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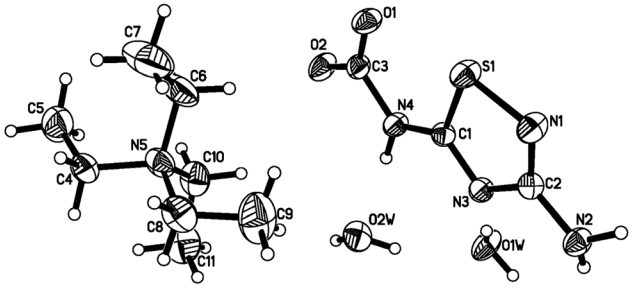
# Crystal structure of the dihydrate of tetraethylammonium 1,3,5-thiadiazole-5-amido-2-carbamate, $C_{11}H_{27}N_5O_4S$

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**Abstract**

$C_{11}H_{27}N_5O_4S$, triclinic, $P\bar{1}$ (no. 2), $a$ = 7.8359 (2) Å, $b$ = 8.6966 (3) Å, $c$ = 12.9907(4) Å, $\alpha$ = 92.047 (2)°, $\beta$ = 92.041 (2)°, $\gamma$ = 96.087 (2)°, $V$ = 879.02 (5) Å$^3$, $Z$ = 2, $R_{gt}(F)$ = 0.0412, $wR_{ref}(F^2)$ = 0.1265, T = 298 K.

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The asymmetric unit of the title crystal structure is shown in the figure. Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

**Table 1:** Data collection and handling.

| | |
|---|---|
| Crystal: | Colorless block |
| Size: | 0.40 × 0.31 × 0.13 mm |
| Wavelength: | Mo *K*α radiation (0.71073 Å) |
| $\mu$: | 0.21 mm$^{-1}$ |
| Diffractometer, scan mode: | Bruker SMART, $\varphi$ and $\omega$-scans |
| $\theta_{max}$, completeness: | 27.7°, >99% |
| $N(hkl)_{measured}$, $N(hkl)_{unique}$, $R_{int}$: | 8700, 4050, 0.013 |
| Criterion for $I_{obs}$, $N(hkl)_{gt}$: | $I_{obs} > 2\ \sigma(I_{obs})$, 3438 |
| $N(param)_{refined}$: | 211 |
| Programs: | Bruker programs [1], SHELX [2, 3] |

## Source of materials

Amidinothiourea and tetraethylammonium hydroxide (25% aqueous solution) were mixed in a molar ratio of 1:2. The mixture was dissolved in a minimum amount of ethanol and water and then vigorously stirred for about 2 h. Subsequently the clean solution was covered with the plastic film with pinholes and set aside to allow it to proceed with slow evaporation in open air at room temperature. Large colorless block crystals were obtained about 15 days later. It can be concluded that amidinothiourea should absorb $CO_2$ of the atmosphere and can be changed into 1,3,5-thiadiazole-5-amido-2-carbamate under basic conditions.

## Experimental details

Hydrogen atoms were placed in their geometrically idealized positions and constrained to ride on their parent atoms.

## Comment

1,3,5–Thiadiazole-5-amido-2-carbamate is a derived compound of amidinothiourea that is an essential pharmaceutical intermediate of famotidine [4], but the related structures of the carbamate were rarely reported. In 2012, only two inclusion compounds of the carbamate were reported as salts of tetrapropylammonium and tetrabutylammonium cations [5]. The title structure can help us further understand the crystallographic characteristics of 1,3,5-thiadiazole-5-amido-2-carbamate under the inductiveness of the template of tetraalkylammonium.

In the asymmetric unit of the crystal structure, there exist one tetraethylammonium, one 1,3,5-thiadiazole-5-amido-2-carbamate and two water molecules. In the title structure each carbamate can link with another inversion-center-related carbamate to generate anionic dimers by

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**Table 2:** Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å$^2$).

