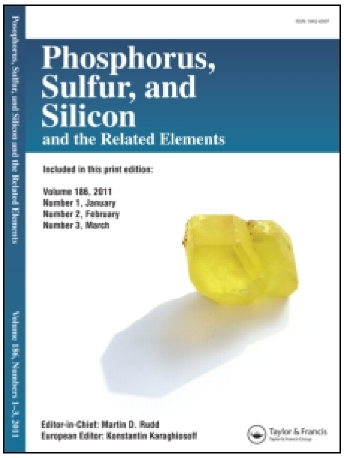

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Synthesis and Investigation of 1,2,3,4-Thiatriazol-5-ylcarbamates

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SYNTHESIS AND INVESTIGATION OF 1,2,3,4-THIATRIAZOL-5-YLCARBAMATES

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Methyl 1,2,3,4-thiatriazol-5-ylcarbamate (2a), ethyl 1,2,3,4-thiatriazol-5-ylcarbamate (2b), 2-butyl 1,2,3,4-thiatriazol-5-ylcarbamate (2c), allyl 1,2,3,4-thiatriazol-5-ylcarbamate (2d), and 3-(1,2,3,4-thiatriazol-5-yl)oxazolidin-2-one (2e) were synthesized with high yields by the reaction of the corresponding carbon(isothiocyanatidic) acid, alkyl esters, and sodium azide in aqueous solution. The compounds were characterized by ^1H, ^{13}C, and ^{15}N NMR, vibrational spectroscopy (IR), and single crystal X-ray diffraction. The thermal stability was investigated by differential scanning calorimetry.

Keywords ^{15}N NMR; single crystal X-ray diffraction; 1,2,3,4-thiatriazol-5-ylcarbamates

INTRODUCTION

1,2,3,4-Thiatriazoles have been known for many years. The association of three nitrogen atoms, one carbon atom and one sulfur atom in one five-membered ring continues to fascinate chemists. The synthesis of these heterocycles can yield rather dangerous intermediates, such as the 5-chloro-1,2,3,4-thiatriazole, synthesized by E. Lieber et al. which detonated spontaneously.[1] Besides their interesting structures, thiatriazoles are sought intermediates in organic chemistry. Thermal decomposition of derivatives of 1,2,3,4-thiatriazole yielded the corresponding cyanate[2] or aroyl thiocyanates.[3] Moreover, the cognate iminothiatriazolines bearing two substituents (one at the carbon atom and the other at the nitrogen atom) were suitable for thermal decomposition yielding carbodiimides.[4] Beside their ability to be decomposed to useful functional groups, 1,2,3,4-thiatriazoles can be used

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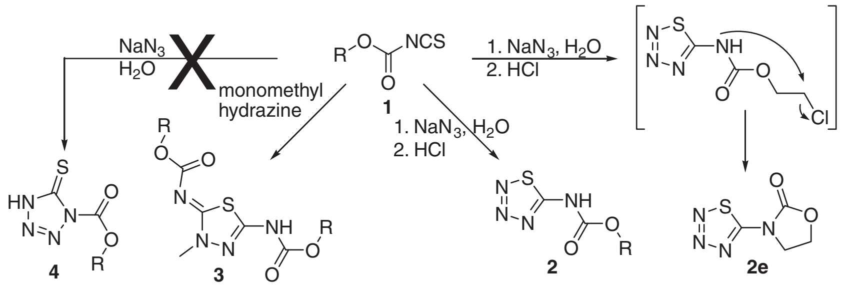
to prepare other heterocyclic compounds such as thiadiazole derivatives by the reaction with isocyanates.[5] Another field of application of 1,2,3,4-thiatriazoles is in pharmaceutical chemistry.[6]

