$\omega/2\theta$ scans
Absorption correction: empirical *via* ψ scans (*MolEN*; Fair, 1990)
$T_{\rm min} = 0.792$, $T_{\rm max} = 0.862$
3335 measured reflections
3022 independent reflections

$R_{\rm int} = 0.064$
$\theta_{\rm max} = 26.3°$
$h = 0 \rightarrow 13$
$k = 0 \rightarrow 15$
$l = -13 \rightarrow 13$
3 standard reflections every 250 reflections intensity decay: 1%

Refinement

Refinement on F
$R = 0.045$
$wR = 0.063$
$S = 2.15$
2432 reflections
144 parameters
H atoms riding
$w = 1/[\sigma(I)^2 + (0.04F^2)^2]^{1/2}$

$(\Delta/\sigma)_{\rm max} = 0.01$
$\Delta\rho_{\rm max} = 0.50$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.16$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for X-ray Crystallography* (Vol. IV)

Table 1. *Selected geometric parameters (Å, °)*

Cl1—P1	1.987 (1)	P2—N2	1.596 (2)
Cl2—P1	1.989 (1)	N2—C1	1.489 (4)
Cl3—P2	2.012 (1)	N2—C4	1.502 (3)
Cl4—P2	1.991 (1)	C1—C2	1.523 (5)
P1—O1	1.449 (2)	C1—C3	1.517 (5)
P1—N1	1.564 (3)	C4—C5	1.510 (5)
P2—N1	1.537 (3)	C4—C6	1.528 (5)
Cl1—P1—Cl2	102.03 (7)	Cl3—P2—N2	108.4 (1)
Cl1—P1—O1	110.8 (1)	Cl4—P2—N1	113.9 (1)
Cl1—P1—N1	103.8 (1)	Cl4—P2—N2	109.1 (1)
Cl2—P1—O1	109.7 (1)	N1—P2—N2	112.2 (1)
Cl2—P1—N1	106.5 (1)	P1—N1—P2	134.0 (2)
O1—P1—N1	122.1 (2)	C2—C1—C3	111.9 (3)
Cl3—P2—Cl4	100.75 (5)	C5—C4—C6	112.6 (3)
Cl3—P2—N1	111.8 (1)		
Cl1—P1—N1—P2	−140.2 (3)	N2—P2—N1—P1	−170.2 (2)
Cl2—P1—N1—P2	112.5 (3)	Cl3—P2—N2—C1	−56.0 (2)
O1—P1—N1—P2	−14.4 (4)	Cl3—P2—N2—C4	119.3 (2)
Cl3—P2—N1—P1	67.8 (3)	Cl4—P2—N2—C1	52.9 (2)
Cl4—P2—N1—P1	−45.6 (3)	Cl4—P2—N2—C4	−131.9 (2)

The title structure was solved by the Patterson method. Atoms H1 and H4 were obtained from the difference synthesis and refined isotropically. The other H-atom positions were calculated geometrically, with $U_{\rm cq}({\rm H}) = 1.3U_{\rm eq}$(parent atom). A riding model was used in the refinement.

Data collection: *MolEN* (Fair, 1990). Cell refinement: *MolEN*. Data reduction: *MolEN*. Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *MolEN*. Molecular graphics: *ORTEP*II (Johnson, 1976). Software used to prepare material for publication: *MolEN*.

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p-Toluidinium 2′-Hydroxy-3′-methoxy-5′-flavonesulfonate Hydrate

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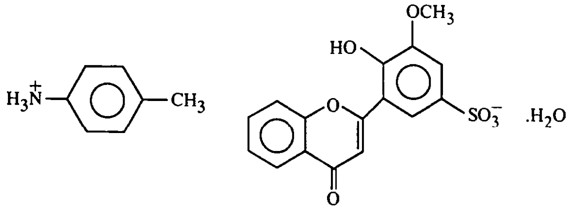
Abstract

The title complex, $C_7H_{10}N^+.C_{16}H_{11}O_7S^-.H_2O$, is a *p*-toluidinium hydrated salt of 2′-hydroxy-3′-methoxy-5′-flavonesulfonic acid [systematic name: 4-hydroxy-5-methoxy-3-(4-oxo-4*H*-[2]benzopyran-2-yl)benzenesulfonic acid]. The N atom of *p*-toluidine is protonated to give the cation. The self-assembly of the ionic counterparts is mediated by a water molecule of hydration.

Comment

The X-ray structure determination of the title compound, (I), was carried out as a preliminary study of interactions between sulfonated flavones and nucleic acid bases. *p*-Toluidine hydrochloride is a useful co-precipitating compound for sulfonic acids (Vogel, 1967). On the other hand, sulfonated compounds have proven to be effective co-precipitating agents for peptides and other biomolecules (Conroy & Lovrien, 1992).



