3-Benzyl-2,4,6-triphenylpyrylium tetrafluoroborate

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Key indicators

Single-crystal X-ray study
T = 120 K
Mean σ(C–C) = 0.003 Å
R factor = 0.055
wR factor = 0.142
Data-to-parameter ratio = 12.7

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

The central OC_5 ring in the cation of the title compound, $C_{30}H_{23}O^+ \cdot BF_4^-$, has considerable aromatic character but the pendant aromatic rings are not coplanar. The crystal structure comprises undulating layers of cations separated by BF_4^- anions with significant intermolecular interactions between them.

Comment

The structure of the title compound, (I) (Fig. 1 and Table 1), shows significant twisting of the pendant aromatic rings out of the central plane. The dihedral angles between the O1/C1–C5 ring and the C6–C11, C12–C17 and C25–C30 rings are 28.14 (10), 56.70 (11) and 83.44 (10)°, respectively. Within the central ring, which formally carries a positive charge, the two O—C distances are equal within experimental error and the C—C distances lie in the relatively narrow range 1.361 (3)–1.414 (3) Å. These observations strongly suggest substantial delocalization of π-electron density over this ring.


(I)
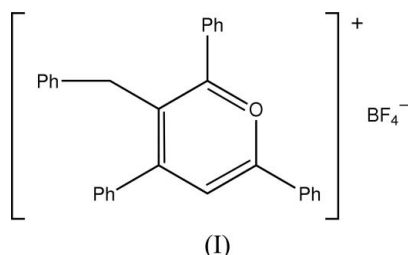
In the crystal structure there are a number of intermolecular interactions linking the ions. The primary interactions operating in the crystal structure are illustrated in Fig. 2. Here, B—F···H contacts are highlighted as golden dashed lines. The first contact occurs between the two components of the asymmetric unit so that C20—H20···F4 is 2.49 Å, C20···F4 is 3.355 (3) Å and the angle at H20 is 152°. The second F···H contact involves the C8[i] and F3 atoms so that C8[i]—H8[i]···F3 is 2.55 Å, C8[i]···F3 is 3.321 (3) Å and the angle at H8 is 139 Å [symmetry code: (i) $1 - x, 1 - y, -z$]. The remaining two F atoms serve to link two central O1/C1–C5 rings. The parameters associated with these interactions are B1—F1···ring centroid(O1/C1–C5) = 3.091 (2) Å and angle at F1 = 114.52 (15)°, and B1—F2···ring centroid(O1/C1–C5)[ii] = 3.080 (2) Å and angle at F2 = 118.59 (17)° [symmetry code: (ii) $1 - x, \frac{1}{2} + y, \frac{1}{2} - z$]. Formally, these might be considered as F···ring centroid(O1/C1–C5) interactions, but as seen in the B—F···ring centroid(O1/C1–C5) angles there is significant bending in the angles so that F1 approaches atoms O1 and C1 at 2.9508 (24) and 2.9766 (28) Å, respectively, with the other F1···C distances being greater than 3.3 Å. A similar situation pertains for the interactions involving atom F2. Thus, the
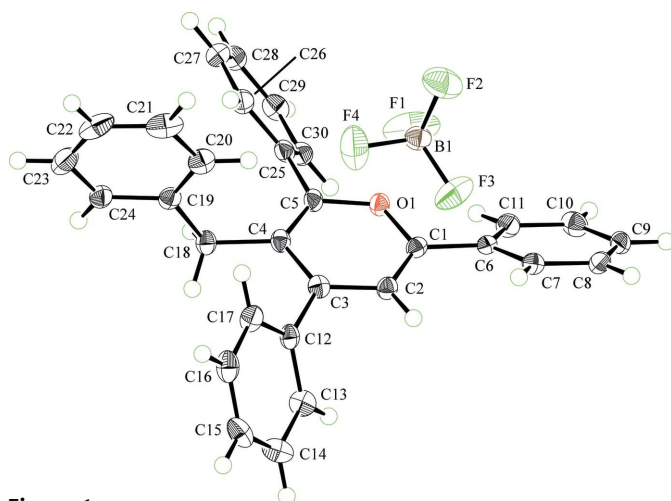
Figure 1
The asymmetric unit of (I) showing 50% probability displacement ellipsoids and the atom-numbering scheme.
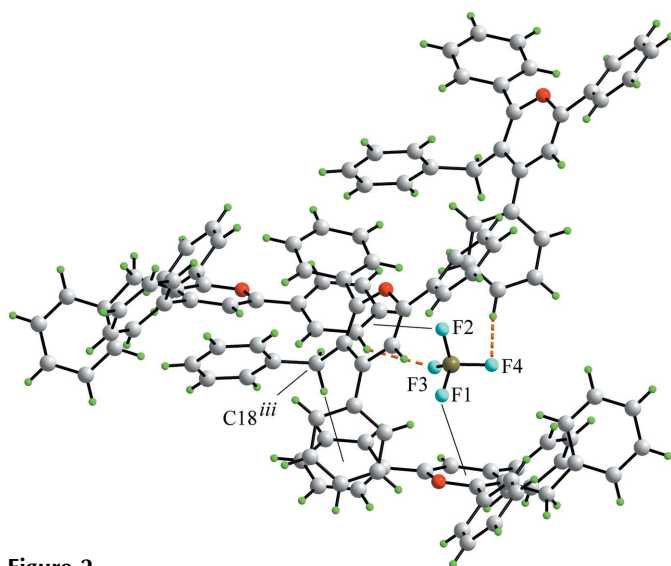
Figure 2
Environment about the BF_4^- anion in (I) (Crystal Impact, 2006). Color code: O (red), C (grey), B (brown) and H (green).