RESULTS AND DISCUSSION

Synthesis

The synthesis of the 1,2,3,4-thiatriazol-5-ylcarbamates (**2**) was carried out using an excess of sodium azide and the corresponding carbon(isothiocyanatidic) acid alkyl esters (**1**). Common solvents for the cyclization of isothiocyanates and azide are dioxane,[7] acetonitrile,[8] or water. Another method for the preparation of thiatriazoles is the diazotation of thiosemicarbazides.[9] The synthesis of **2** was carried out by adding the corresponding isothiocyanates **1** to a boiling solution of and excess of sodium azide in water. The reactivity of the azide anion in aqueous solution is high enough to prevent the highly electrophilic isothiocyanate moiety from hydrolysis. The corresponding sodium salts of **2** that were formed in the aqueous solution could be precipitated as pure powder by acidification of the reaction mixture. The formation of byproducts such as 5*H*-tetrazole-5-thiones (**4**) was not observed. Decomposition products as a result of a hydrolysis of **1** would be CO_2, H_2S, HNCO, and the corresponding alcohols. In view of the high yield of **2**, further investigation of these decomposition products was not carried out. In the case of **2d**, the thiatriazole could only be obtained as a slightly impure compound, due to a decomposition of the allyl moiety.

As illustrated in Scheme 1, the formation of the chloroethyl 1,2,3,4-thiatriazol-5-ylcarbamate was not possible, due to an intramolecular attack of the nitrogen atom.



R = methyl (**a**), ethyl (**b**), 2-butyl (**c**), allyl (**d**)

Scheme 1 Synthesis of the 1,2,3,4-thiatriazol-5-ylcarbamates.

X-Ray Structures

Compound **2e** crystallizes in the orthorhombic spacegroup *Fdd*2 with 16 formula units per unit cell (Figure 1). Inter- or intramolecular interactions between the molecules such as hydrogen bridges are not found. The molecules in the crystal structure are arranged in such a manner that the sulfur atom points towards the O2 of a neighboring molecule (Figure 2). Since the distance between S1 and the neighbouring O2 with 3.28 Å is nearly

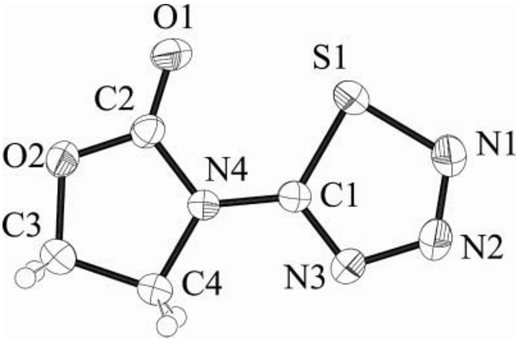


Figure 1 Molecular structure of **2e**. Thermal ellipsoids are drawn at the 50% probability level.

equivalent with the sum of the van der Waals radii of sulfur and oxygen (3.25 Å[10]), an interaction between S1 and O2 could be excluded. On the other hand, the direction of the sulfur atom towards the oxygen atom allows for the assumption that an intermolecular interaction exists, but a distance equivalent to the sum of the van der Waals radii contradicts this thesis.

In case of the other 1,2,3,4-thiatriazol-5-ylcarbamates **2a**–**d**, no suitable crystals for single crystal X-ray diffraction could be obtained. Compound **5** crystallizes in the monoclinic spacegroup *C*2/*m* with four molecular formulas per unit cell. The crystal structure of **5** contains two molecules of crystal water, attached to the sodium atom (Figure 3). The structure is stabilized by several hydrogen bridges, one between the hydrogen atom H4a of O4 to N4 (distance: 2.804 Å, angle: 169.87°) and the other of the same hydrogen atom to O1 (distance: 3.031 Å, angle: 115.38°). Further hydrogen bridges are formed between the hydrogen atom H4b of O4 to N1 (distance: 3.181 Å, angle: 158.14°) and to O1 (distance: 3.031 Å, angle: 112.89°). The second water molecule forms two hydrogen bridges, one between the hydrogen atom H3a of O3 to N2 (distance: 2.899 Å, angle: 178.04°) and the other between the hydrogen atom H3b of O3 to N3 (distance: 2.876 Å, angle: 177.37°). The sodium atoms are arranged in chains, connected by the bridging oxygen atoms of the crystal water or the O1 atoms of the thiatriazole moieties (Figure 4). Thereby, two chains are connected by the hydrogen bridge of H4a to N1.

