Acta Crystallographica Section E
**Structure Reports Online**
ISSN 1600-5368

# Naphthalene-1,4,5,8-tetracarboxylic 1,8-anhydride

**Yan-Qing Xu, Da-Qiang Yuan, You-Fu Zhou, Ming-Yan Wu and Mao-Chun Hong***

State Key Laboratory of Structural Chemistry, Fujian Institute of Research on the Structure of Matter, Fuzhou, Fujian 350002, People's Republic of China

Correspondence e-mail: hmc@ms.fjirsm.ac.cn

**Key indicators**

Single-crystal X-ray study
*T* = 173 K
Mean σ(C–C) = 0.004 Å
*R* factor = 0.063
*wR* factor = 0.139
Data-to-parameter ratio = 12.8

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

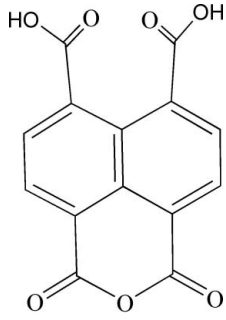


In the title compound, $C_{14}H_6O_7$, strong O—H···O hydrogen-bonding interactions between the molecules result in a one-dimensional chain-like supramolecular structure.

Received 8 March 2005
Accepted 24 March 2005
Online 9 April 2005

## Comment

The hydrothermal reaction of $SmCl_3.nH_2O$ and ntcd (ntcd is naphthalene-1,4,5,8-tetracarboxylic dianhydride) at pH 2.1 yields crystals of the title compound, ntcaa (naphthalene-1,4,5,8-tetracarboxylic 1,8-anhydride), (I). The single-crystal X-ray structure of this compound is reported here.




(I)

The structure of (I) shows that the ntcd is partially hydrolyzed into ntcaa, with an unchanged naphthyl moiety. Apart

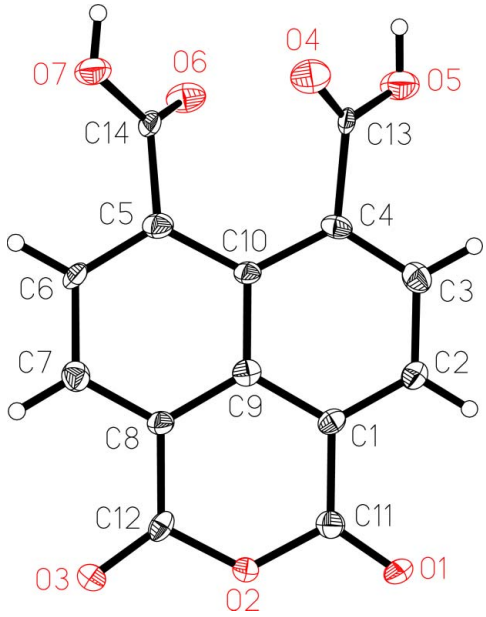



**Figure 1**
The structure of the title complex. Displacement ellipsoids are plotted at the 50% probability level.

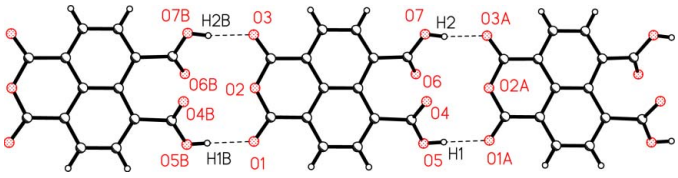



**Figure 2**
The one-dimensional chain structure of the title complex [symmetry codes: (*A*) $x, y-1, z$; (*B*) $x, 1+y, z$]. Dashed lines indicate the hydrogen bonds.

from the carboxyl groups, the molecule exhibits a pseudo-twofold axis along the C9—C10 bond direction (Fig. 1).

Two strong O—H···O hydrogen bonds are formed, with the two carboxylic acid OH groups as donors and the O atoms of another molecules as acceptors (Table 2), resulting in the formation of a one-dimensional chain structure, as shown in Fig. 2.

The geometric parameters of this compound are in general agreement with analogous parameters for naphthalic anhydrides reported previously (Blackburn *et al.*, 1997; Fitzgerald *et al.*, 1991, 1992).

## Experimental

The title compound was prepared by the hydrothermal reaction of $SmCl_3.nH_2O$ (0.091 g) and naphthalene-1,4,5,8-tetracarboxylic dianhydride (0.067 g) in water (16 ml). After heating at 433 K for 3 d and cooling to room temperature at a rate of 13 K $h^{-1}$, yellow needle-shaped crystals of (I) were obtained. Analysis calculated for $C_{14}H_6O_7$: C 58.75, H 2.11%; found: 58.60, H, 2.32%.