$F2 \cdots O1^{ii}$, $F2 \cdots C1^{ii}$ and $F2 \cdots C5^{ii}$ distances are 2.9270 (22), 3.1280 (28) and 3.1845 (26) Å, respectively, the remaining $F2 \cdots C^{ii}$ distances being greater than 3.5 Å. The only other significant intermolecular contact in the structure of (I) is also illustrated in Fig. 2, i.e. a $C18^{iii}$—$H18A^{iii} \cdots$ring centroid(C6–C11) contact with an $H18A^{iii} \cdots$ring centroid distance of 2.98 Å and an angle of 152° at the $H18A^{iii}$ atom. The global crystal structure may be described as comprising undulating layers of cations interspersed with anions (see Fig. 3).

Experimental

The title compound was isolated as a by-product in the preparation of 2-(4-hyroxyphenyl)-4,6-diphenylpyrylium tetrafluoroborate from 1,3-diphenylpropenone and 4-hydroxyphenylethanone in the presence of boron trifluoride etherate (Aliaga et al., 1997). The two products were separated by fractional crystallization from acetic acid. The title compound, recrystallized from AcOH, had melting point and spec-
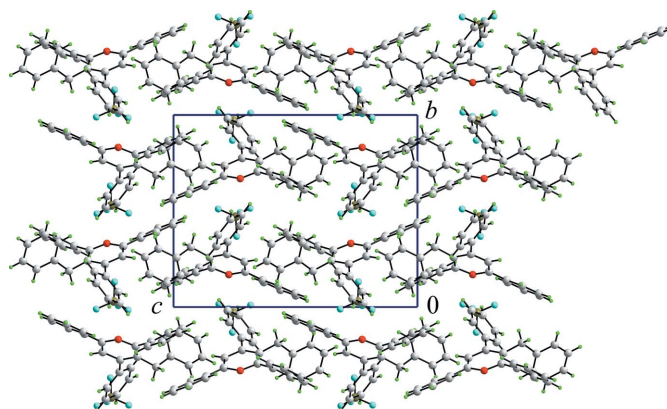
Figure 3
Packing diagram for (I), viewed approximately down the a axis (Crystal Impact, 2006). Color code as for Fig. 2.

troscopic properties in agreement with literature values (Marton et al., 1999).

Crystal data

$C_{30}H_{23}O^+ \cdot BF_4^-$	$Z = 4$
$M_r = 486.29$	$D_x = 1.374$ Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation
$a = 10.6170$ (3) Å	$\mu = 0.10$ mm^{-1}
$b = 13.2065$ (3) Å	$T = 120$ (2) K
$c = 17.1985$ (5) Å	Block, pale yellow
$\beta = 102.784$ (1)°	$0.24 \times 0.10 \times 0.08$ mm
$V = 2351.68$ (11) Å^3	

Data collection

Bruker–Nonius KappaCCD diffractometer	25644 measured reflections
φ and ω scans	4136 independent reflections
Absorption correction: multi-scan (SADABS; Sheldrick, 2003)	3347 reflections with $I > 2\sigma(I)$
$T_{min} = 0.834$, $T_{max} = 1$	$R_{int} = 0.042$
	$\theta_{max} = 25.0°$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0667P)^2$
$R[F^2 > 2\sigma(F^2)] = 0.055$	$+ 2.1496P]$
$wR(F^2) = 0.142$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.05$	$(\Delta/\sigma)_{max} < 0.001$
4136 reflections	$\Delta\rho_{max} = 0.80$ e Å^{-3}
325 parameters	$\Delta\rho_{min} = -0.40$ e Å^{-3}
H-atom parameters constrained	

Table 1
Selected geometric parameters (Å, °).

O1—C1	1.341 (3)	C2—C3	1.397 (3)
O1—C5	1.350 (3)	C3—C4	1.414 (3)
C1—C2	1.361 (3)	C4—C5	1.381 (3)
C1—O1—C5	122.38 (17)		

All H atoms were allowed to ride on their parent atoms in the riding-model approximation at C—H distances of 0.95–0.99 Å, and with $U_{iso}(H) = 1.2U_{eq}(C)$. Evidence of some disorder in the position of the BF_4^- anion can be noted from Fig. 1. However, multiple positions could not be resolved.

Data collection: COLLECT (Hooft, 1998); cell refinement: DENZO (Otwinowski & Minor, 1997) and COLLECT; data reduc-

tion: *DENZO* and *COLLECT*; program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPII* (Johnson, 1976) and *DIAMOND* (Crystal Impact, 2006); software used to prepare material for publication: *SHELXL97*.

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