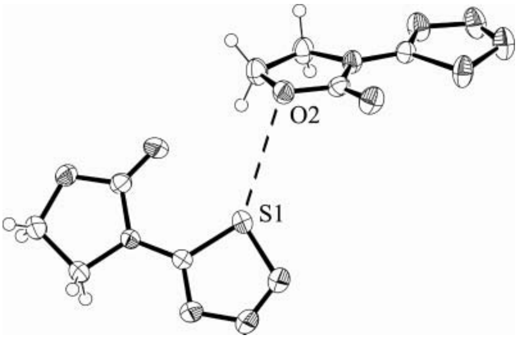


Figure 2 Packing of **2e**. Thermal ellipsoids are drawn at the 50% probability level.

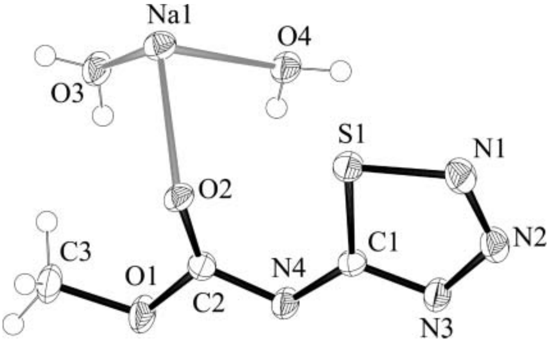


Figure 3 Molecular structure of **5**. Thermal ellipsoids are drawn at the 50% probability level.

Besides the reaction of **1** with sodium azide, the preparation of thiosemicarbazides bearing a carbamate moiety was investigated. Several attempts for the reaction of hydrazine and the corresponding carbon(isothiocyanatidic) acid alkyl esters **1** to form thiosemicarbazides were performed without success. The reaction of carbon(isothiocyanatidic) acid ethyl ester and monomethyl hydrazine led to a condensation product of two molecules of carbon(isothiocyanatidic) acid ethyl ester and one molecule of monomethyl hydrazine (Scheme 1). Compound **3** crystallizes with one water molecule in the triclinic space group *P*-1 with eight formula units per unit cell (Figure 5). Three hydrogen bridges stabilize the structure. The first is located between the ring-nitrogen atom N2 as acceptor and N3-H as donor of the neighboring molecule (distance: 3.073 Å, angle: 171.77°). The water molecule forms two hydrogen bridges, one between O-H as donor to N4 as acceptor (distance: 2.947 Å, angle: 177.67°) and the other between the second proton of the water molecule and O5 of a neighboring water molecule (distance: 2.785 Å, angle: 1.45.64°). The proton of **3** was located at N3. Besides this isomer, two more tautomers could be possible, the tautomer protonated at N2 with the double bond between N3 and C1 or the tautomer protonated at N4. An explanation could be the sterical effect of the methyl group C3. A hydrogen bridge

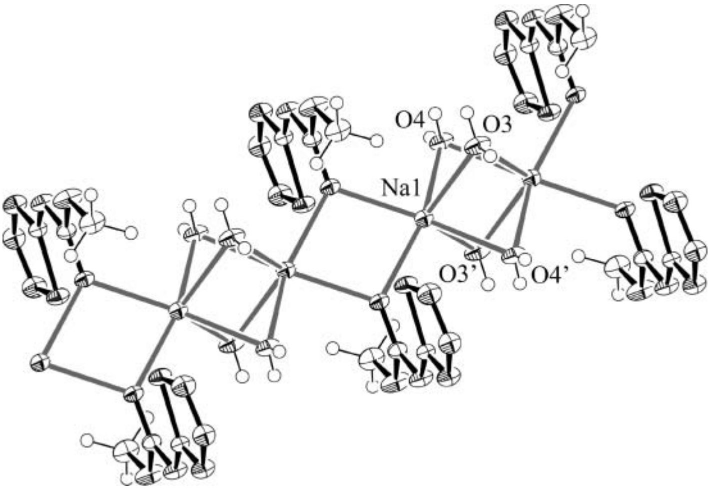


Figure 4 Packing of the sodium salt of **2a**. Thermal ellipsoids are drawn at the 50% probability level.

