

Zurina Shaameri, Ning Shan* and William Jones

Department of Chemistry, University of Cambridge, Lensfield Road, Cambridge, CB2 1EW, UK

Correspondence e-mail: ns261@cam.ac.uk

Key indicators

Single-crystal X-ray study

$T = 295\text{ K}$

Mean $\sigma(\text{C}-\text{C}) = 0.008\text{ \AA}$

Disorder in main residue

R factor = 0.064

wR factor = 0.211

Data-to-parameter ratio = 12.2

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

Diphenic acid–acridine (1/1)

The structure of the title complex, $\text{C}_{14}\text{H}_{10}\text{O}_4 \cdot \text{C}_{13}\text{H}_9\text{N}$, which was cocrystallized from an ethanol solution containing a 1:1 mixture of diphenic acid and acridine, has been determined. Hydrogen bonding exists between acid–acid and acid–base as well as acridine π – π interactions. Proton transfer occurs in one of the two carboxylic acid–aromatic nitrogen interactions.

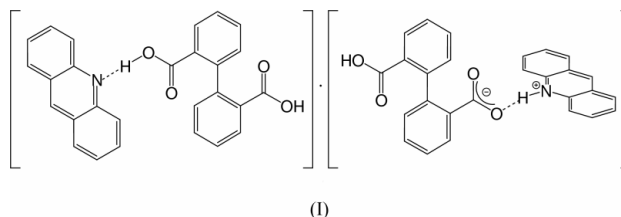
Received 21 September 2001

Accepted 1 October 2001

Online 20 October 2001

Comment

Diphenic acid (biphenyl-2,2'-dicarboxylic acid) forms infinite zigzag acid chains *via* $R_2^2(8)$ carboxylic acid pairs (Fronczek *et al.*, 1987). It is of interest since it is potentially a good building block in crystal engineering with organic bases. To date, no organic co-crystal structure involving diphenic acid exists in the Cambridge Structural Database (*ConQuest*; CCDC 2001). We report here the first diphenic acid cocrystal structure with acridine as the base, (I).



The asymmetric unit of complex (I) comprises two acid and two base molecules (Fig. 1). The biphenyl units of the two acid molecules adopt twisted conformations, with the least-squares planes through the two phenyl units forming angles of 72° and 89° . A two-acid–two-base unit exists (Fig. 2), involving an $\text{O1}-\text{H01}\cdots\text{O4}$ intramolecular hydrogen bond, an $\text{O7}-\text{H03}\cdots\text{O3}$ acid–acid interaction and several acid–base intermolecular interactions (Table 2). In one of the two pairs of acid–base contacts, proton transfer occurs from the carboxylic acid group $\text{O3}-\text{C14}-\text{O4}$ to aromatic N2 , evidenced by the $\text{O3}-\text{C14}$ and $\text{O4}-\text{C14}$ bond distances (Table 1). H02 , the proton involved in the other acid–base contact (located from a difference map) remains within the carboxylic group attached to O5 . The O6 site was refined with disorder over two distinct positions (O6A and O6B) with equal occupancy; possible weak hydrogen bonds $\text{C40}-\text{H40}\cdots\text{O6A}$ and $\text{C40}-\text{H40}\cdots\text{O6B}$ are noted. An infinite supramolecular unit is formed along the a axis by packing the two-acid–two-base units *via* π – π interactions between acridine molecules. Acridine molecular planes are approximately perpendicular to the a axis, with a distance of approximately 3.5 \AA between adjacent planes (Fig. 2). The infinite supramolecular units assemble *via* close packing (Fig. 3).

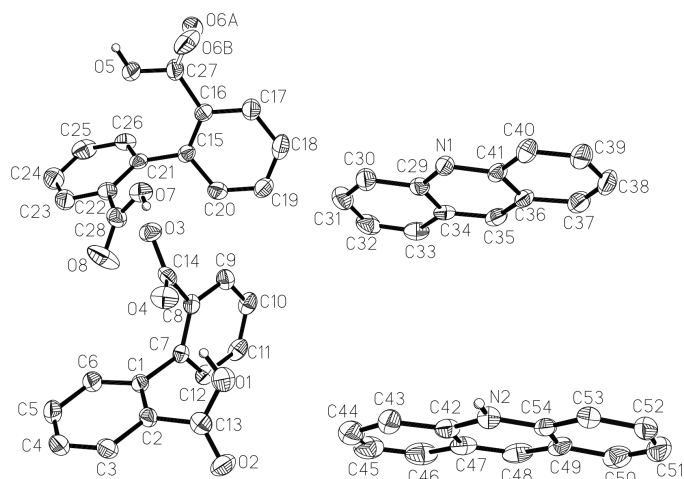


Figure 1
The asymmetric unit of (I) showing displacement ellipsoids at the 30% probability level. H atoms bonded to carbon have been omitted for clarity (Sheldrick, 1993).

