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Urea–*N*,*N*-dimethylacetamide (1/1)

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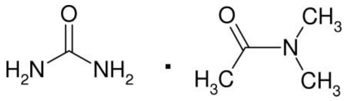
Key indicators: single-crystal X-ray study; T = 120 K; mean σ(O–C) = 0.003 Å; disorder in main residue; R factor = 0.050; wR factor = 0.150; data-to-parameter ratio = 14.9.

Urea forms a 1:1 solvate with *N*,*N*-dimethylacetamide (DMA) [systematic name: diaminomethanal–*N*,*N*-dimethylacetamide (1/1), $C_4H_9NO\cdot CH_4N_2O$] with both molecules positioned on a twofold axis, giving rise to rotational disorder of the DMA molecule. The molecules display a layered structure in which urea molecules form hydrogen-bonded ribbons bounded by molecules of solvent.

Related literature

For details on experimental methods used to obtain this crystalline compound, see: Florence *et al.* (2003). For crystal structures of urea, see: Fernandes *et al.* (2007); Vaughan & Donohue (1952), and references therein; Swaminathan *et al.* (1984); Pryor & Sanger (1970); Guth *et al.* (1980); Weber *et al.* (2002). For related literature, see: Etter (1990).



Experimental

Crystal data

$C_4H_9NO\cdot CH_4N_2O$
M_r = 147.18
Monoclinic, $C2/c$
a = 7.2770 (3) Å
b = 17.5394 (9) Å
c = 7.3789 (4) Å
β = 119.450 (3)°
V = 820.11 (7) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 120 K
0.40 × 0.12 × 0.04 mm

Data collection

Bruker–Nonius KappaCCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2007) T_{min} = 0.867, T_{max} = 1 (expected range = 0.864–0.996)
5338 measured reflections
941 independent reflections
552 reflections with $I > 2.0\sigma(I)$
R_{int} = 0.048

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.050
$wR(F^2)$ = 0.150
S = 0.89
939 reflections
63 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.31 e Å^{-3}
$\Delta\rho_{min}$ = −0.39 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N3—H31···O2^{i}	0.87 (2)	2.06 (2)	2.930 (2)	180 (3)
N3—H32···O9^{ii}	0.87 (2)	2.09 (2)	2.878 (3)	149.7 (19)

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

Data collection: *COLLECT* (Hooft, 1998); cell refinement: *DENZO* (Otwinowski & Minor, 1997) and *COLLECT*; data reduction: *DENZO* and *COLLECT*; program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994); program(s) used to refine structure: *CRYSTALS* (Betteridge *et al.*, 2003); molecular graphics: *ORTEP-3* (Farrugia, 1997) and *Mercury* (Macrae *et al.*, 2006); software used to prepare material for publication: *enCIFer* (Allen *et al.*, 2004) and *publCIF* (Westrip, 2008).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: GA2020).

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Urea–*N,N*-dimethylacetamide (1/1)

Philippe Fernandes, Alastair J. Florence, Francesca Fabbiani, William I. F. David and Kenneth Shankland

S1. Comment

The crystal structure of urea has been widely studied (see for example, Vaughan and Donohue (1952) and references therein; Swaminathan *et al.* (1984), Pryor and Sanger (1970), Guth *et al.* (1980) and Weber *et al.* (2002)). This previously unreported crystalline solvate of urea was discovered during an investigation into the influence of different crystallization solvents on urea crystal morphology (see also Fernandes *et al.*, 2007). The sample was obtained by slow evaporation from a saturated *N,N*-dimethylacetamide (DMA) solution at 298 K and identified by using multi-sample foil transmission X-ray powder diffraction analysis (Florence *et al.*, 2003). Subsequent recrystallization produced a single-crystal suitable for X-ray diffraction at 120 K (Fig. 1).

Both molecules lie over a two fold rotation axis resulting in the DMA being disordered (see refinement section for details). Each urea molecule interacts with adjacent urea molecules *via* contact 1 (Fig. 2, entry 1, Table 1), forming a hydrogen bonded ribbon that runs in the direction [-1 0 1]. Molecules of DMA lie on the edge of the ribbons, connected through a second hydrogen bond (contact 2), (entry 2, Table 1).The DMA-bordered ribbons of urea pack side-by-side to form a two-dimensional sheet.

S2. Experimental

The compound was sourced from Sigma-Aldrich and used as supplied. A single-crystal sample of the 1/1 solvate was recrystallized from a saturated *N,N*-dimethylacetamide solution by isothermal solvent evaporation at room temperature (298 K).