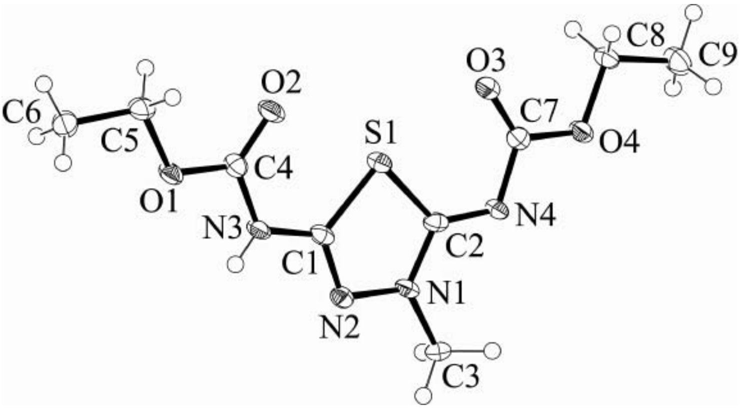


Figure 5 Molecular structure of **3**. Thermal ellipsoids are drawn at the 50% probability level.

to a neighboring N2-atom is formed by the proton at N3. In the case of N4, the methyl group at C3 would complicate these intermolecular interactions due to its sterical hindrance.

NMR Studies

The ^{13}C NMR shifts of **2a–e** were assigned according to known values from the literature. The quaternary carbon atom of the formate moiety at 155 ppm is independent of the different substituents (ranging from 155.1 ppm to 156.3 ppm, Table I). In comparing the quaternary carbon atoms of the thiatriazole moieties, the difference between **2a–d** and **2e** becomes apparent. In contrast to the structures **2a–d**, with only one substituent at the nitrogen atom N4, the chemical shift of the ring carbon atom of **2e** is shifted about 1.5 ppm upfield (172.0 ppm of **2d** compared to 169.5 ppm of **2e**, Table II). Compared to common ^{13}C NMR values of the quaternary carbon atom of amino-1,2,3,4-thiatriazoles known from literature, the quaternary carbon atom is shifted upfield by about 7 ppm (N-(2,4,6-trimethylpheny1)-1,2,3,4-thiatriazol-5-amine: 179.4 ppm[11]; 5-[2-(ethoxycarbonylvinyl)benzyl]amino-1,2,3,4-thiatriazole: 178.2 ppm, $CDCl_3$[12]). In order to compare the ^{15}N NMR spectra of the two different types of thiatriazoles (**2a–d** and **2e**), ^{15}N NMR spectra of **2a** and **2e** were recorded (Figure 6. The three signals ranging between 67.6 ppm and −47.4 ppm were assigned to the three-ring nitrogen atoms. The signals are in accordance with the values of the literature.[13] The comparison between N4 (−267.0 ppm **2a** and 268.3 ppm **2e**) does not show any differences, except in the intensity of the signal. This effect can easily explained by the proton attached to N4 of **2a** and the CH_2-moiety in case of **2e**.

DSC Studies

Besides the analysis of the crystal structures and NMR values, the investigation of the thermal and physical stability is of great interest. A comparison between the thermal stabilities of compounds **2a** to **2e** shows that the point of decomposition depends strongly on the ester moiety. Among the alkyl esters, the 2-butyl derivative **2c** is the most stable compound (119°C) whereas the point of decomposition of **2a** (109°C) and **2b** (111°C) are significantly lower. The allyl derivative **2d** (101°C) possesses the lowest point of

Table I Selected crystal data for **5**, **2e**, and **3**

	5	**2e**	**3**
Formula	$C_3H_3N_4O_2SNa$	$C_4H_4N_4O_2S$	$C_9H_{14}N_4O_4S$
M_r [g mol^{-1}]	182.13	172.17	274.30
Crystal system	Monoclinic	Orthorhombic	Triclinic
Space group	*C*2/*m*	*Fdd*2	*P*-1
a [Å]	15.8754	13.5064	5.1032
b [Å]	6.5299	18.4533	10.8530
c [Å]	8.7428	10.9037	12.8182
α [°]	90	90	66.935
β [°]	110.932	90	88.309
γ [°]	90	90	88.464
V [Å^3]	846.51	2717.61	652.81
Z	4	16	2
F(000)	448.0	1408.0	308
$\rho_{\text{calc.}}$ [g cm^{-3}]	1.712	1.683	1.487
μ [mm^{-1}]	0.42	0.43	0.272
2 theta [°]	54	54	54
Index range	$-20 \leq h \leq 20$	$-17 \leq h \leq 17$	$-6 \leq h \leq 6$
	$-8 \leq h \leq 8$	$-23 \leq h \leq 23$	$-13 \leq h \leq 13$
	$-11 \leq h \leq 11$	$-13 \leq h \leq 13$	$-16 \leq h \leq 16$
Λ	0.71073	0.71073	0.71073
T [K]	200(2)	200(2)	200(2)
Reflections collected	4644	7195	6818
Unique reflections	1004	1477	2829
Parameter	90	116	236
S	1.067	1.139	0.912
R_{int}	0.0214	0.0223	0.0450
R_1/wR_2 [I>2σ(I)]	0.0231/0.0629	0.0228/0.0581	0.0380/0.0628
R_1/wR_2	0.0318/0.0682	0.0265/0.0605	0.0790/0.0628

