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Key indicators

Single-crystal X-ray study
 $T = 123$ K
 Mean $\sigma(C-C) = 0.005$ 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>.

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

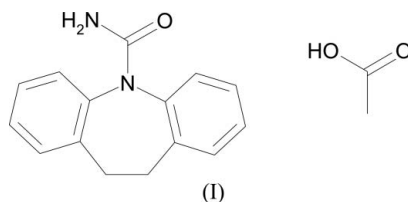
In the title compound [systematic name: 10,11-dihydro-5*H*-dibenz[*b,f*]azepine-5-carboxamide–ethanoic acid (1/1)], $C_{15}H_{14}N_2O \cdot C_2H_4O_2$, the dihydrocarbamazepine and acetic acid molecules are hydrogen bonded to form an $R_2^2(8)$ motif, which is further connected into a centrosymmetric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

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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.



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···O3ⁱ [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

C₁₅H₁₄N₂O·C₂H₄O₂
M_r = 298.33
 Monoclinic, *P*2₁/*c*
a = 5.3104 (4) Å
b = 15.4246 (17) Å
c = 18.732 (2) Å
 β = 95.106 (7)°
V = 1528.3 (3) Å³

Z = 4
D_x = 1.297 Mg m⁻³
 Mo *K*α 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* > 2σ(*I*)
*R*_{int} = 0.103
 θ_{max} = 25.0°

Refinement

Refinement on *F*²
R[*F*² > 2σ(*F*²)] = 0.076
wR(*F*²) = 0.147
S = 1.13
 2652 reflections
 212 parameters

w = 1/[σ²(*F_o*²) + (0.0421*P*)² + 0.9028*P*]
 where *P* = (*F_o*² + 2*F_c*²)/3
 (Δ/σ)_{max} < 0.001
 Δρ_{max} = 0.20 e Å⁻³
 Δρ_{min} = -0.21 e Å⁻³

H atoms treated by a mixture of independent and constrained refinement

Table 1

Hydrogen-bond geometry (Å, °).

| <i>D</i> — <i>H</i> ⋯ <i>A</i> | <i>D</i> — <i>H</i> | <i>H</i> ⋯ <i>A</i> | <i>D</i> ⋯ <i>A</i> | <i>D</i> — <i>H</i> ⋯ <i>A</i> |
|--------------------------------|---------------------|---------------------|---------------------|--------------------------------|
| 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) = 1.2*U*_{eq}(C), or *U*_{iso}(H) = 1.5*U*_{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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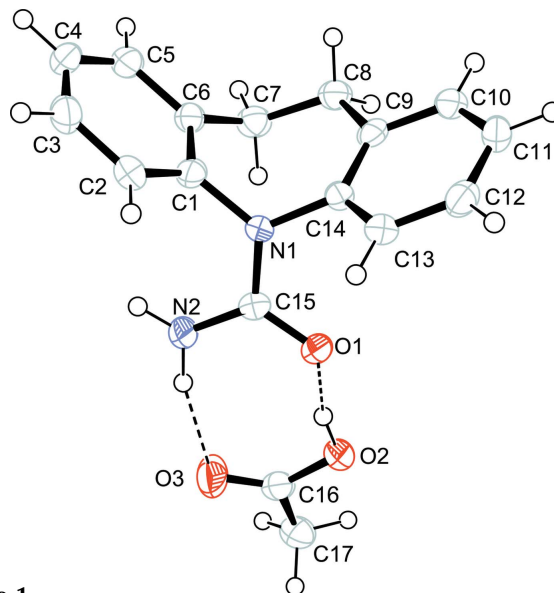


Figure 1 The asymmetric unit of (I), showing 50% probability displacement ellipsoids. Hydrogen bonds are shown as dashed lines.

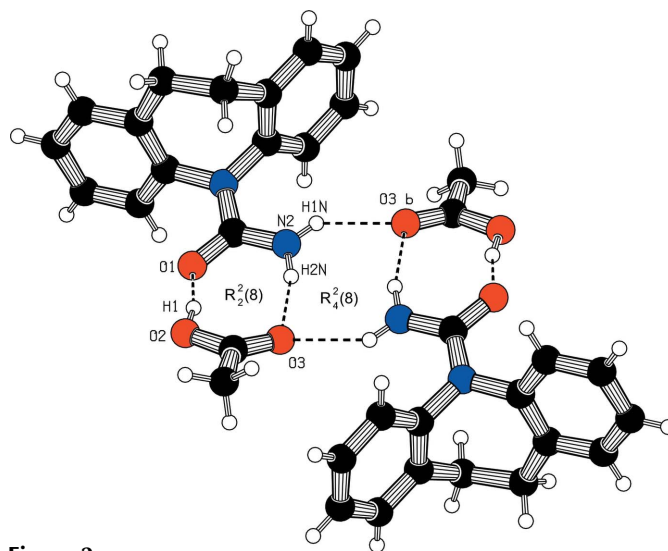


Figure 2 The hydrogen bonded *R*₂²(8) motifs of (I) joined in a centrosymmetric arrangement via an *R*₄²(8) motif. Hydrogen bonds are shown as dashed lines. [Symmetry code: (b) 1 - *x*, 1 - *y*, -*z*.]

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