S3. Refinement

The DMA moiety was found to be disordered over a 2-fold rotation axis, with atoms C7 and O9 sitting on this axis. The site occupancies of N4 and C8 were consequently fixed to 1/2, whilst that of C6 was fixed to 1.0 as this atom acts as a methyl carbon both attached to N4 and to C8 in the disordered model. All non-H-atoms were modelled with anisotropic displacement parameters. H-atoms attached to N3 were located in a difference Fourier map and their positions were freely refined. H-atoms attached to C6 and C7 were positioned geometrically, taking into account disorder and occupancy of the parents atoms, and their positions were fixed during refinement. U_{iso}(H) were assigned in the range 1.2–1.5 times U_{eq} of the parent atom.

Note that both the (1 1 0) and the (-2 0 2) reflections were excluded from the final refinement as they were significant outliers on the F_o *versus* Fc plot.

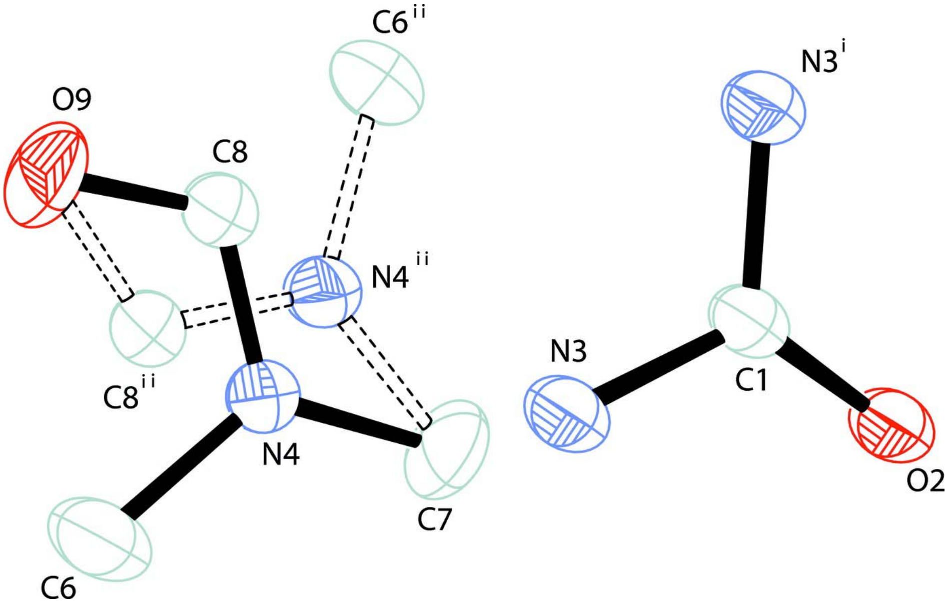

Figure 1

The molecular structure of the title compound showing 50% probablility displacement ellipsoids. Hydrogen atoms have been omitted for clarity. A twofold axis runs through C_1, O_2 of urea and O_9, C_7 of DMA, giving rise to the rotational disorder of the DMA molecule. Symmetry codes: (i) -*x*, *y*, 1/2 - *z*. (ii) -*x*, *y*, 3/2 - *z*.