decomposition, and **2e** the highest (126°C). In contrast to the decomposition of compounds **2a–d**, the decomposition of **2e** proceeds in two steps. A possible explanation for the second exothermic signal at about 155°C could be the second ring system. The point of decomposition at 126°C could be a result of the decomposition of the thiatriazole ring,

Table II ^{13}C NMR shifts in ppm of **2a–2e**

	2a[a]	**2b**[b]	**2c**[a]	**2d**[a]	**2e**[a]
CH_3	54.6	14.4	19.2	—	—
CH_2	—	64.7	73.5	68.1	—
CH	—	—	27.9	—	—
$CH_{2(\text{allyl})}$	—	—	—	132.3	—
$CH_{(\text{allyl})}$	—	—	—	119.5	—
N-CH_2	—	—	—	—	45.6
O-CH_2	—	—	—	—	66.1
C_qO	155.7	154.2	155.5	155.1	156.3
C_qS	171.8	171.6	172.1	172.0	169.5

[a] Measured in d_6-DMSO.

[b] Measured in $CDCl_3$.

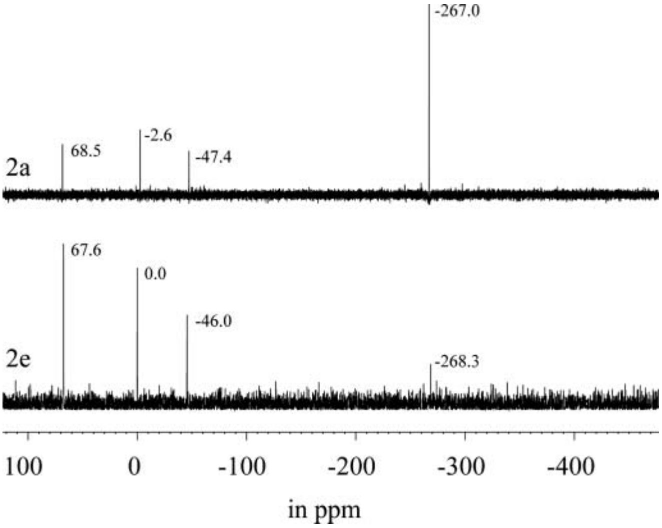


Figure 6 Comparison of the ^{15}N NMR of **2a** and **2e**.

whereas the second decomposition at 155°C could be due to the remaining oxazolidin-2-one moiety. The investigation of the thermal stabilities shows that unsaturated derivatives (**2d**) are the most unstable compounds, whereas the substitution of the proton of N4 by an alkyl moiety (**2e**) leads to an enhanced thermal stability (Figure 7).

The determination of the physical stability of **2a–e** shows only insignificant differences between the five compounds. The sensitivity towards friction and impact was

