Central European Journal of Energetic Materials, **2015**, *12*(3), 403-416
ISSN 1733-7178
e-ISSN 2353-1843

Preparation and Characterization of 1-(5-Azido-1*H*-1,2,4-triazol-3-yl)tetrazole

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Abstract: This study presents the preparation of 1-(5-azido-1*H*-1,2,4-triazol-3-yl)tetrazole (**5**) from commercially available chemicals in a five step synthesis. The energetic title compound was comprehensively characterized by various means, including vibrational (IR, Raman) and multinuclear (^{1}H, ^{13}C, ^{14}N, ^{15}N) NMR spectroscopy, mass spectrometry and differential scanning calorimetry. The sensitivities towards various outer stimuli (impact, friction) were determined according to BAM standards. The enthalpy of formation was calculated at the CBS-4M level of theory.

Keywords: energetic materials, heterocycles, triazoles, tetrazoles, azides

1 Introduction

Several research groups world-wide are focused on the development and characterization of energetic nitrogen-rich heterocycles, due to their high enthalpies of formation [1]. The latter can be further increased by introducing the azido group. When heterocycles with at least two carbon atoms are present, then one carbon can be linked to a second nitrogen-rich heterocycle, for example a tetrazole, while the other can carry a highly energetic group like the aforementioned azide. Two recently published compounds featuring a 5-azido-1*H*-1,2,4-triazole and a tetrazole are 5-(5-azido-1*H*-1,2,4-triazol-3-yl)tetrazole (AzTT) and its hydroxy-analogue 5-(5-azido-1*H*-1,2,4-triazol-3-yl)tetrazol-1-ol (AzTTO) [2]. Both compounds have a C–C bond between the two rings.

A second possibility would be a C–N inter-ring connection, resulting in a higher energy content due to the nitrogen linked tetrazole in the form of 1-(5-azido-1*H*-1,2,4-triazol-3-yl)tetrazole (**5**). This compound was recently reported for the first time [3], but interestingly the decomposition temperature was stated as quite high (223 °C with a heating rate of 10 °C·min^{-1}). The present investigation reports a clearly different value.

The goal of this study has therefore been the preparation and characterization of **5**, and its comparison with AzTT and AzTTO (see Figure 1), especially with respect to decomposition temperatures and sensitivity towards external stimuli.

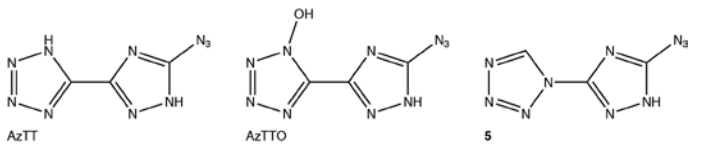
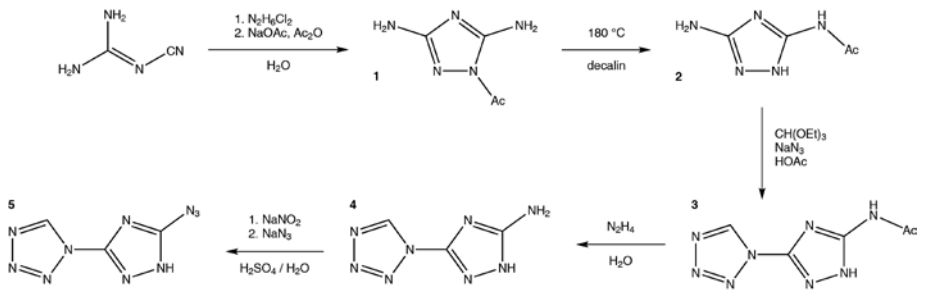


Figure 1. Structures of AzTT, AzTTO and **5**.

2 Results and Discussion

The first step in the synthesis of **5** was the formation of 1-acetyl-3,5-diamino-1,2,4-triazole (**1**) [4]. The literature method is the direct reaction of the rather expensive 3,5-diamino-1*H*-1,2,4-triazole (DAT) with acetic anhydride, resulting in yields of around 80%. A more cost-effective way on a laboratory scale is the synthesis of DAT utilizing cyanoguanidine and hydrazinium dichloride, which can be reacted with acetic anhydride directly in the reaction mixture after neutralization with sodium acetate [5]. The resulting yield of **1** was around 64%. Isolation and purification of the DAT intermediate is not necessary, and is also rather complicated, often resulting in low yields. The next step was the rearrangement of **1** to 5-acetamido-3-amino-1*H*-1,2,4-triazole (**2**) at temperatures above 180 °C [4], thus resulting in the protection of one of the amino groups. The two isomers, **1** and **2**, are easy to distinguish due to the much lower solubility of **2** in practically all common solvents, as well as the shift of the C=O vibration from 1710 to 1682 cm^{-1} in the IR spectra. The remaining free amine was then reacted with triethyl orthoformate and sodium azide in acetic acid, a method analogous to that first described by Gaponik *et al.* for various primary amines [6], to yield 1-(5-acetamido-1*H*-1,2,4-triazol-3-yl)tetrazole (**3**) [7]. Although the literature procedure for this particular compound reported a yield of around 80% [7], it was not possible to obtain any product; only starting material could be recovered. Even variations in the amount of acetic acid, heating time or heating

temperature did not yield any product. Whilst the utilization of trifluoroacetic acid as solvent resulted in a clear solution (acetic acid gives a slurry), it still did not yield any detectable product. Only stirring of the reaction mixture for several days at room temperature prior to heating overnight finally yielded the desired result. Further reaction of **3** with hydrazine hydrate resulted in the deprotection of the amine, yielding 1-(5-amino-1*H*-1,2,4-triazol-3-yl)tetrazole (**4**) [7]. The final step was diazotization with sodium nitrite in sulfuric acid, followed by reaction with sodium azide to yield the title compound 1-(5-azido-1*H*-1,2,4-triazol-3-yl)tetrazole (**5**).