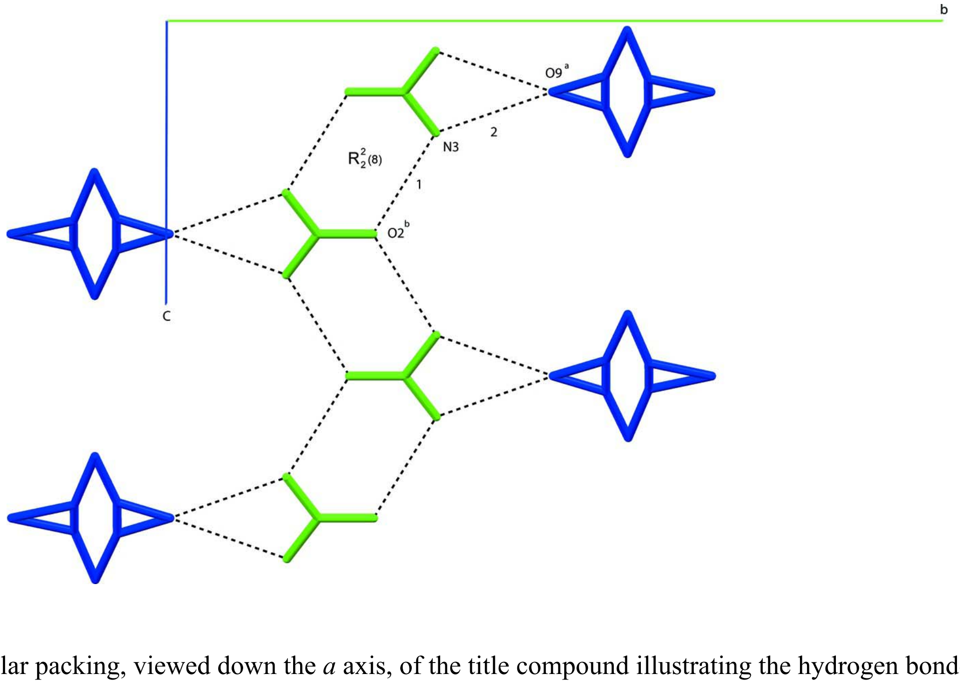

Figure 2

Selected molecular packing, viewed down the *a* axis, of the title compound illustrating the hydrogen bonded network. Urea molecules (green) form an $R_2^2(8)$ motif (Etter, 1990) involving contact 1 (entry 1, Table 1) that propagates to form an infinite ribbon. DMA molecules (shown in blues with rotational disorder) are hydrogen bonded *via* N—H···O contacts 2 (entry 2, Table 1) at the edges of the ribbon. Hydrogen bonds are shown as dashed lines and hydrogen atoms have been omitted for clarity. Symmetry codes: (*a*) -*x*, 1 - *y*, 1 - *z*; (*b*) 1/2 - *x*, 1/2 - *y*, 1 - *z*.

Diaminomethanal–*N*,*N*-dimethylacetamide solvate (1:1)

Crystal data

$C_4H_9NO{\cdot}CH_4N_2O$
$M_r = 147.18$
Monoclinic, $C2/c$
Hall symbol: -C 2yc
$a = 7.2770$ (3) Å
$b = 17.5394$ (9) Å
$c = 7.3789$ (4) Å
$\beta = 119.450$ (3)°
$V = 820.11$ (7) Å^3
$Z = 4$

$F(000) = 320$
$D_x = 1.192$ Mg m^{-3}
Melting point: 406 K
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2218 reflections
$\theta = 3$–27°
$\mu = 0.09$ mm^{-1}
$T = 120$ K
Lath, colourless
0.40 × 0.12 × 0.04 mm

Data collection

Bruker–Nonius KappaCCD diffractometer
Radiation source: Bruker-Nonius FR591 rotating anode
Graphite monochromator
Detector resolution: 9.091 pixels mm^{-1}
φ & ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2007)

$T_{\min} = 0.867$, $T_{\max} = 1$
5338 measured reflections
941 independent reflections
552 reflections with $I > 2.0\sigma(I)$
$R_{\text{int}} = 0.048$
$\theta_{\max} = 27.6°$, $\theta_{\min} = 3.4°$
$h = -9 \rightarrow 9$
$k = -22 \rightarrow 22$
$l = -9 \rightarrow 9$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.050$
$wR(F^2) = 0.150$
$S = 0.89$
939 reflections
63 parameters
0 restraints
Primary atom site location: structure-invariant direct methods

Hydrogen site location: geom + difmap
H atoms treated by a mixture of independent and constrained refinement
Method = Modified Sheldrick $w = 1/[\sigma^2(F^2) + (0.07P)^2]$, where $P = (\max(F_o^2,0) + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.000129$
$\Delta\rho_{\max} = 0.31$ e Å^{-3}
$\Delta\rho_{\min} = -0.39$ e Å^{-3}

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	y	z	U_{iso}*/U_{eq}	Occ. (<1)
C1	0.0000	0.30587 (14)	0.2500	0.0293	
O2	0.0000	0.23473 (9)	0.2500	0.0380	
N3	0.1649 (2)	0.34642 (10)	0.3935 (3)	0.0360	
N4	0.0668 (4)	0.37977 (18)	0.7962 (5)	0.0347	0.5000
C6	0.2835 (3)	0.40670 (13)	0.9670 (4)	0.0525	
C7	0.0000	0.29897 (16)	0.7500	0.0508	
C8	−0.0735 (5)	0.4351 (2)	0.6946 (6)	0.0347	0.5000
O9	0.0000	0.50307 (11)	0.7500	0.0628	
H31	0.264 (3)	0.3224 (11)	0.499 (4)	0.0365*	
H32	0.158 (3)	0.3959 (13)	0.393 (3)	0.0360*	
H71	−0.1409	0.2969	0.6380	0.0608*	0.5000
H72	0.0074	0.2760	0.8696	0.0608*	0.5000
H73	0.0908	0.2732	0.7124	0.0608*	0.5000

H61	0.2880	0.4608	0.9665	0.0542*	0.5000
H62	0.3056	0.3894	1.0980	0.0542*	0.5000
H63	0.3889	0.3866	0.9409	0.0542*	0.5000
H64	0.2827	0.3526	0.9657	0.0542*	0.5000
H65	0.3108	0.4244	1.0994	0.0542*	0.5000
H66	0.3885	0.4250	0.9377	0.0542*	0.5000