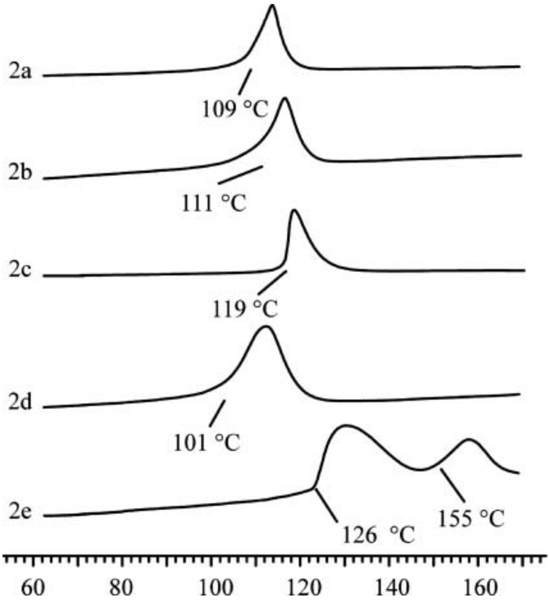


Figure 7 DSC plot of compounds **2a–e**.

determined by BAM standards.[14] The sensitivity towards friction of **2a**–**e** was determined to be >360 N, and the sensitivity towards impact was <30 J.

CONCLUSION

Methyl 1,2,3,4-thiatriazol-5-ylcarbamate (**2a**), ethyl 1,2,3,4-thiatriazol-5-ylcarbamate (**2b**), 2-butyl 1,2,3,4-thiatriazol-5-ylcarbamate (**2c**), allyl 1,2,3,4-thiatriazol-5-ylcarbamate (**2d**), and 3-(1,2,3,4-thiatriazol-5-yl)oxazolidin-2-one (**2e**) were synthesized and characterized by NMR, vibrational spectroscopy (IR), and single crystal X-ray diffraction. In addition, the thermal behavior of **2a**–**e** was investigated by differential scanning calorimetry. The thiatriazoles proved to be thermal stable up to 126°C and showed no sensitivity towards friction but were sensitive towards impact (<30 J). The advantage of the presented synthesis by using carbon(isothiocyanatidic) acid, alkyl esters is the high yield along with a low preparative effort. The alkyl 1,2,3,4-thiatriazol-5-ylcarbamates can be used as cheap and easily available starting materials for the preparation of the 1,2,3,4-thiatriazol-5-amine or to introduce the 1,2,3,4-thiatriazol moiety into more complex molecules.

EXPERIMENTAL

CAUTION! Azides are highly energetic compounds with sensitivity towards heat and impact. Although we had no problems in the synthesis, proper protective measures (safety glasses, face shield, leather coat, earthened equipment and shoes, Kevlar® gloves and ear plugs) should be used when undertaking work involving these compounds.

All chemical reagents and solvents of analytical grade were obtained from Sigma-Aldrich or Acros Organics and used as supplied. ^{1}H, ^{13}C, and ^{15}N NMR spectra were recorded using a JEOL Eclipse 400 instrument. The spectra were measured in d_6-DMSO or $CDCl_3$. The chemical shifts are given relative to tetramethylsilane (^{1}H, ^{13}C) or nitromethane (^{15}N) as external standards. Coupling constants (J) are given in hertz (Hz). Infrared (IR) spectra were recorded using a Perkin-Elmer Spectrum One FT-IR instrument and KBr pellets or NaCl-plates at room temperature. Elemental analyses were performed with a Netsch Simultaneous Thermal Analyzer STA 429. Melting points were determined using differential scanning calorimetry (Linseis DSC PT-10) instrument. Measurements were performed at a heating rate of 5°C min^{-1} in closed aluminium containers with a hole (1 μm) on the top for gas release with a nitrogen flow of 5 mL min^{-1}. The reference sample was a closed aluminium container. The sensitivity data were performed using a BAM drophammer and a BAM friction tester. The crystallographic data were collected using an Oxford Xcalibur3 diffractometer with a Spellman generator (voltage 50 kV, current 40 mA) and a Kappa CCD area detector with graphite-monochromated Mo Kα radiation ($\lambda = 0.71073$ Å). The structure was solved using direct methods (SHELXS-97)[15] and refined using SHELXL-97.[16] All non-hydrogen atoms were refined anisotropically. ORTEP plots showed thermal ellipsoids with 50% probability for the non-hydrogen atoms. CCDC-740461 (**5**), CCDC-740462 (**3**), and CCDC-740463 (**2e**) contain the supplementary crystallographic data for this article. These data can be obtained free of charge from the Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data request/cif.