Experimental

Diphenic acid and acridine were obtained from Aldrich. 28 mg of the acid and 18 mg of the base were dissolved in 15 ml of ethanol. Crystals were obtained by slow evaporation of the ethanol solution at room temperature.

Crystal data

$C_{14}H_{10}O_4 \cdot C_{14}H_9O_4^- \cdot C_{13}H_9N \cdot C_{13}H_{10}N^+$
 $M_r = 842.86$
 Monoclinic, $P2_1/c$
 $a = 14.461$ (3) Å
 $b = 15.863$ (3) Å
 $c = 19.166$ (3) Å
 $\beta = 108.98$ (2)°
 $V = 4157.5$ (13) Å³
 $Z = 4$

$D_x = 1.347$ Mg m⁻³
 Mo $K\alpha$ radiation
 Cell parameters from 25 reflections
 $\theta = 8.0$ – 15.0°
 $\mu = 0.09$ mm⁻¹
 $T = 295$ (2) K
 Block, yellow
 $0.30 \times 0.30 \times 0.20$ mm

Data collection

Enraf–Nonius CAD-4 diffractometer
 $\omega/2\theta$ scans
 Absorption correction: none
 7509 measured reflections
 7275 independent reflections
 3160 reflections with $I > 2\sigma(I)$
 $R_{int} = 0.045$

$\theta_{max} = 25.0^\circ$
 $h = -17 \rightarrow 16$
 $k = 0 \rightarrow 18$
 $l = 0 \rightarrow 22$
 3 standard reflections every 200 reflections
 intensity decay: none

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.064$
 $wR(F^2) = 0.211$
 $S = 1.01$
 7275 reflections
 598 parameters

H atoms treated by a mixture of independent and constrained refinement
 $w = 1/[\sigma^2(F_o^2) + (0.0914P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{max} = 0.002$
 $\Delta\rho_{max} = 0.23$ e Å⁻³
 $\Delta\rho_{min} = -0.25$ e Å⁻³

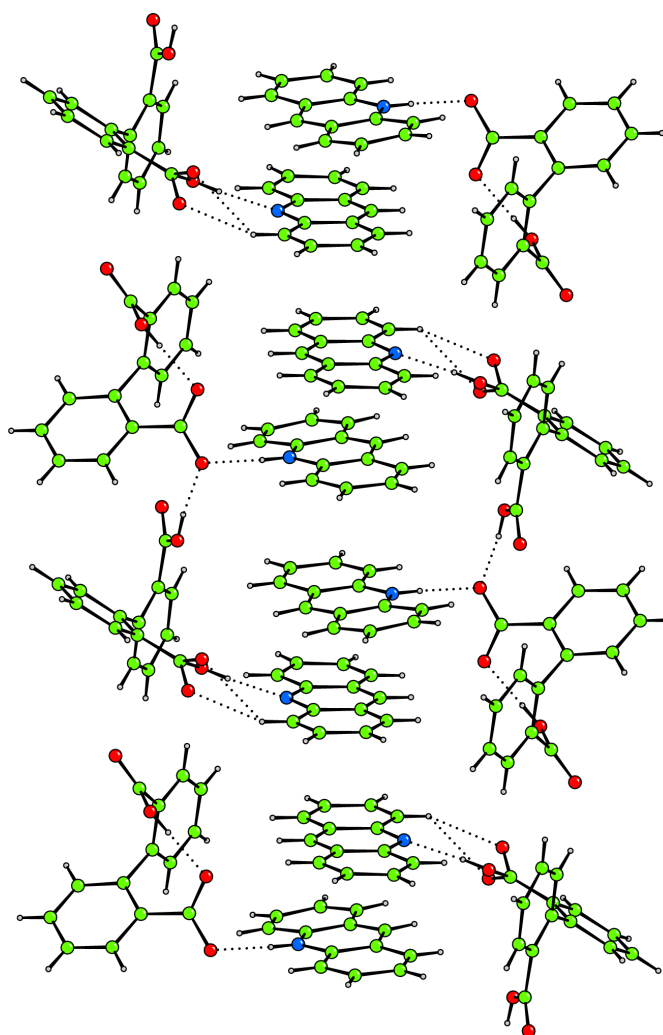


Figure 2
Projection of the molecular packing of (I) on to the (010) plane, showing the acridine molecules stacked along the a axis. (Watkin *et al.*, 1996).

