



Short Note (3-Ammonio-2,2-dimethyl-propyl)carbamate Dihydrate

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Abstract: (3-Ammonio-2,2-dimethylpropyl)carbamate dihydrate was synthesised. The title compound was characterised by single crystal X-ray diffraction and IR-/Raman-spectroscopy. It has been demonstrated that amixtureofdiluteaceticacidand2,2-dimethyl-1,3-diaminopropane is able to capture CO_2 spontaneously from the atmosphere. An intramolecular hydrogen bond stabilises the conformation of the ylide-type title molecule. Intermolecular hydrogen bonds between all moieties connect them to a strand-type chain structure.

Keywords: carbamate; CO₂ absorption; hydrogen bond; X-ray crystallography

1. Introduction

Carbamates are organic compounds which are derived from carbamic acid: H_2NCOOH . All of the hydrogen atoms in carbamic acid can be replaced by substituents to give a vast number of possible compounds. Surprisingly enough, the mechanism of formation of carbamic acid by the simple reaction of aqueous ammonia and CO_2 gas is still under investigation [1–3]. Furthermore, the kinetics of the formation and the breakdown of aryl-/alkyl-carbamates have been studied for decades (Scheme 1) [4]. Since then, numerous carbamates with various alkyl [5,6] and cyclic alkyl [7] groups have been synthesised.



Scheme 1. Possible functional groups for compounds known from the literature for carbamates (upper part); reactions scheme for the title compound (lower part).

Some carbamates are utilised as potent insecticides [8,9] and herbicides [10–12]. Another important application is in progress: decreasing the amount of CO_2 in the environment with amines

will help to reduce climate change [11–17]. Among other methods, this can be achieved by using amine sorbents for microalgae cultivation [18], amine-functionalised ionic liquid production [19,20], and porous material production [21,22].

There are different methods to synthesise carbamates: through the reaction of sterically hindered primary amines H_2NR (R = alkyl, aryl) with CO_2 [23,24], or through the reaction of CO_2 with secondary amines [25]. For these syntheses, solutions of carbon dioxide [26], as well as the absorption from the air [27], can be used. Recently, a metal organic framework (MOF: $Mg_2(dobpdc)$, (dobpdc = 4,4'-Dioxido-biphenyl-3,3'-dicarboxylate)), which was modified by 2,2-dimethyl-1,3-diaminopropane, was used to absorb CO_2 [28].

2. Results and Discussion

The reaction of 2,2-dimethyl-1,3-diaminopropane (*dmpn*) withacetic acid results in the formation of the title carbamate by CO_2 uptake. The CO_2 uptake from the atmosphere, presumably *via* first dissolving in solvent water, was a surprise to us. Both the timescale (many hours) and scale (>1 g) of reaction completion convince us that further absorption from the air beyond any CO_2 already dissolved in the reagent solutions has occurred.

2.1. Analysis of the X-ray Crystal Structure

The asymmetric unit comprises one carbamate molecule and two water molecules. The molecular geometric parameters are all within the expected ranges (Table 1) [29,30]. The organic title molecule is found in the expected ylidic form and forms one intramolecular hydrogen bond (Figure 1). Hence, the conformation of the backbone of the title compound is fixed. Considering the hydrogen-bonding graph set nomenclature [31,32], a $S_1^1(8)$ graph set descriptor can be given for the ring (H21–N2–C3–C2–C1–N1–C6–O2). Such a hydrogen bond supported cyclic arrangement has been reported for the analogous 2-amino-2-methyl-1-propanol carbamate (AMPC) [33]. The analogy between the title compound and AMPC is a pure hydrogen bond acceptor ($-CO_2^-$) on one side of the molecule and a hydrogen bond donor ($-NH_3^+$ vs. -OH) on the other side. The intramolecular N \cdots O distance in the title structure is very similar to that derived from the structure of AMPC. As mentioned before, 2,2-dimethyl-1,3-diaminopropane has already been used to capture CO₂ in a MOF environment [28]. The deposited crystal structure (CSD reference code ACEWOX) of this compound suggests that the formed carbamate is extended with the NH₂ group coordinated to the Zn atom of the metal organic framework. Unfortunately, there is considerable disorder of this molecule in the crystal [28]. Thus, it may be worth re-investigation.