The used carbon(isothiocyanatidic) acid, alkyl esters **2** were all prepared after known procedures from the literature.[17]

General Synthesis for the Preparation of 1,2,3,4-Thiatriazol-5-ylcarbamates

Sodium azide (0.75 g, 11.5 mmol) was dissolved in water (20 mL) and heated to 70°C. Carbon(isothiocyanatidic) acid, alkyl ester (7.6 mmol) was dissolved in hexane (1.5 mL) and added dropwise to the hot solution of sodium azide in water. The reaction mixture was stirred for 3 h. After cooling to ambient temperature, the aqueous solution was washed with ethyl acetate (50 mL). The aqueous solution was acidified with 2N hydrochloric acid. The neutral 1,2,3,4-thiatriazol-5-ylcarbamates precipitated from the solution and were obtained by filtration.

Methyl 1,2,3,4-Thiatriazol-5-ylcarbamate (2a)

Yield: 75%; dec. 109°C; IR (KBr, cm^{-1}): $\tilde{\nu} = 3430$ (m), 3137 (m), 2962 (m), 2927 (m), 2765 (m), 1717 (vs), 1547 (s), 1468 (m), 1336 (m), 1320 (m), 1271 (s), 1236 (s), 1088 (m), 1020 (w), 948 (vw), 933 (vw), 795(vw), 767 (m), 752 (m), 597 (vw), 581 (vw); ^1H NMR (d_6-DMSO) δ: 3.83 (s, 1H, C*H₃*), 12.29 (br. s, 1H, N*H*); ^{13}C NMR (d_6-DMSO) δ: 54.6 (*C*H₃), 155.7 (*C*O), 171.8 (CS); ^{15}N NMR (d_6-DMSO) δ: 68.5, -2.6, -47.4. -267.0; m/z (DCI^+) 161.1 (69%) (M+H), 133 (28), 101 (24), 69 (16), 58 (9), 57 (100), 56 (9), 43 (52), 42 (8), 41 (21), 39 (8), 33 (7); $C_3H_4N_4O_2S$ calculated: C 22.50, H 2.52, N 34.98; found: C 22.19, H 2.46, N 34.70.

Ethyl 1,2,3,4-Thiatriazol-5-ylcarbamate (2b)

Yield: 72%; dec. 111°C; IR (KBr, cm^{-1}): $\tilde{\nu} = 3448$ (m), 3147 (w), 3086 (w), 3063 (w), 3030 (m), 3004 (m), 2925 (m), 2856 (w), 2769 (w), 1733 (s), 1720 (m), 1604 (w), 1546 (vs), 1384 (w), 1372 (w), 1331 (m), 1316 (m), 1271 (s), 1235 (vs), 1160 (vw), 1091 (m), 1023 (m), 1002 (w), 945 (vw), 877 (vw), 805 (vw), 763 (m), 741 (vw), 590 (vw); ^1H NMR ($CDCl_3$) δ: 1.43 (t, 3H, $^3J = 7.15$ Hz, CH_3), 4.44 (q, 2H, $^3J = 7.15$ Hz, CH_2), 11.77 (br. s, 1H, N*H*); ^{13}C NMR ($CDCl_3$) δ: 14.4 (CH_3), 64.7 (CH_2), 154.2 (*C*O), 172.6 (CS); m/z (DCI^+) 175 (40%) (M+H), 147 (12), 133 (10), 132 (21), 58 (12), 57 (100), 56 (11), 47 (8), 43 (71), 42 (9), 41 (25), 39 (8); $C_4H_6N_4O_2S$ calculated: C 27.58, H 3.47, N 32.17; found: C 27.73, H 3.52, N 31.90.

2-Butyl 1,2,3,4-Thiatriazol-5-ylcarbamate (2c)