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0239 (11)	0.0284 (14)	0.0251 (13)	0.0000	0.0038 (10)	0.0000
O2	0.0315 (9)	0.0245 (10)	0.0342 (11)	0.0000	−0.0022 (8)	0.0000
N3	0.0289 (8)	0.0261 (9)	0.0325 (10)	−0.0009 (6)	−0.0006 (7)	0.0011 (7)
N4	0.0290 (18)	0.0271 (17)	0.038 (2)	0.0003 (11)	0.0085 (16)	−0.0020 (14)
C6	0.0327 (10)	0.0554 (14)	0.0474 (14)	−0.0003 (9)	0.0026 (10)	0.0046 (11)
C7	0.0663 (19)	0.0236 (14)	0.059 (2)	0.0000	0.0286 (17)	0.0000
C8	0.0332 (19)	0.030 (2)	0.031 (2)	−0.0004 (14)	0.0084 (16)	0.0008 (16)
O9	0.0855 (16)	0.0210 (11)	0.0587 (16)	0.0000	0.0176 (13)	0.0000

Geometric parameters (Å, º)

O9—C8	1.288 (4)	C6—H63	0.9500
O9—C8^{i}	1.288 (4)	C6—H64	0.9500
O2—C1	1.248 (3)	C6—H65	0.9500
N4—C6	1.531 (4)	C6—H61	0.9500
N4—C8	1.339 (5)	C6—H66	0.9500
N4—C7	1.483 (4)	C7—H72^{i}	0.9500
N3—C1	1.348 (2)	C7—H73^{i}	0.9500
N3—H32	0.87 (2)	C7—H71^{i}	0.9500
N3—H31	0.87 (2)	C7—H71	0.9500
C6—C8^{i}	1.488 (5)	C7—H72	0.9500
C6—H62	0.9500	C7—H73	0.9500
C6—N4—C7	124.9 (2)	C8^{i}—C6—H61	72.00
C6—N4—C8	115.5 (3)	H62—C6—H63	110.00
C7—N4—C8	119.4 (3)	H62—C6—H64	72.00
C1—N3—H32	119.8 (14)	C8^{i}—C6—H66	109.00
H31—N3—H32	120.7 (19)	N4—C7—H71^{i}	75.00
C1—N3—H31	118.4 (14)	N4—C7—H72^{i}	119.00
N4—C8—C6^{i}	114.0 (3)	N4—C7—H73	109.00
O9—C8—N4	114.2 (3)	H71—C7—H72	110.00
O9—C8—C6^{i}	131.8 (3)	H71—C7—H73	110.00
N4—C6—H61	109.00	N4—C7—H73^{i}	126.00
N4—C6—H62	109.00	H71—C7—H71^{i}	176.00
N4—C6—H63	109.00	H71—C7—H72^{i}	68.00
N4—C6—H64	72.00	H71—C7—H73^{i}	68.00
H61—C6—H63	110.00	H72—C7—H73	110.00
H61—C6—H64	178.00	N4^{i}—C7—H72	119.00

H61—C6—H65	72.00	H71^{i}—C7—H72	68.00
H61—C6—H66	68.00	N4^{i}—C7—H71	75.00
N4—C6—H65	124.00	N4^{i}—C7—H73	126.00
N4—C6—H66	122.00	H71^{i}—C7—H73	68.00
H61—C6—H62	110.00	N4^{i}—C7—H71^{i}	109.00
H62—C6—H66	126.00	N4^{i}—C7—H72^{i}	109.00
C8^{i}—C6—H62	121.00	N4^{i}—C7—H73^{i}	109.00
H63—C6—H64	68.00	H71^{i}—C7—H72^{i}	110.00
H63—C6—H65	123.00	H71^{i}—C7—H73^{i}	110.00
C8^{i}—C6—H63	125.00	N4—C7—H71	109.00
H64—C6—H65	110.00	N4—C7—H72	109.00
H64—C6—H66	110.00	O2—C1—N3	121.84 (12)
C8^{i}—C6—H64	109.00	O2—C1—N3^{ii}	121.84 (12)
H65—C6—H66	110.00	N3—C1—N3^{ii}	116.3 (2)
C8^{i}—C6—H65	109.00		

Symmetry codes: (i) $-x, y, -z+3/2$; (ii) $-x, y, -z+1/2$.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N3—H31···O2^{iii}	0.87 (2)	2.06 (2)	2.930 (2)	180 (3)
N3—H32···O9^{iv}	0.87 (2)	2.09 (2)	2.878 (3)	149.7 (19)

Symmetry codes: (iii) $-x+1/2, -y+1/2, -z+1$; (iv) $-x, -y+1, -z+1$.