*Crystal data*

$C_{14}H_6O_7$
$M_r = 286.19$
Orthorhombic, $P2_12_12_1$
$a = 7.6811\ (15)$ Å
$b = 9.754\ (2)$ Å
$c = 14.357\ (3)$ Å
$V = 1075.6\ (4)$ Å$^3$
$Z = 4$
$D_x = 1.767$ Mg m$^{-3}$
Mo $K\alpha$ radiation
Cell parameters from 54 reflections
$\theta = 2.1$–$27.5°$
$\mu = 0.15$ mm$^{-1}$
$T = 173\ (2)$ K
Needle, yellow
0.50 × 0.05 × 0.05 mm

*Data collection*

Rigaku Mercury CCD area-detector diffractometer
$\omega$ scans
Absorption correction: multi-scan (*CrystalClear*; Rigaku Corporation, 2000)
$T_{min} = 0.906$, $T_{max} = 0.989$
8417 measured reflections
2465 independent reflections
2015 reflections with $I > 2\sigma(I)$
$R_{int} = 0.049$
$\theta_{max} = 27.5°$
$h = -9 \rightarrow 9$
$k = -8 \rightarrow 12$
$l = -18 \rightarrow 18$

*Refinement*

Refinement on $F^2$
$R[F^2 > 2\sigma(F^2)] = 0.063$
$wR(F^2) = 0.139$
$S = 1.09$
2465 reflections
192 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0582P)^2 + 0.5047P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.32$ e Å$^{-3}$
$\Delta\rho_{min} = -0.27$ e Å$^{-3}$

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

| | | | |
|---|---|---|---|
| O1—C11 | 1.205 (4) | O5—C13 | 1.293 (4) |
| C1—C2 | 1.370 (4) | C5—C6 | 1.367 (4) |
| C1—C9 | 1.413 (4) | C5—C10 | 1.436 (4) |
| C1—C11 | 1.476 (5) | C5—C14 | 1.513 (5) |
| O2—C11 | 1.370 (4) | O6—C14 | 1.208 (4) |
| O2—C12 | 1.374 (4) | C6—C7 | 1.413 (4) |
| C2—C3 | 1.408 (4) | O7—C14 | 1.311 (4) |
| O3—C12 | 1.206 (4) | C7—C8 | 1.369 (4) |
| C3—C4 | 1.372 (4) | C8—C9 | 1.414 (4) |
| O4—C13 | 1.222 (4) | C8—C12 | 1.464 (4) |
| C4—C10 | 1.432 (4) | C9—C10 | 1.422 (4) |
| C4—C13 | 1.514 (4) | | |
| C2—C1—C9 | 121.4 (3) | C1—C9—C10 | 119.6 (3) |
| C2—C1—C11 | 119.3 (3) | C8—C9—C10 | 120.2 (3) |
| C9—C1—C11 | 119.3 (3) | C9—C10—C4 | 117.7 (3) |
| C11—O2—C12 | 124.1 (2) | C9—C10—C5 | 117.3 (3) |
| C1—C2—C3 | 119.4 (3) | C4—C10—C5 | 124.9 (3) |
| C4—C3—C2 | 121.0 (3) | O1—C11—O2 | 117.6 (3) |
| C3—C4—C10 | 120.7 (3) | O1—C11—C1 | 124.2 (3) |
| C3—C4—C13 | 117.7 (3) | O2—C11—C1 | 118.2 (3) |
| C10—C4—C13 | 121.1 (3) | O3—C12—O2 | 116.5 (3) |
| C6—C5—C10 | 120.7 (3) | O3—C12—C8 | 125.0 (3) |
| C6—C5—C14 | 117.5 (3) | O2—C12—C8 | 118.5 (3) |
| C10—C5—C14 | 121.3 (3) | O4—C13—O5 | 125.7 (3) |
| C5—C6—C7 | 121.3 (3) | O4—C13—C4 | 120.7 (3) |
| C8—C7—C6 | 119.4 (3) | O5—C13—C4 | 113.3 (3) |
| C7—C8—C9 | 120.9 (3) | O6—C14—O7 | 125.4 (3) |
| C7—C8—C12 | 119.5 (3) | O6—C14—C5 | 121.5 (3) |
| C9—C8—C12 | 119.5 (3) | O7—C14—C5 | 113.0 (3) |
| C1—C9—C8 | 120.1 (3) | | |

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| O5—H1···O1$^{i}$ | 0.84 | 1.90 | 2.735 (4) | 175 |
| O7—H2···O3$^{i}$ | 0.84 | 1.88 | 2.710 (4) | 172 |

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

H atoms were positioned geometrically (C—H = 0.95 Å and O—H = 0.84Å) and allowed to ride on their carrier atoms, with $U_{iso}$(H) = $1.2U_{eq}$(C,O). In the absence of significant anomalous scattering, Friedel pairs were merged.

Data collection: *CrystalClear* (Rigaku Corporation, 2000); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; 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*.

This work was supported by the Natural Science Foundation of China and the Natural Science Foundation of Fujian Province.

## References

Blackburn, A. C., Fitzgerald, L. J. & Gerkin, R. E. (1997). *Acta Cryst.* C**53**, 1991–1995.
Bruker (1997). *SHELXTL*. Version 5.11. Bruker AXS Inc., Madison, Wisconsin, USA.

Fitzgerald, L. J., Gallucci, J. C. & Gerkin, R. E. (1991). *Acta Cryst.* C**47**, 2315–2319.
Fitzgerald, L. J., Gallucci, J. C. & Gerkin, R. E. (1992). *Acta Cryst.* C**48**, 460–465.
Rigaku Corporation (2000). *CrystalClear*. Version 1.3. Rigaku Corporation, Tokyo, Japan.
Sheldrick, G. M. (1997). *SHELXL97* and *SHELXS97*. University of Göttingen, Germany.