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| S1 | 0.59804 (5) | 0.88454 (5) | 0.38336 (3) | 0.05189 (14) |
| O1 | 0.33214 (13) | 0.81543 (13) | 0.25718 (9) | 0.0552 (3) |
| N1 | 0.79516 (16) | 0.90703 (17) | 0.44013 (10) | 0.0525 (3) |
| C1 | 0.66759 (17) | 0.77412 (14) | 0.28372 (10) | 0.0370 (3) |
| O1W | 0.97872 (15) | 0.69772 (17) | 0.09675 (9) | 0.0644 (3) |
| H1WA | 1.0872 (13) | 0.697 (3) | 0.1029 (18) | 0.097* |
| H1WB | 0.940 (3) | 0.715 (3) | 0.1570 (11) | 0.097* |
| O2 | 0.32129 (13) | 0.68111 (15) | 0.10675 (9) | 0.0600 (3) |
| N2 | 1.06508 (16) | 0.82503 (16) | 0.41048 (10) | 0.0485 (3) |
| H2A | 1.112 (2) | 0.9001 (18) | 0.4578 (13) | 0.073* |
| H2B | 1.134 (2) | 0.807 (2) | 0.3566 (12) | 0.073* |
| C2 | 0.89723 (17) | 0.83194 (15) | 0.38338 (10) | 0.0397 (3) |
| O2W | 0.70329 (18) | 0.47303 (15) | 0.08461 (10) | 0.0649 (3) |
| H2WB | 0.673 (3) | 0.425 (3) | 0.0274 (12) | 0.097* |
| H2WA | 0.8022 (18) | 0.523 (3) | 0.0794 (19) | 0.097* |
| N3 | 0.83132 (14) | 0.75493 (13) | 0.29389 (9) | 0.0400 (3) |
| C3 | 0.39345 (17) | 0.73935 (16) | 0.18786 (11) | 0.0424 (3) |
| N4 | 0.56721 (15) | 0.71445 (14) | 0.20178 (9) | 0.0443 (3) |
| H4 | 0.611 (2) | 0.6488 (19) | 0.1552 (12) | 0.067* |
| C4 | 0.0542 (3) | 0.1052 (2) | 0.21862 (17) | 0.0678 (5) |
| H4A | 0.0038 | 0.0912 | 0.2852 | 0.081* |
| H4B | 0.1119 | 0.0145 | 0.2028 | 0.081* |
| N5 | 0.1874 (2) | 0.24451 (16) | 0.22769 (12) | 0.0599 (4) |
| C5 | −0.0901 (3) | 0.1123 (3) | 0.1383 (2) | 0.0965 (8) |
| H5A | −0.1675 | 0.0190 | 0.1385 | 0.145* |
| H5B | −0.1512 | 0.1996 | 0.1541 | 0.145* |
| H5C | −0.0428 | 0.1227 | 0.0714 | 0.145* |
| C6 | 0.1031 (4) | 0.3898 (2) | 0.2545 (2) | 0.0887 (7) |
| H6A | 0.1910 | 0.4774 | 0.2583 | 0.106* |
| H6B | 0.0222 | 0.4065 | 0.1988 | 0.106* |
| C7 | 0.0101 (5) | 0.3870 (3) | 0.3538 (3) | 0.1337 (14) |
| H7A | −0.0386 | 0.4827 | 0.3643 | 0.201* |
| H7B | −0.0797 | 0.3027 | 0.3504 | 0.201* |
| H7C | 0.0894 | 0.3737 | 0.4100 | 0.201* |
| C8 | 0.3162 (3) | 0.2108 (2) | 0.31093 (15) | 0.0741 (5) |
| H8A | 0.3626 | 0.1152 | 0.2918 | 0.089* |
| H8B | 0.2566 | 0.1943 | 0.3744 | 0.089* |
| C9 | 0.4637 (4) | 0.3360 (4) | 0.3313 (2) | 0.1202 (11) |
| H9A | 0.5391 | 0.3056 | 0.3849 | 0.180* |
| H9B | 0.5260 | 0.3514 | 0.2695 | 0.180* |
| H9C | 0.4198 | 0.4307 | 0.3523 | 0.180* |
| C10 | 0.2740 (3) | 0.2705 (2) | 0.12662 (15) | 0.0712 (5) |
| H10A | 0.1876 | 0.2900 | 0.0748 | 0.085* |
| H10B | 0.3551 | 0.3629 | 0.1348 | 0.085* |
| C11 | 0.3675 (3) | 0.1398 (3) | 0.08749 (18) | 0.0923 (7) |
| H11A | 0.4181 | 0.1665 | 0.0234 | 0.138* |
| H11B | 0.4561 | 0.1213 | 0.1371 | 0.138* |
| H11C | 0.2882 | 0.0482 | 0.0770 | 0.138* |

intermolecular N–H···N hydrogen bonding bonds. Adjacent dimers are connected with each other by N–H···O hydrogen bonds to construct a wide anionic ribbon. Consecutively, the wide ribbons are connected by water molecules to generate a 2-dimensional hydrogen-bonded layer. At last, tetraethylammonium cations are between the layers to form the typical sandwich-like crystal structure. Obviously, 1,3,5-thiadiazole-5-amido-2-carbamate can be regarded as a qualified hydrogen-bond donor and acceptor due to its N–H and –COO groups.

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