005*a*)
(VII)	9.3 (2)	77.2 (2)	Sikorski *et al.* (2005*b*)
(VIII)	41.2 (2)	51.0 (2)	Sikorski *et al.* (2005*d*)
(IX)	45.5 (2)	54.3 (2)	To be published
(X)	27.2 (2)	89.0 (2)	To be published
(XI)	33.4 (2)	62.0 (2)	Sikorski *et al.* (2005*b*)
(XII)	35.9 (3)	60.6 (3)	Sikorski *et al.* (2005*c*)
(XIII)	67.1 (2)	55.5 (2)	Sgarabotto *et al.* (1989)
(XIV)	73.7 (3)	69.9 (3)	Sgarabotto *et al.* (1989)

Compounds: (I) 9-(2,6-difluorophenoxycarbonyl)-10-methylacridinium trifluoromethanesulfonate; (II) 2,5-difluorophenyl acridine-9-carboxylate; (III) 2-methylphenyl 2-methoxyacridine-9-carboxylate; (IV) 2-methylphenyl acridine-9-carboxylate; (V) 2-ethylphenylacridine-9-carboxylate; (VI) 2,5-dimethylphenyl acridine-9-carboxylate; (VII) 2,5-dichlorophenyl acridine-9-carboxylate; (VIII) 2,5-dibromophenyl acridine-9-carboxylate; (IX) 2,5-diiodophenyl acridine-9-carboxylate; (X) 9-(2-methylphenoxycarbonyl)-10-methylacridinium trifluoromethanesulfonate; (XI) 9-(2,6-dichlorophenoxycarbonyl)-10-methylacridinium trifluoromethanesulfonate; (XII) 9-(2,6-dibromophenoxycarbonyl)-10-methylacridinium trifluoromethanesulfonate; (XIII) (*E*)-9-styrylacridine; (XIV) (*Z*)-9-(2,5-dimethylstyryl)acridine.

The methyl H atoms in (I) were located in difference Fourier syntheses and refined as a rigid rotating group, with C—H = 0.96 Å and $U_{iso}(H) = 1.5U_{eq}(C)$. These H atoms were assumed to have two unique disordered orientations with an occupancy factor of 0.5. All other H atoms in (I) and (II) were placed geometrically and refined using a riding model, with C—H distances of 0.93 Å and with $U_{iso}(H) = 1.2U_{eq}(C)$.

For both compounds, data collection: *KM-4 Software* (Kuma, 1989); cell refinement: *KM-4 Software*; data reduction: *KM-4 Software*; 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).

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: LN1184). Services for accessing these data are described at the back of the journal.

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