Atoms	Bond Lengths [Å]	Atoms	Angles [°]
O1–C6	1.2782(9)	C1-N1-C6	123.43(7)
O2–C6	1.2639(10)	C1C2C3	111.74(6)
C1–C2	1.5358(11)	N1-C1-C2	115.43(7)
C2–C3	1.5290(11)	C2-C3-N2	114.74(7)
N1-C1	1.4466(10)	C1C2C3N2	-52.87(9)
N1-C6	1.3609(10)	C6-N1-C1-C2	103.29(9)
N2-C3	1.4901(11)	N1-C1-C2-C4	-179.04(7)

Fable 1. Sel	lected geometr	c parameters o	f the title com	pound (bond le	engths [Å	A]; angles [°]).
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Figure 1. Displacement ellipsoid plot of the asymmetric unit of the title structure (3-ammonio-2, 2-dimethylpropyl)carbamate dihydrate. Ellipsoids are shown at the 50% probability level. Dashed blue lines represent hydrogen bonds (cf. Table 2).

Atoms	D–H [(Å)]	H … A [(Å)]	D … A [(Å)]	D- Н … А [°]			
N1–H11 … O4 ′	0.867(13)	2.198(13)	3.0455(11)	165.8(11)			
N2–H21 … O2	0.912(13)	1.887(14)	2.7747(10)	163.9(12)			
N2–H22 … O1 ″	0.915(13)	1.870(14)	2.7830(10)	175.0(12)			
N2–H23 … O3 ′′′	0.893(13)	2.001(13)	2.8861(11)	171.0(12)			
O3–H31 … O1	0.838(16)	1.963(16)	2.7934(10)	170.5(14)			
O3–H32 … O4 ′	0.835(18)	2.058(18)	2.8764(11)	166.5(16)			
O4–H41 … O2	0.850(17)	2.010(17)	2.8583(10)	175.3(15)			
O4–H42 ··· O1 ////	0.798(17)	2.243(18)	3.0037(11)	159.4(16)			
' = 1 + x y z; $'' = 1 - x 1 - y 1 - z$; $''' = -1 + x - 1 + y - 1 + z$; $'''' = 1 - x 1 - y 2 - z$							

Table 2. Geometric parameters of classical hydrogen bonds in the title structure.

The analysis of intermolecular interactions show that adjacent moieties form a polymeric strand by hydrogen bonds between water molecules and carbamate molecules. This results in a $C_3^3(8)$ graph set description for the chain along the strand (Figure 2). Furthermore, two adjacent carbamates form a ring with $R_4^4(12)$ graph set descriptor (ring A). These dimers are linked by water molecules and form another ring with $R_6^4(12)$ graph set descriptor (ring B).



Figure 2. Hydrogen bonds in the title structure. Basic ring motifs along the propagation direction are given as ring A ($R_4^4(12)$ graph set descriptor) and B ($R_6^4(12)$). The asymmetric unit is labeled and shown with ellipsoids (50%). The intramolecular hydrogen bond N2 … O2 leads to a ring with the S₁¹(8) graph set descriptor (ring C).

2.2. Spectroscopy

The IR and Raman spectra show all expected and characteristic bands and signals, respectively. The comparison of the IR and Raman spectra of the product with those of the pure diamine shows that the bands and signals are more numerous and sharper for (3-ammonio-2,2-dimethylpropyl)carbamate dihydrate (Figure 3: green line). This fact is in excellent accord with our expectations as the 2,2-dimethyl-1,3-diaminopropane was measured at room temperature as a liquid, which may contain several conformers leading to broad bands. On the other hand, the title compound, measured as a crystalline sample, contains the conformer characterised by X-ray methods only (Figure 3). As shown in Figure 1 there is an intramolecular hydrogen bond. The sterical fixation of the backbone of the organic title compound molecule leads to shifts in accord with expectations.



Figure 3. Raman and IR spectra of 2,2-dimethyl-1,3-diaminopropane (red) and (3-ammonio-2, 2-dimethylpropyl) carbamate dihydrate (green).

In detail, the spectra show the signals of the CH-valence modes at around 3370 cm⁻¹, CH-deformation-modes at around 1450 cm⁻¹, and CN-valence vibrations at around 750 cm⁻¹. In the product spectra, the NH-valence-vibration which was noted at 3326 cm⁻¹ disappears. Therefore, the characteristic bands for the N–CO₂⁻-vibration are at around 670 cm⁻¹, 1110 cm⁻¹, and 1500 cm⁻¹ [34,35].

