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2′,6′-Dimethoxyflavone–bromoacetic acid (1/1)

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.015 Å
R factor = 0.075
wR factor = 0.249
Data-to-parameter ratio = 11.5

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

In the title compound, $C_{17}H_{14}O_4{\cdot}C_2H_3BrO_2$, pairs of molecules are linked *via* a hydrogen bond involving the carbonyl group of the flavone (2-phenyl-4*H*-1-benzopyran-4-one) and the carboxyl group of bromoacetic acid. This acid is in the antiplanar Br—C—C═O conformation.

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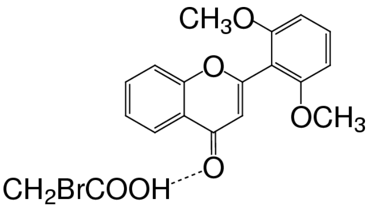
Comment

2′,6′-Dimethoxyflavone is a synthetic flavone which gives inclusion compounds with alkyl carboxylic acids: acetic (Wallet *et al.*, 1989), propionic (Tinant *et al.*, 1991) which crystallize in space group $P\overline{1}$, and chloroacetic and trichloroacetic acids (Espinosa *et al.*, 1999) which crystallize in non-centrosymmetric space groups. We were interested in knowing whether the presence of a haloacetic acid would favour the crystallization in a non-centrosymmetric space group. The substitution of one halogen by another had been reported as not altering the existing packing of crystals (Kálmán *et al.*, 1993; Wallet *et al.*, 2000). The space group is of prime importance for the phenomenon of second harmonic generation. 2′,6′-Dimethoxyflavone–acid complexes may provide the possibility to get acid molecules in different conformations from those given by pure acid crystals. Earlier examples are given by complexes with formic acid (Tinant *et al.*, 1991) and 2,6-dimethoxybenzoic acid (Wallet *et al.*, 1998). However, the control of the structure of solids *via* hydrogen-bonding interactions is known to be difficult (Desiraju, 1989).



(I)

The molecular structure of the title compound, (I), is shown in Fig. 1. The torsion angle O1—C2—C1′—C6′ in the flavone is −66.5 (11)°. The backbone of the bromoacetic acid is almost planar, Br1—C11—C12—O122 = −175.8 (9)°. Two forms of crystal structure have been reported for bromoacetic acid (Vor der Bruck & Leiserowitz, 1975). In (I), the acid molecule shows the same conformation as in the pure acid crystals (synplanar). The difference is that the bromoacetic acid is in the *anti* Br—C—C═O conformation (*syn* in the two forms of the pure acid). In addition to the O121—H121···O4 hydrogen bond (Table 1) there is a short contact C3—H3···O122, which exists in the complexes with acetic and propionic acids (space group $P\overline{1}$) but does not exist in the complexes with chloro-

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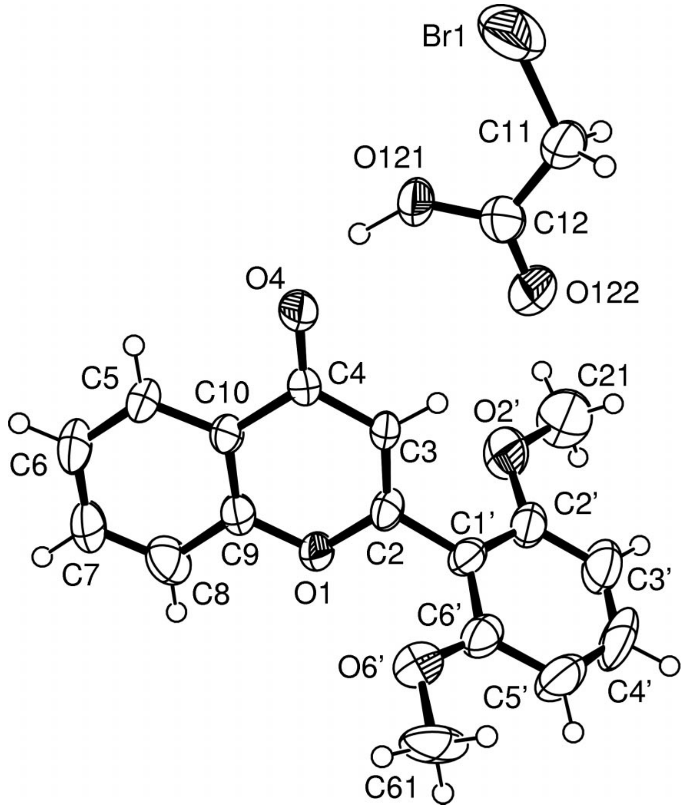

Figure 1
View of the title complex showing the labelling of the non-H atoms. Displacement ellipsoids are shown at the 50% probability level.

acetic and trichloroacetic acids which crystallize in non-centrosymmetric space groups. Thus, a skewed non-planar arrangement of the carboxyl group to the benzopyrone moiety of flavone seems to be a driving force leading to non-centrosymmetric structure.

Experimental

The synthesis of 2′,6′-dimethoxyflavone has been previously reported (Wallet *et al.*, 1989). Bromoacetic acid was from Aldrich. Crystals of (I) were obtained by slow evaporation of an ethanol solution of the stoichiometric mixture.

Crystal data

$C_{17}H_{14}O_4 \cdot C_2H_3BrO_2$
$M_r = 421.24$
Triclinic, $P\overline{1}$
$a = 7.663$ (1) Å
$b = 9.693$ (1) Å
$c = 12.877$ (1) Å
$\alpha = 86.56$ (1)°
$\beta = 78.56$ (1)°
$\gamma = 76.30$ (2)°
$V = 910.71$ (17) Å^3

$Z = 2$
$D_x = 1.536$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
$\theta = 8.5$–12.5°
$\mu = 2.29$ mm^{-1}
$T = 293$ (2) K
Prism, pale yellow
0.66 × 0.30 × 0.04 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Non-profiled $\omega/2\theta$ scans
Absorption correction: ψ scan (North *et al.*, 1968)
$T_{\min} = 0.440$, $T_{\max} = 0.913$
2993 measured reflections
2760 independent reflections
1072 reflections with $I > 2\sigma(I)$

$R_{\rm int} = 0.065$
$\theta_{\max} = 23.7°$
$h = 0 \rightarrow 8$
$k = -10 \rightarrow 10$
$l = -14 \rightarrow 14$
3 standard reflections
frequency: 60 min
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.075$
$wR(F^2) = 0.249$
$S = 0.96$
2760 reflections
240 parameters

H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.1319P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.006$
$\Delta\rho_{\max} = 0.65$ e Å^{-3}
$\Delta\rho_{\min} = -0.66$ e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O121—H121···O4	1.09 (14)	1.51 (14)	2.534 (9)	152 (11)
C3—H3···O122	0.93	2.44	3.305 (12)	156

The positional parameters of the hydroxyl H121 atom were refined. All the other H atoms were introduced in calculated positions and constrained to ride on their bonded atom.

Data collection: *CAD*-4 *EXPRESS* (Enraf–Nonius, 1994); cell refinement: *CAD*-4 *EXPRESS*; data reduction: *XCAD*4 (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *PLUTON*93 (Spek, 1993).

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