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Trimesic acid bis(*N,N*-dimethylformamide) solvate at 150 K

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The crystal structure of the title compound, $C_{15}H_{20}N_2O_8$, has been redetermined using low-temperature single-crystal X-ray diffraction.

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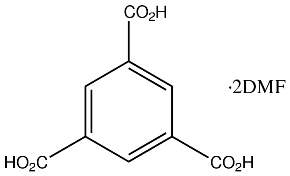
Key indicators

Single-crystal X-ray study
T = 150 K
Mean σ(C–C) = 0.004 Å
R factor = 0.051
wR factor = 0.134
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.

Comment

The title compound was originally synthesized by Chatterjee *et al.* (2000) by the crystallization of trimesic acid (TMA) from *N,N*-dimethylformamide (DMF) in the presence of benzene, and characterized using single-crystal X-ray diffraction at room temperature.



(I)

We have now synthesized the same 1:2 TMA:DMF adduct, (I), in the absence of benzene, characterizing the compound using low-temperature single-crystal X-ray diffraction. The same structure is found in both cases, ruling out the benzene co-solvent as an essential templating agent in the previous synthesis (Chatterjee *et al.*, 2000). Carboxylic acid–DMF $R_2^2(7)$ ring motifs (Etter, 1990; Etter & MacDonald, 1990; Bernstein *et al.*, 1995), utilizing strong O—H···O and weaker C—H···O hydrogen bonding (Desiraju & Steiner, 1999), exist at two of the three carboxylic acid groups, preventing the formation of the carboxylic acid head-to-tail $R_2^2(8)$ dimer motif.

The determination at low temperature (150 K) gives a slight improvement in the final R value (5.14% compared to 5.87%), with reductions of 1.5, 0.4 and 2.2% in the a, b and c unit-cell dimensions, resulting in an overall 4.1% contraction in the unit-cell volume compared to that determined at room temperature.

Experimental

The title compound was prepared as X-ray quality colourless crystals by the slow evaporation of an *N,N*-dimethylformamide solution of trimesic acid at room temperature.

Crystal data

$C_{15}H_{20}N_2O_8$
M_r = 356.33
Monoclinic, $P2_1/c$
a = 16.6529 (18) Å
b = 14.4143 (16) Å
c = 7.2310 (8) Å
β = 90.719 (2)°
V = 1735.6 (3) Å^3
Z = 4
D_x = 1.364 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 1416 reflections
θ = 2.5–26.4°
μ = 0.11 mm^{-1}
T = 150 (2) K
Block, colourless
0.27 × 0.16 × 0.02 mm

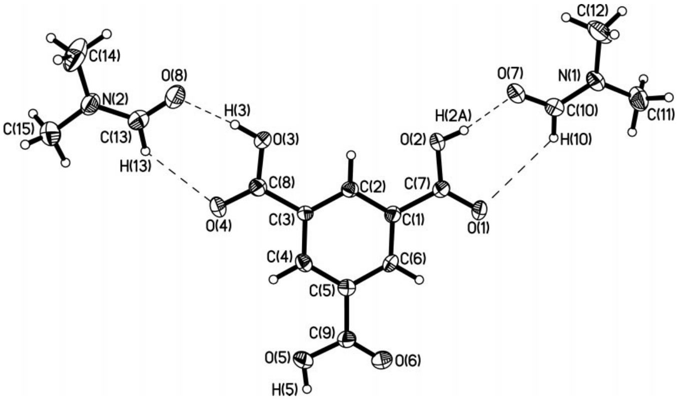


Figure 1
View of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms are represented by circles of arbitrary size. Hydrogen bonding between TMA and DMF molecules is indicated by dashed lines.

Data collection

Bruker SMART 1000 CCD diffractometer
ω rotation scans with narrow frames
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2001)
$T_{\min} = 0.971$, $T_{\max} = 0.998$
8644 measured reflections
3064 independent reflections
1664 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.056$
$\theta_{\max} = 25.0°$
$h = -19 \rightarrow 19$
$k = -9 \rightarrow 17$
$l = -8 \rightarrow 8$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.051$
$wR(F^2) = 0.134$
$S = 1.03$
3064 reflections
240 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0475P)^2 + 0.6362P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.23$ e Å^{-3}
$\Delta\rho_{\min} = -0.22$ e Å^{-3}
Extinction correction: *SHELXTL*
Extinction coefficient: 0.0005 (4)

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

D—H···A	D—H	H···A	D···A	D—H···A
O2—H2A···O7	0.79 (4)	1.73 (4)	2.507 (3)	168 (4)
O3—H3···O8	0.85 (4)	1.75 (4)	2.599 (3)	176 (4)
O5—H5···O1$^{\rm i}$	0.86 (4)	1.88 (4)	2.732 (3)	171 (4)

Symmetry code: (i) $1 - x, \frac{1}{2} + y, -\frac{1}{2} - z$.

H atoms were positioned geometrically. The coordinates of those attached to oxygen were refined freely; all other H atoms were refined using a riding model. $U_{\rm iso}$ values were set to be 1.2 times $U_{\rm eq}$ for aryl and aldehyde H, and 1.5 times $U_{\rm eq}$ for methyl and hydroxy H atoms.

Data collection: *SMART* (Siemens, 1994); cell refinement: *SAINT* (Siemens, 1994); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Bruker, 1997); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL* and local programs.

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References

Bernstein, J., Davis, R. E., Shimoni, L. & Chang, N.-L. (1995). *Angew. Chem. Int. Ed. Engl.* **34**, 1555–1573.
Bruker (1997). *SHELXTL*. Bruker AXS Inc., Madison, Wisconsin, USA.
Chatterjee, S., Pedireddi, V. R., Ranganathan, A. & Rao, C. N. R. (2000). *J. Mol. Struct.* **520**, 107–115.
Desiraju, G. R. & Steiner, T. (1999). *The Weak Hydrogen Bond*. Oxford University Press.
Etter, M. C. (1990). *Acc. Chem. Res.* **23**, 120–126.
Etter, M. C. & MacDonald, J. C. (1990). *Acta Cryst.* B**46**, 256–262.
Sheldrick, G. M. (2001). *SADABS*. Version 2.03. University of Göttingen, Germany.
Siemens (1994). *SMART* and *SAINT*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.