On further analysis of the spectra, structurally related CH_2 -groups show a variety of vibration modes. The following signals were found in amino acids [36], which have a similar structure to the title compound. Firstly, there are two signals of the wagging vibrations at 1378 and 1356 cm⁻¹. At 1296 cm⁻¹, the signal of the torsion-mode appears. The CH_2 rocking mode causes a strong signal at 752 cm⁻¹.

3. Materials and Methods

3.1. General Information

IR Perkin Elmer FTIR-spectra with a LiTaO₃-detector [37,38]. Raman spectrometer (Bruker MultiRAM FT-Raman-spectrometer) with a Nd:YAG Laser und max. output of 500 mW [39].

3.2. Synthesis of (3-Ammonio-2,2dimethylpropyl)carbamate Dihydrate

Dilute acetic acid (20 drops = 1 g) is added dropwise to 1 g 2,2-dimethyl-1,3-diaminopropane. A colorless solution is obtained. The solution (in an unsealed petri dish) was left in a fume hood overnight. The title compound (3-ammonio-2,2-dimethylpropyl)carbamate dihydrate crystallised as colourless crystals. A small amount of liquid is observed besides the solid title compound. The raw product was pressed between two pieces of filter paper and dried in air for one day (Yield: 1.42 g = 80%).

3.3. Spectra

A sample of 2,2-dimethyl-1,3-diaminopropane was analysed as a liquid sample at room temperature. (3-Ammonio-2,2-dimethylpropyl)carbamate dihydrate was measured as a solid crystalline sample.

3.4. X-ray Diffraction Studies

The single crystal diffraction data were collected by using a Bruker APEX2 Duo diffractometer. The cell determination and the data collection strategy followed the standard procedures of the APEX2 software ($T_{min}/T_{max} = 0.9046/0.9282$) [40]. Structure solution [41] and structure refinement [42,43] were done with programs from the SHELX family. All figures and the analysis were done with Diamond software [44]. All coordinates of the hydrogen atoms in the water molecules, the hydrogen atom at the nitrogen atom, and the hydrogen atoms of the methylene groups are refined freely. Their individual U_{iso} parameters were refined too. The hydrogen atoms of the methyl groups were included in the structural model, using a riding model (AFIX 138 [42]). The crystal data for C₆H₁₈N₂O₄ (M = 182.22 g/mol): triclinic, space group $P\overline{1}$, a = 7.1045(4) Å, b = 8.9553(5) Å, c = 8.9752(6) Å, $\alpha = 118.307(2)^{\circ}$, $\beta = 01.443(2)^{\circ}$, $\gamma = 100.500(2)^{\circ}$, V = 466.80(5) Å³, Z = 2, T = 293 K, λ (MoK_{α}) = 0.71073 Å, 16221 reflections measured, 3374 unique reflections ($R_{int} = 0.023$), 3119 observed reflection (I > 2 σ (I)); $R_{ef}(F) = 0.035$, $R_{ref}(F^2$; all data) = 0.076.

Powder diffraction on a representative part of the bulk material showed that the obtained solid is phase pure crystalline title compound (*cf.* the Supplementary Materials).

3.5. Elemental Analysis

Elemental analysis calculated for C₆H₁₈N₂O₄: C 39.55, H 9.96, N 15.37; found: C 39.43, H 9.77, N 15.18.

4. Conclusions

2,2-Dimethylpropane-1,3-diamine, a α,ω -diamine, has already shown its potential of capturing CO₂ as functional part of a MOF material. We succeeded in the quick and effective absorption of CO₂ from the atmosphere *via* dissolution in the reaction medium. The resulting carbamate is very stable as a solid and capable of holding the CO₂ tightly until a temperature of around 130 °C. Above this temperature, the title compound decomposes. These results may be very valuable for future studies on the stability and mechanism of chemical carbon dioxide absorption in the context of recycling carbon dioxide. This contribution forms part of our longstanding interest in salts of diamines in general [45–47] and those of *dmpn* salts in detail [48].

Supplementary Materials: The following are available online, CCDC 1844554 contains the supplementary crystallographic data for this paper. The data can be obtained free of charge from The Cambridge Crystallographic Data Centre.

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Sample Availability: Smallsamples of the compounds (3-Ammonio-2,2-dimethylpropyl)carbamatedihydrateand2, 2-dimethyl-1,3-diaminopropane are available from the authors.



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