Table 1

Selected geometric parameters (Å, °).

O1—C13	1.325 (6)	O5—C27	1.287 (6)
O2—C13	1.205 (6)	O6A—C27	1.297 (12)
O3—C14	1.265 (5)	O6B—C27	1.223 (11)
O4—C14	1.246 (5)	C28—O8	1.199 (6)
O7—C28	1.308 (6)		

Table 2

Hydrogen-bonding geometry (Å, °).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
O1—H01 \cdots O4	0.84	1.74	2.578 (6)	178
O7—H03 \cdots O3	0.85	1.75	2.596 (5)	173
O5—H02 \cdots N1 ⁱ	0.84	1.86	2.675 (5)	162
C40—H40 \cdots O6A ⁱⁱ	0.93	2.39	3.258 (14)	155
C40—H40 \cdots O6B ⁱⁱ	0.93	2.57	3.282 (12)	133
N2—H2 \cdots O3 ⁱⁱⁱ	0.86	1.88	2.734 (5)	177

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

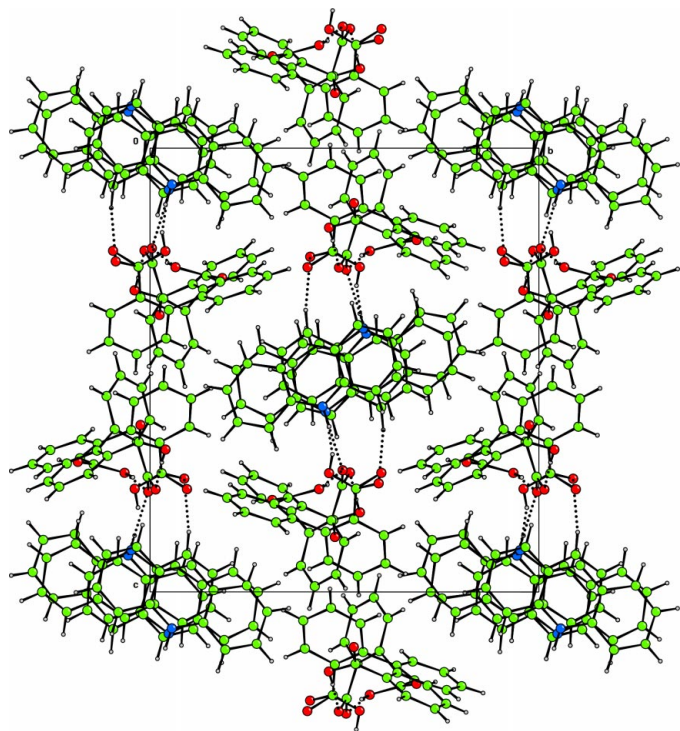


Figure 3
Projection on to (100), showing the packing of the infinite supramolecular unit. (Watkin *et al.*, 1996).

Owing to large anisotropic displacement parameters, atom O6 was refined over two distinct positions with equal fixed occupation factors (from refinement).

All H atoms bonded to C atoms were placed geometrically and refined using a riding model with the U_{iso} values for each H atom taken as $1.2 U_{\text{eq}}$ of the carrier atom. The O-bound H01, H02 and H03 were located from difference Fourier maps and were included in the model with the O–H distance restrained to $0.82 \pm 0.02 \text{ \AA}$. Atom H2 was located from the difference Fourier map and was refined using a riding model with the U_{iso} value taken as $1.2 U_{\text{eq}}$ of N2.

Data collection: *CAD-4 Software* (Enraf–Nonius, 1988); cell refinement: *CAD-4 Software (SETANG)*; data reduction: *TEXSAN* (Molecular Structure Corporation, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *XP* (Sheldrick, 1993); software used to prepare material for publication: *SHELXL97*.

We thank the DWEF Cambridge Scholarship and ORS Award for funding (NS).

References

- CCDC. (2001). *ConQuest*. Version 1.2. CCDC, Cambridge, UK.
 Enraf–Nonius (1988). *CAD-4 Software*. Version 5.0. Enraf–Nonius, Delft, The Netherlands.
 Fronczek, F. R., Davis, S. T., Gehrig, L. M. B. & Gandour, R. D. (1987). *Acta Cryst. C* **43**, 1615–1618.
 Molecular Structure Corporation (1995). *TEXSAN*. MSC, 3200 Research Forest Drive, The Woodlands, TX 77381, USA.
 Sheldrick, G. M. (1993). *XP*. University of Göttingen, Germany.
 Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
 Watkin, D. J., Prout, C. K. & Pearce, L. J. (1996). *CAMERON*. Chemical Crystallography Laboratory, University of Oxford, UK.