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A 2:1:1 2-(2-carboxyphenylamino)isophthalic acid–ethyl acetate–butan-2-one inclusion complex

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In the title compound, $C_{15}H_{11}NO_6 \cdot 0.5C_4H_8O_2 \cdot 0.5C_4H_8O$, extensive hydrogen bonding establishes a rigid host matrix in which a disordered mixture of ethyl acetate and butan-2-one solvent molecules is held.

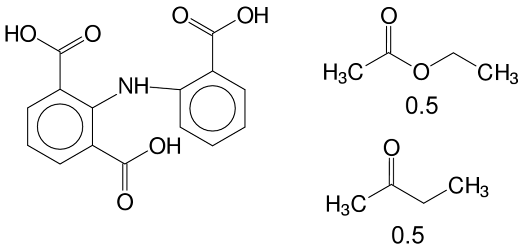
Key indicators

Single-crystal X-ray study
T = 295 K
Mean σ(C–C) = 0.002 Å
H-atom completeness 58%
Disorder in solvent or counterion
R factor = 0.052
wR factor = 0.167
Data-to-parameter ratio = 13.6

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

Comment

The host compound in the title inclusion complex, (I), forms a robust supramolecular framework in the crystal structure *via* intramolecular and intermolecular hydrogen bonding (Fig. 1 and Table 1). A characteristic intramolecular bifurcated hydrogen bond establishes a V-shaped molecular skeleton, reducing the available hydrogen-bond functions towards the exterior. A hydrogen-bonding link to the keto O atom of the guest molecules establishes an important contact site for guest binding in the crystal structure (Fig. 2). A reduced melting point (519–521 K *versus* the reported 531–532 K for the pure compound; Rewcastle & Denny, 1985) may also be attributed to solvent encapsulation. As indicated by difference electron density maps, successful refinement and hydrogen-bonding geometry, all ionizable groups remain in the neutral form. As also demonstrated by a subsequent NMR analysis the crystals, originally recrystallized from butan-2-one, still retained about 20% butan-2-one in the solid after recrystallizing the first batch from ethyl acetate. A 1:1 ethyl acetate–butan-2-one proportion was adopted in the final structure model, omitting H atoms of the disordered guests. The guest molecules appear to be statistically distributed over a single crystallographically unique site, with the two components related by a local pseudo-twofold rotation axis. The packing involves an undulating host framework, with guests held in channels running parallel to the crystallographic c axis (Fig. 2).



(I)

Experimental

2-(2-Carboxyphenylamino)isophthalic acid was prepared as reported previously (Rewcastle & Denny, 1985), and was obtained from solution in butan-2-one. It was subsequently recrystallized from ethyl acetate by slow evaporation of the solvent, yielding large transparent crystals.

Crystal data

$C_{15}H_{11}NO_6{\cdot}0.5C_4H_8O_2{\cdot}0.5C_4H_8O$
$M_r = 381.35$
Monoclinic, $P2_1/c$
$a = 7.487$ (1) Å
$b = 16.444$ (1) Å
$c = 15.748$ (1) Å
$\beta = 100.21$ (1)°
$V = 1908.1$ (3) Å^3
$Z = 4$
$D_x = 1.327$ Mg m^{-3}
Cu $K\alpha$ radiation
Cell parameters from 25 reflections
$\theta = 37.0$–39.8°
$\mu = 0.88$ mm^{-1}
$T = 295$ (2) K
Prism, colourless
0.56 × 0.55 × 0.43 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
ω–2θ scans
Absorption correction: none
4100 measured reflections
3834 independent reflections
3372 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.012$
$\theta_{\rm max} = 75.7°$
$h = -9 \rightarrow 9$
$k = 0 \rightarrow 20$
$l = 0 \rightarrow 19$
3 standard reflections every 100 reflections intensity decay: 1%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.052$
$wR(F^2) = 0.167$
$S = 1.10$
3834 reflections
281 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.1071P)^2 + 0.2617P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max} = 0.44$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.22$ e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.0052 (8)

