

2-[4-(Diethylamino)-2-hydroxybenzoyl]-  
3,4,5,6-tetrafluorobenzoic acidFan-Yong Yan,<sup>a\*</sup> Feng-Yan Ge,<sup>a</sup>  
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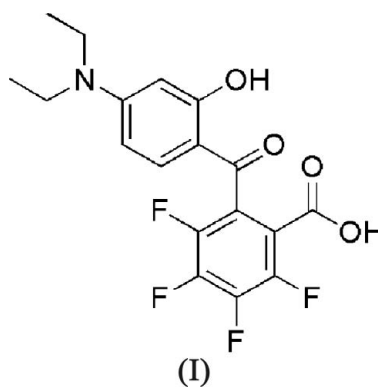
## Key indicators

Single-crystal X-ray study  
 $T = 294$  K  
Mean  $\sigma(\text{C}-\text{C}) = 0.004$  Å  
 $R$  factor = 0.045  
 $wR$  factor = 0.131  
Data-to-parameter ratio = 14.4For details of how these key indicators were  
automatically derived from the article, see  
<http://journals.iucr.org/e>.The title compound,  $\text{C}_{18}\text{H}_{15}\text{F}_4\text{NO}_4$ , is an intermediate in the  
synthesis of tetrafluororhodamine. The dihedral angle  
between the benzene rings is  $73.5(3)^\circ$ . There is an intra-  
molecular  $\text{O}-\text{H}\cdots\text{O}$  hydrogen bond. In addition, the  
structure is stabilized by intermolecular  $\text{O}-\text{H}\cdots\text{O}$  hydrogen  
bonds.

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## Comment

The title compound, (I), is an intermediate in the synthesis of  
tetrafluororhodamine (Lee *et al.*, 2002). Luo *et al.* (1994)  
synthesized 2-carboxyl-4'-diethylamino-2'-hydroxybenzo-  
phenone with 3-diethylaminophenol and phthalic anhydride  
as reactants with the same reaction mechanism as for the title  
compound. In spite of these accounts of its synthesis, no  
crystal structure determination of the title compound has been  
reported. We therefore report the crystal structure in this  
work.The dihedral angle between the benzene rings is  $73.5(3)^\circ$ .  
There is an intramolecular  $\text{O}-\text{H}\cdots\text{O}$  hydrogen bond (Table 1).  
In addition, the structure is stabilized by intermolecular  $\text{O}-\text{H}\cdots\text{O}$  hydrogen  
bonds.

## Experimental

A solution of 3-diethylaminophenol (0.36 g, 22 mmol) and tetra-  
fluorophthalic anhydride (0.48 g, 22 mmol) in toluene (15 ml) was  
refluxed for 3 h. The solution was cooled to room temperature and  
the precipitate was collected to yield the title compound (0.59 g,  
70%). The crude product was purified by silica-gel flash chromato-  
graphy (methanol-dichloromethane, 1:8). Crystals (m.p. 441–442 K)  
suitable for X-ray diffraction were obtained by slow evaporation of a  
solution in ethyl acetate and acetone (1:3 *v/v*).

Crystal data

C<sub>18</sub>H<sub>15</sub>F<sub>4</sub>NO<sub>4</sub>  
*M<sub>r</sub>* = 385.31  
 Monoclinic, *P*2<sub>1</sub>/*c*  
*a* = 12.954 (2) Å  
*b* = 15.480 (3) Å  
*c* = 9.0431 (16) Å  
 $\beta$  = 100.321 (3)°  
*V* = 1784.1 (5) Å<sup>3</sup>  
*Z* = 4

*D<sub>x</sub>* = 1.435 Mg m<sup>-3</sup>  
 Mo *K*α radiation  
 Cell parameters from 2700 reflections  
 $\theta$  = 3.1–24.3°  
 $\mu$  = 0.13 mm<sup>-1</sup>  
*T* = 294 (2) K  
 Prism, colourless  
 0.40 × 0.30 × 0.24 mm

Data collection

Bruker SMART CCD area-detector diffractometer  
 $\varphi$  and  $\omega$  scans  
 Absorption correction: multi-scan (*SADABS*; Bruker, 1997)  
*T<sub>min</sub>* = 0.951, *T<sub>max</sub>* = 0.970  
 9906 measured reflections

3639 independent reflections  
 2097 reflections with *I* > 2σ(*I*)  
*R<sub>int</sub>* = 0.040  
 $\theta_{\text{max}}$  = 26.4°  
*h* = -16 → 11  
*k* = -16 → 19  
*l* = -10 → 11

Refinement

Refinement on *F*<sup>2</sup>  
*R* [*F*<sup>2</sup> > 2σ(*F*<sup>2</sup>)] = 0.045  
*wR* (*F*<sup>2</sup>) = 0.131  
*S* = 1.00  
 3639 reflections  
 253 parameters  
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0596P)^2 + 0.2523P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta\sigma)_{\text{max}} < 0.001$   
 $\Delta\rho_{\text{max}} = 0.17 \text{ e \AA}^{-3}$   
 $\Delta\rho_{\text{min}} = -0.18 \text{ e \AA}^{-3}$   
 Extinction correction: *SHELXL97*  
 Extinction coefficient: 0.049 (3)

Table 1

Hydrogen-bond geometry (Å, °).