(I)

The bond S21—O22 [1.472 (1) Å] is longer than S21—O24 and S21—O23 [1.432 (2) and 1.450 (2) Å, respectively] because the O22 atom is involved in two intermolecular hydrogen bonds (see below).

The flavone system is almost planar, as indicated by the value of the torsion angle O10—C9—C12—C17 [174.2 (2)°], which is equivalent to the values observed in the two independent molecules of 2′-hydroxyflavone [±170 (1)°; Seetharaman & Rajan, 1995]. This planarity is a result of stabilizing electrostatic interactions between atoms O18 and H8, and atoms O10 and H13, with very short O···H distances of 2.06 (3) and 2.27 (3) Å, respectively. In 2′,6′-dihydroxyflavone (Molins *et al.*, 1996), where an additional hydroxy group at position 6′ induces repulsive electrostatic and steric interactions between its O atom and the ring O atom, the inter-ring torsion angle is 110.4 (3)°. The other geometric parameters of the anion are equivalent to those observed in the cited compounds.

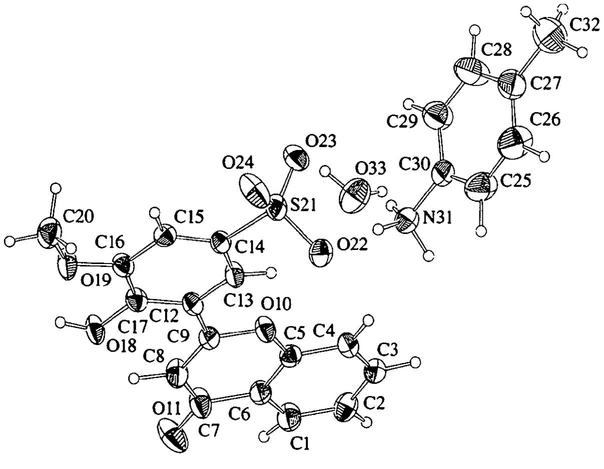


Fig. 1. *ORTEP*II (Johnson, 1976) drawing of the title compound, with displacement ellipsoids of 50% probability.

Bond lengths and valence angles in the *p*-toluidinium cation are in good agreement with those of other salts of *p*-toluidinium (Colapietro *et al.*, 1982).

The crystal packing is characterized by two two-dimensional networks of hydrogen bonds, which are located in planes parallel to the *ab* plane. At the level $z = 0$, flavone anions are bound into pairs through their respective carbonyl and hydroxy groups, O11 and O18. At the level $z = \frac{1}{2}$, the water molecule O33 is involved in three hydrogen bonds with atoms N31, O23 and O24 belonging to three different asymmetric units (Table 2). Atom N31 is also involved in two other symmetrical hydrogen bonds with O22 atoms from two different asymmetric units. The two-dimensional hydrogen-bonding networks cross-link to give a three-dimensional framework.

Experimental

The sulfoflavone (0.5 g) was dissolved in the minimum volume of boiling water. A saturated aqueous solution of *p*-toluidine hydrochloride (0.5 g) was added. After cooling, the white precipitate of the *p*-toluidinium salt was filtered off and recrystallized from hot water, giving, after 24 h, fine colourless needles.

Crystal data

$C_7H_{10}N^+.C_{16}H_{11}O_7S^-.H_2O$
$M_r = 473.48$
Monoclinic
$P2_1/a$
$a = 8.546\,(1)$ Å
$b = 17.865\,(2)$ Å
$c = 15.044\,(2)$ Å
$\beta = 98.14\,(2)°$
$V = 2273.7\,(5)$ Å^3
$Z = 4$
$D_x = 1.383$ Mg m^{-3}
$D_m = 1.36\,(2)$ Mg m^{-3}
D_m measured by flotation in benzene–chloroform

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 8$–$14°$
$\mu = 0.192$ mm^{-1}
$T = 293\,(2)$ K
Prismatic needle
$0.43 \times 0.32 \times 0.26$ mm
Colourless

Data collection

Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: none
5071 measured reflections
5071 independent reflections
3645 reflections with $I > 2\sigma(I)$

$\theta_{\max} = 29.93°$
$h = -12 \rightarrow 12$
$k = 0 \rightarrow 24$
$l = 0 \rightarrow 21$
2 standard reflections frequency: 60 min intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.043$
$wR(F^2) = 0.088$
$S = 1.003$
5071 reflections
358 parameters

$w = 1/[\sigma^2(F_o^2) + (0.0345P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.229$ e Å^{-3}
$\Delta\rho_{\min} = -0.236$ e Å^{-3}
Extinction correction: none

H atoms refined with $U = 1.2U_{eq}$ of the connected atom, with a length restraint of 1.00 ± 0.05 Å.

Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Selected geometric parameters (Å, °)*

C5—C6	1.392 (2)	C16—O19	1.362 (2)
C6—C7	1.468 (2)	C17—O18	1.348 (2)
C7—O11	1.241 (2)	O19—C20	1.423 (2)
C7—C8	1.423 (3)	S21—O24	1.432 (2)
C8—C9	1.360 (2)	S21—O23	1.450 (2)
C9—O10	1.370 (2)	S21—O22	1.472 (1)
C14—S21	1.770 (2)		
O11—C7—C8	123.6 (2)	O23—S21—O22	109.8 (1)
O11—C7—C6	119.3 (2)	O24—S21—C14	107.6 (1)
C5—O10—C9	121.12 (12)	O23—S21—C14	107.2 (1)
O24—S21—O23	114.5 (1)	O22—S21—C14	105.7 (1)
O24—S21—O22	111.6 (1)		
C8—C9—C12—C13	174.8 (2)	C15—C14—S21—O24	16.6 (2)
O10—C9—C12—C13	−6.0 (2)	C13—C14—S21—O23	72.2 (2)
C8—C9—C12—C17	−5.1 (3)	C15—C14—S21—O23	−107.1 (2)
O10—C9—C12—C17	174.2 (2)	C13—C14—S21—O22	−44.9 (2)
C17—C16—O19—C20	176.0 (2)	C15—C14—S21—O22	135.9 (1)
C13—C14—S21—O24	−164.2 (2)		

Table 2. *Hydrogen-bonding geometry (Å, °)*

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O18—H18···O11^{i}	0.92 (2)	1.79 (2)	2.658 (3)	155 (3)
N31—H311···O22^{ii}	1.01 (2)	1.82 (2)	2.820 (3)	174 (3)
N31—H312···O22	1.03 (2)	1.81 (2)	2.823 (3)	169 (3)
N31—H313···O33	0.95 (2)	1.80 (2)	2.726 (3)	166 (3)
O33—H331···O23^{iii}	0.92 (2)	1.91 (2)	2.812 (3)	165 (3)
O33—H332···O24^{iv}	0.97 (2)	1.80 (2)	2.762 (3)	176 (3)

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

Data collection: *CAD*-4 *Operations Manual* (Enraf–Nonius, 1977). Cell refinement: *CAD*-4 *Operations Manual*. Data reduction: *DATARED* (Pèpe, 1979). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1985). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *ORTEP*II (Johnson, 1976). Software used to prepare material for publication: *SHELXL*93.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: LN1030). Services for accessing these data are described at the back of the journal.

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Thiophene-2-carbaldehyde *N*-(2,4-Dinitrophenyl)-*N*-methylhydrazone

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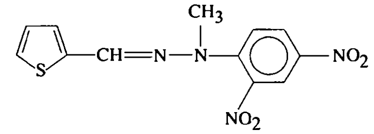
Abstract

Molecules of the title compound, $C_{12}H_{10}N_4O_4S$, are linked through intermolecular hydrogen bonds to form a dimeric structure. The crystal structure of the dimer is stabilized by two intermolecular hydrogen bonds of the C—H···O type. The C···O intermolecular contact distance is 3.339 (3) Å, and the C═N and N—N distances are 1.279 (2) and 1.371 (7) Å, respectively. One O atom of the 2-nitro group is disordered over two sites.

Comment

As a result of their anti-HIV and anticancer activities, the chemical and pharmacological properties of arylhydrazones have been studied extensively (Öcal & Kaban, 1998). In this paper, we report the structure of thiophene-2-carbaldehyde *N*-(2,4-dinitrophenyl)-*N*-methylhydrazone, (I).



(I)

An *ORTEP*II (Johnson, 1976) plot of the title molecule is shown in Fig. 1. The bond lengths and angles in the thiophene and dinitrophenyl rings are in agreement with related structures reported previously (Rodino-Pinera *et al.*, 1996; Lu *et al.*, 1996). The hydrazone moiety (C7—N3—N4—C8) is almost in the plane of the thiophene ring. The dihedral angle between the dinitrophenyl and thiophene rings is 23.79 (11)°, and these two planes form angles of 25.43 (10) and 2.86 (10)°, respectively, with the plane of the central hydrazone moiety. The 2-nitro group is tilted 49.8 (2)° from the plane of the phenyl ring, whereas the 4-nitro group is almost coplanar with the ring. However, of the two disordered components of O1, the first (O1*A*)

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