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N,*N*-Dimethylammonium *N*′,*N*′-dimethylcarbamate

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Dimethylamine and carbon dioxide form a 2:1 adduct, which is liquid at ambient temperature. Interested in the stabilizing features of the solid, dimethylammonium *N*,*N*-dimethylcarbamate, $C_2H_8N^+\cdot C_3H_6NO_2^-$, we grew single crystals from the pure solution; the structure is reported here. The salt crystallizes as dimers, as a result of hydrogen bonding within and across adjacent ion pairs.

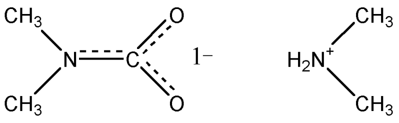
Key indicators

Single-crystal X-ray study
T = 123 K
Mean σ(N–C) = 0.002 Å
R factor = 0.043
wR factor = 0.110
Data-to-parameter ratio = 19.7

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

Comment

Dimethylamine and carbon dioxide form *N*,*N*-dimethylcarbamate complexes with certain metals, of which structures have been reported recently by Klunker *et al.* (1998). The two gaseous compounds also form a 2:1 adduct, which is a liquid at ambient temperature. This compound, called DIMCARB, has been investigated as a safe source for dimethylamine, carbon dioxide or *N*,*N*-dimethylcarbamate in organic synthesis by the groups of Schroth (Schroth *et al.*, 1989) and Hess (Hess *et al.*, 1997). Further, Maschmeier & Matschiner (1992) investigated it as an electrolyte in electrochemical synthesis. Interested in the stabilizing features of the solid DIMCARB, (I), we grew single crystals from the neat solution, of which the structure is reported here. The salt crystallizes as dimers as a result of Hydrogen bonding within and across adjacent ion pairs.



(I)

Experimental

DIMCARB was synthesized by literature methods (Houben-Weyl, 1985), by introducing gaseous dimethylamine into a flask with dry ice and warming it up to ambient temperature. The resulting product is a colourless viscous oil, which is stable at ambient temperature and pressure. Large colourless crystals of *N*,*N*-dimethylammonium *N*′,*N*′-dimethylcarbamate, which melt at 302 K, formed upon standing at ambient temperature within two weeks.

Crystal data

$C_2H_8N^+\cdot C_3H_6NO_2^-$
M_r = 134.18
Monoclinic, $P2_1/c$
a = 7.4171 (2) Å
b = 9.2543 (3) Å
c = 11.0343 (4) Å
β = 95.073 (1)°
V = 754.43 (4) Å^3
Z = 4

D_x = 1.181 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 5677 reflections
θ = 3.5–28.3°
μ = 0.09 mm^{-1}
T = 123 (2) K
Prismatic, colourless
0.20 × 0.20 × 0.10 mm

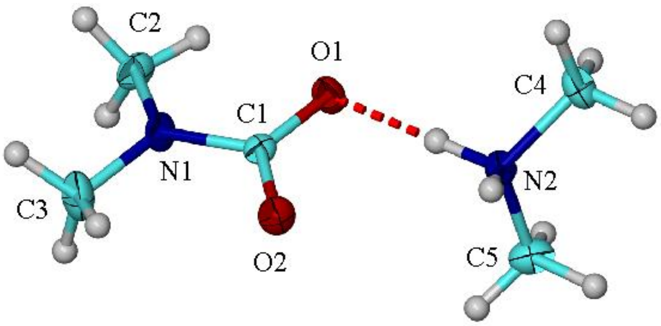


Figure 1
View of *N,N*-dimethylammonium *N′,N′*-dimethylcarbamate (50% probability displacement ellipsoids).

Data collection

Nonius KappaCCD diffractometer
CCD rotation images, thick slice scans
Absorption correction: none
5677 measured reflections
1853 independent reflections

1262 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.059$
$\theta_{\rm max} = 28.3°$
$h = -7 \rightarrow 9$
$k = -10 \rightarrow 12$
$l = -14 \rightarrow 10$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.043$
$wR(F^2) = 0.110$
$S = 1.04$
1853 reflections
94 parameters

H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0532P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max} = 0.18$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.21$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O1—C1	1.2694 (15)	N2—H1	0.983 (16)
O2—C1	1.2628 (15)	C1—N1	1.3788 (17)
N2—C4	1.4726 (17)	N1—C3	1.4485 (19)
N2—C5	1.4776 (17)	N1—C2	1.4490 (16)
N2—H2	0.899 (15)		
C4—N2—C5	112.52 (11)	C1—N1—C3	120.97 (11)
O2—C1—O1	124.33 (12)	C1—N1—C2	121.84 (11)
O2—C1—N1	118.10 (12)	C3—N1—C2	115.11 (11)
O1—C1—N1	117.57 (11)		

H atoms attached to the N atom of the ammonium cation were located and refined. All other H atoms were placed in idealized positions.

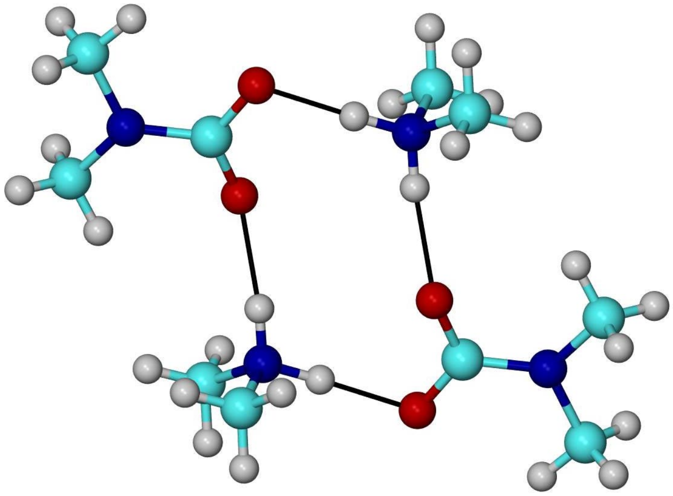


Figure 2
View of the hydrogen-bonded ion-pair dimer.

Data collection: *COLLECT* (Nonius, 1997–2000); cell refinement: *HKL SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *HKL DENZO* (Otwinowski & Minor, 1997) and *SCALEPACK*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: XSEED (Barbour, 1999); software used to prepare material for publication: *XSEED*.

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