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

D—H···A	D—H	H···A	D···A	D—H···A
O2—H2O···O7$^{\rm i}$	0.87	1.78	2.645 (2)	175
O4—H4O···O1$^{\rm ii}$	0.98	1.83	2.6576 (18)	140
O5—H5O···O6$^{\rm iii}$	0.83	1.83	2.6441 (19)	166
N1—H1N···O6	0.98	1.96	2.7047 (16)	131
N1—H1N···O3	0.98	2.23	2.7388 (19)	111

Symmetry codes: (i) $1 - x, \frac{1}{2} + y, \frac{3}{2} - z$; (ii) $1 - x, y - \frac{1}{2}, \frac{3}{2} - z$; (iii) $1 - x, 1 - y, 2 - z$.

H atoms bonded to O and N were found in difference maps, while those bonded to C were positioned geometrically; they were all refined with a riding model, with C—H = 0.93 Å, N—H = 0.98 Å and O—H = 0.83–0.98 Å, and with $U_{\rm iso}$(H) = $1.3U_{\rm eq}$ of the parent atom. H atoms were not located for the disordered solvent guest molecules, which were found to be disordered over a single site. A 1:1 distribution of the two guest molecules was assumed. No constraints or restraints were applied, and the geometry of the guest molecules is unreliable.

Data collection: Locally modified *CAD*-4 *Software* (Enraf–Nonius, 1989); cell refinement: *SET*4 (de Boer & Duisenberg, 1984); data reduction: *XCAD*4 (Harms, 1996); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *PLATON*.

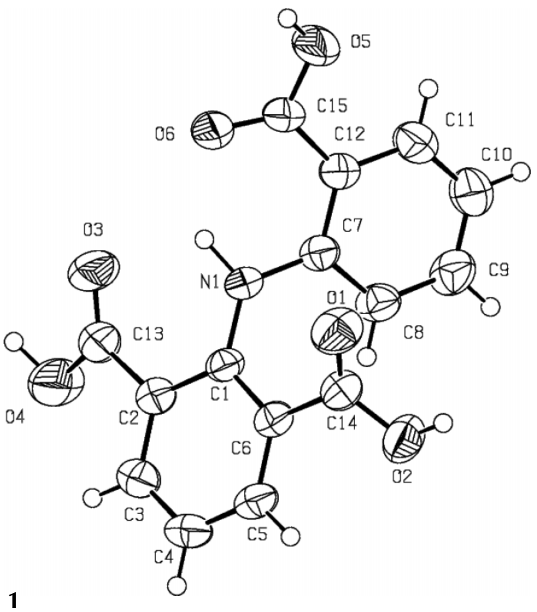


Figure 1
The molecular structure, with displacement ellipsoids drawn at the 50% probability level. The disordered solvent molecules are not shown.

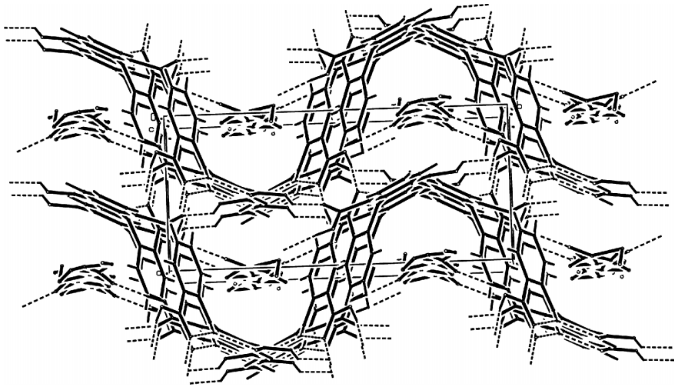


Figure 2
Packing diagram, showing the hydrogen-bonded host matrix with disordered guest molecules bound in channels parallel to the *c* axis. Hydrogen bonds are shown as dashed lines.

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