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10,11-Dihydrocarbamazepine–acetic acid (1/1)

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Key indicators

Single-crystal X-ray study
T = 123 K
Mean σ(C–C) = 0.005 Å
R factor = 0.076
wR factor = 0.147
Data-to-parameter ratio = 12.5

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

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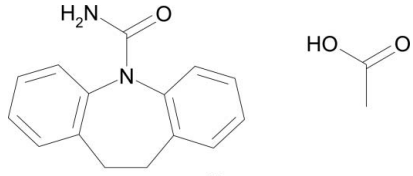
In the title compound [systematic name: 10,11-dihydro-5*H*-dibenz[*b*,*f*]azepine-5-carboxamide–ethanoic acid (1/1)], $C_{15}H_{14}N_2O\cdot C_2H_4O_2$, the dihydrocarbamazepine and acetic acid molecules are hydrogen bonded to form an $R_2^2(8)$ motif, which is further connected into a centrosymmetric double motif arrangement.

Comment

10,11-Dihydrocarbamazepine (DHC) is a recognized impurity in carbamazepine (CBZ), a dibenzazepine drug used to control seizures (Cyr *et al.*, 1987). DHC is known to crystallize in three polymorphic forms: monoclinic form I (Bandoli *et al.*, 1992), orthorhombic form II (Harrison *et al.*, 2006) and triclinic form III (Leech *et al.*, 2006). The title compound, (I), was produced during an automated parallel crystallization study (Florence, Johnston, Fernandes *et al.*, 2006) of DHC as part of a wider study into the predicted and experimental structures of CBZ (Florence, Johnston, Price *et al.*, 2006; Florence, Leech *et al.*, 2006). The sample was identified as a new form using multi-sample foil transmission X-ray powder diffraction analysis (Florence *et al.*, 2003). Subsequent manual recrystallization from a saturated acetic acid solution by slow evaporation at 298 K yielded single crystals of (I) suitable for X-ray diffraction.



(I)

The crystal structure of (I) is essentially isostructural with that of CBZ–acetic acid (1/1) (Fleischman *et al.*, 2003). Accordingly, it displays the same space group with very similar unit-cell parameters and packing arrangements. Specifically, the DHC and acetic acid molecules are connected *via* O2—H1···O1 and N2—H2N···O3 hydrogen bonds (Table 1) to form an $R_2^2(8)$ (Etter, 1990) dimer motif (Fig. 1). A third hydrogen bond, N2—H1N···O3^{i} [symmetry code (i) $1-x$, $1-y$, $-z$], joins adjacent dimers to form a centrosymmetric double motif arrangement (Fig. 2).

Experimental

Crystals of (I) were grown from a saturated acetic acid solution of 10,11-dihydrocarbamazepine by isothermal solvent evaporation at 298 K.

Crystal data

$C_{15}H_{14}N_2O\cdot C_2H_4O_2$
M_r = 298.33
Monoclinic, $P2_1/c$
a = 5.3104 (4) Å
b = 15.4246 (17) Å
c = 18.732 (2) Å
β = 95.106 (7)°
V = 1528.3 (3) Å^3
Z = 4
D_x = 1.297 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 123 (2) K
Needle, colourless
0.35 × 0.08 × 0.04 mm

Data collection

Nonius KappaCCD area-detector diffractometer
φ and ω scans
Absorption correction: none
10078 measured reflections
2652 independent reflections
1605 reflections with $I > 2\sigma(I)$
R_{int} = 0.103
θ_{max} = 25.0°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.076
$wR(F^2)$ = 0.147
S = 1.13
2652 reflections
212 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0421P)^2 + 0.9028P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.20 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O2—H1···O1	1.03 (4)	1.53 (4)	2.547 (3)	167 (4)
N2—H1N···O3^{i}	0.88 (4)	2.20 (3)	2.894 (4)	136 (3)
N2—H2N···O3	0.95 (4)	2.04 (4)	2.970 (4)	164 (4)

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

H atoms bonded to N and O were located in difference maps and refined isotropically (distances are given in Table 1). All other H atoms were positioned geometrically and treated as riding with C—H = 0.95–0.99 Å, and with U_{iso}(H) = 1.2U_{eq}(C), or U_{iso}(H) = 1.5U_{eq}(C) for the methyl group.

Data collection: *COLLECT* (Hooft, 1988) and *DENZO* (Otwinowski & Minor, 1997); cell refinement: *DENZO* and *COLLECT*; data reduction: *DENZO*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97*.

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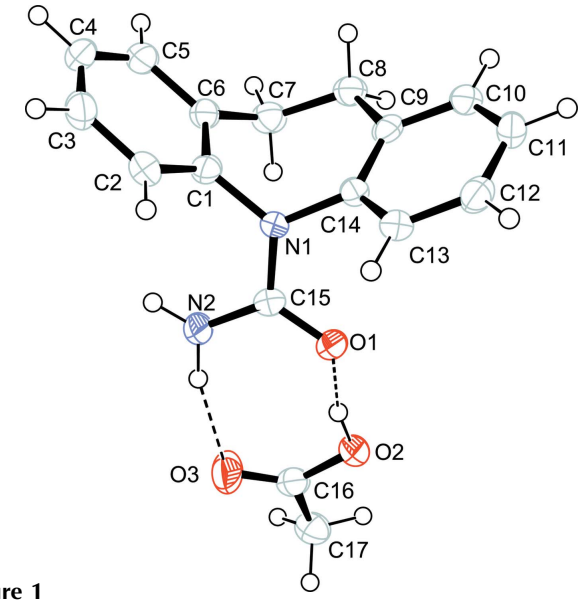

Figure 1
The asymmetric unit of (I), showing 50% probability displacement ellipsoids. Hydrogen bonds are shown as dashed lines.

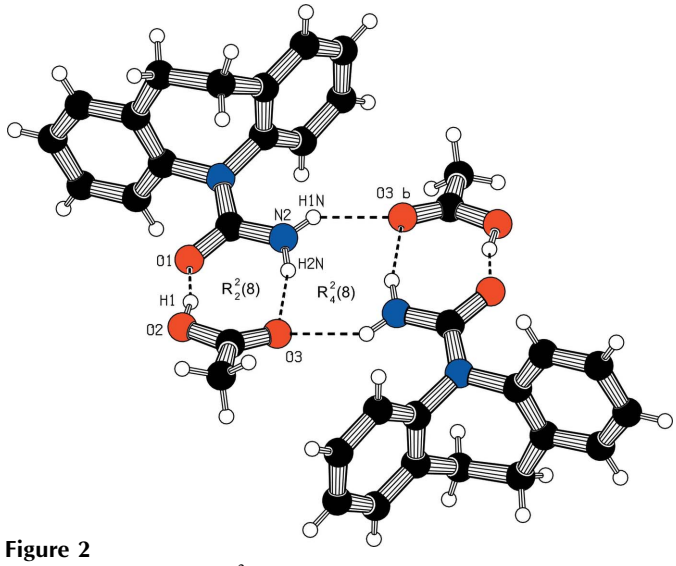

Figure 2
The hydrogen bonded $R_2^2(8)$ motifs of (I) joined in a centrosymmetric arrangement *via* an $R_4^2(8)$ motif. Hydrogen bonds are shown as dashed lines. [Symmetry code: (*b*) $1-x, 1-y, -z$.]

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