Yield: 76%; dec. 119°C; IR (KBr, cm^{-1}): $\tilde{\nu} = 3427$ (m), 3157 (m), 2963 (m), 2915 (m), 2879 (m), 2775 (m), 1731 (vs), 1721 (vs), 1562 (s), 1475 (m), 1466 (m), 1380 (m), 1371 (m), 1345 (m), 1335 (s), 1317 (m), 1277 (s), 1238 (s), 1138 (vw), 1101 (m), 1024 (m), 977 (vw), 966 (vw), 940 (vw), 840 (vw), 799 (w), 770 (m), 756 (m), 594 (vw), 577 (vw), 488 (vw); ^1H NMR (d_6-DMSO) δ: 0.92 (d, 6H, $^3J = 6.83$ Hz, CH_3), 1.97 (sept, 1H, $^3J = 6.83$ Hz, C*H*), 4.07 (d, 2H, $^3J = 6.83$ Hz, CH_2), 13.2 (br. s, 1H, N*H*); ^{13}C NMR (d_6-DMSO) δ: 19.2 (CH_3), 27.9 (*C*H), 73.5 (CH_2), 155.5 (*C*O), 172.1 (CS); m/z (DCI^+) 203 (100%) (M+H), 177 (21), 175 (50), 143 (30), 119 (21), 57 (86), 43 (25), 33 (43); $C_6H_{10}N_4O_2S$ calculated: C 35.63, H 4.98, N 27.70; found: C 35.27, H 4.69, N 27.70.

Allyl 1,2,3,4-Thiatriazol-5-ylcarbamate (2d)

Yield: 60%; dec. 101°C; IR (KBr, cm^{-1}): $\tilde{\nu}$ = 3458 (m), 3159 (m), 2962 (m), 2919 (m), 2765 (m), 1727 (m), 1714 (m), 1647 (m), 1547 (vs), 1454 (m), 1424 (m), 1385 (m), 1359 (m), 1330 (m), 1315 (m), 1262 (vs), 1230 (s), 1097 (m), 1025 (m), 992 (m), 945 (m), 801 (w), 771 (m), 707 (vw), 591 (vw); ^{1}H NMR (d_6-DMSO) δ: 4.75 (d, 2H, $^3J = 5.50$ Hz, CH_2), 5.26 (d, 1H, $^3J = 10.44$ Hz, CH_{cis}), 5.37 (d, 1H, $^3J = 17.04$ Hz, CH_{trans}), 5.95 (m, 1H, CH), 13.25 (br. s, 1H, NH); ^{13}C NMR (d_6-DMSO) δ: 68.1 (CH$_2$), 119.5 (CH), 132.3 (CH$_{2(\text{allyl})}$), 155.1 (CO), 172.0 (CS); $C_5H_6N_4O_2S$ calculated: C 32.25, H 3.25, N 30.09; found: C 32.53, H 3.62, N 31.80.

3-(1,2,3,4-Thiatriazol-5-yl)oxazolidin-2-one (2e)

Yield: 76%; dec. 126°C; IR (KBr, cm^{-1}): $\tilde{\nu}$ = 3427 (m), 2997 (w), 2924 (vw), 1757 (vs), 1506 (vs), 1486 (s), 1471 (s), 1403 (vs), 1365 (m), 1332 (m) 1311 (m), 1255 (m), 1237 (s), 1127 (s), 1082 (m), 1063 (m), 1022 (s), 954 (m), 895 (vw), 804 (vw), 750 (s), 716 (m), 667 (w), 632 (vw), 567 (vw); ^{1}H NMR (d_6-DMSO) δ: 4.33 (t, 2H, $^3J = 7.70$ Hz, N-CH_2), 4.73 (t, 2H, $^3J = 7.70$ Hz, O-CH_2), 13.17 (br. s, 1H, NH); ^{13}C NMR (d_6-DMSO) δ: 45.6 (NCH$_2$), 66.1 (OCH$_2$), 156.3 (CO), 169.5 (CS); ^{15}N NMR (d_6-DMSO) δ: 67.6, 0.0, -46.0, -268.3; m/z (DCI$^+$) 173 (100%) (M+H), 162 (32), 145 (24), 113 (33), 103 (14), 88 (19), 57 (84), 43 (28), 41 (12); $C_4H_4N_4O_2S$ calculated: C 27.91, H 2.34, N 32.54; found: C 27.66, H 2.30, N 32.11.

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14. Impact: insensitive > 40 J, less sensitive > 35 J, sensitive > 4 J, very sensitive < 4 J; friction: insensitive > 360 N, less sensitive = 360 N, sensitive < 360 N > 80 N, very sensitive < 80 N, extreme sensitive < 10 N. According to the UN Recommendations on the Transport of Dangerous Goods (+) indicates: not safe for transport.

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