<i>D</i> –H··· <i>A</i>	<i>D</i> –H	H··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> –H··· <i>A</i>
O1–H1···O2 <sup>i</sup>	0.90 (4)	1.74 (4)	2.637 (2)	176 (3)
O4–H4···O3	0.94 (3)	1.74 (3)	2.600 (3)	151 (3)

Symmetry code: (i)  $-x + 2, -y, -z + 1$ .

All H atoms were initially located in a difference Fourier map. Those bonded to O atoms were refined freely. Those bonded to C atoms were refined using a riding model, with C–H distances in the range 0.93–0.97 Å and *U*<sub>iso</sub>(H) = 1.2*U*<sub>eq</sub>(C) or 1.5*U*<sub>eq</sub>(methyl C).

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINTE* (Bruker, 1997); data reduction: *SAINTE*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *SHELXTL*.

References

Bruker (1997). *SADABS* (Version 2.0), *SMART* (Version 5.10), *SAINTE* (Version 5.10) and *SHELXTL* (Version 5.10). Bruker AXS Inc., Madison, Wisconsin, USA.  
 Lee, L. G., Graham, R. J., Werner, W. E., Swartzman, E. & Lu, L. (2002). US Patent No. 6 372 907.

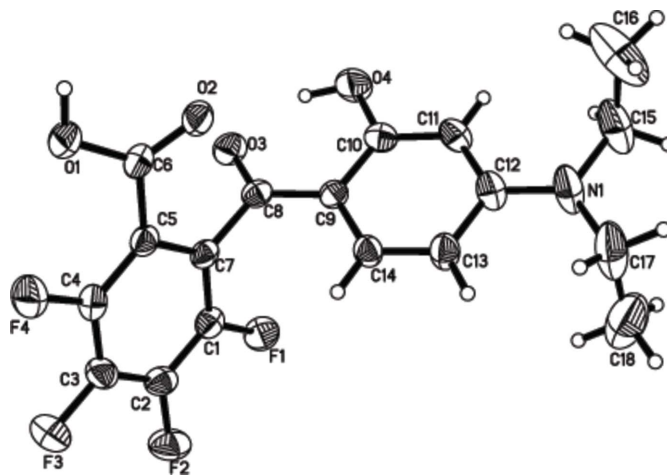


Figure 1

A perspective view of (I). Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as small spheres of arbitrary radii.

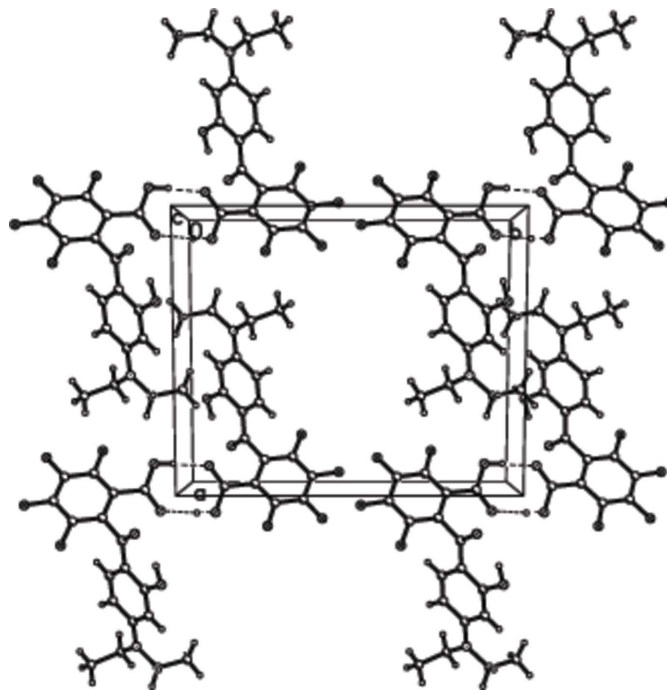


Figure 2

The crystal structure of (I), viewed along the *c* axis. Dashed lines indicate hydrogen-bonding interactions.

Luo, H. P., Pan, J. L. & Lu, W. L. (1994). *J. Zhejiang Univ.* **28**, 349–354.  
 Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.