Scheme 1. Synthetic route to 1-(5-azido-1*H*-1,2,4-triazol-3-yl)tetrazole (**5**).

Compound **5** was characterized by NMR, IR and Raman spectroscopy, mass spectrometry and differential scanning calorimetry. Additionally, the sensitivity towards common external stimuli (impact, friction) was determined according to BAM standards (*Bundesanstalt für Materialforschung und -prüfung*, see Experimental Section). Unfortunately, it was not possible to obtain single crystals for X-ray diffraction, utilizing several solvents and techniques.

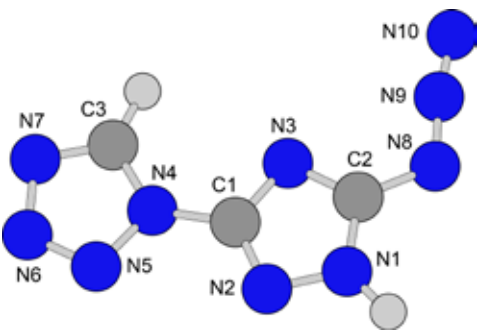


Figure 2. Calculated structure of **5** (B3LYP/aug-cc-pVDZ), showing the atom numbering scheme. C1–N4 1.401 Å, N8–N9–N10 171.95°, N3–C1–N4–N5 179.99°, N3–C2–N8–N9 0.00°.

The ^{1}H NMR spectrum only shows the signal of the tetrazole CH proton at 10.10 ppm. The NH proton of the triazole could not be observed, probably due to its acidity and thus fast exchange in DMSO-d_6. The ^{13}C NMR spectrum (see Figure 3) shows, as expected, three signals at 151.6 (C2), 150.4 (C1), and 142.9 ppm (C3). The shift of the tetrazole carbon (C3) is similar to the shift found in 1*H*-tetrazole (142.9 ppm), 1-phenyltetrazole (140.5 ppm), 1,2-di(tetrazol-1-yl) ethane (144.3 ppm), and 5-(tetrazol-1-yl)-2*H*-tetrazole (143.8 ppm) [8], and quite different from the *C*-bonded tetrazole of AzTT (148.4 ppm, see Figure 3). The corresponding shifts of the amine precursor **4** are located at 157.6 (C2), 150.1 (C1), and 142.7 ppm (C3). The broad signal observed in the ^{14}N NMR spectrum at −139 ppm ($\nu_{½}$ = 384 Hz) can be attributed to the azide beta nitrogen atom (N9), owing to its sufficiently high linear symmetry and the absence of a lone pair. Tetrazole and triazole nitrogen atoms are usually not visible in a ^{14}N NMR spectrum due to their lone pairs and the delocalized electron systems, resulting in strong line broadening. On the other hand, all ten atoms are clearly observable in the ^{15}N NMR spectrum. The tetrazole moiety exhibits signals at 12.0 (d, $^{3}J_{NH}$ = 3.43 Hz, N6), −20.2 (N5), −52.4 (d, $^{2}J_{NH}$ = 12.11 Hz, N7), and −147.3 ppm (d, $^{2}J_{NH}$ = 9.29 Hz, N4), matching the reported shifts for 1-aryltetrazoles very well [9]. The signals at −119.4 (N2), −166.8 (N3), and −185.7 ppm (N1) can be attributed to the triazole moiety and are also in accordance with other *C*-azido-1,2,4-triazoles [2, 10]. The three remaining signals at −143.8 (N9/N10), −146.0 (N10/N9), and −293.8 ppm (N8) belong to the azido group [11], and also show similar shifts to other *C*-azido-1,2,4-triazoles [2, 10]. For the clear assignment of N9 and N10 a ^{15}N labelled azide would be needed, because the gamma atom can have a lower high field shift than the beta atom when the azide is connected to an electron deficient system [12]. Quantum-chemical calculations (MPW1PW91/aug-cc-pVDZ) indicated that in this particular case N10 has indeed the lower high field shift, similar to AzTTO [2b] and 5-azido-3-nitro-1*H*-1,2,3-triazole (AzNT) [10], but the intensities of the signals at −143.8 and −146.0 ppm in **5** are almost identical, whilst in the spectra of AzTTO and AzNT the assumed beta nitrogen atom has a noticeably higher intensity than that of the gamma nitrogen atom.

The only clearly assignable band in the rather complicated IR spectrum is the stretching mode of the terminal azide bond at 2156 cm^{-1}, which is also present in the Raman spectrum at 2166 cm^{-1}.

Low resolution mass spectrometry (DEI+) shows the molecular peak at m/z = 178.2 with a low abundance.

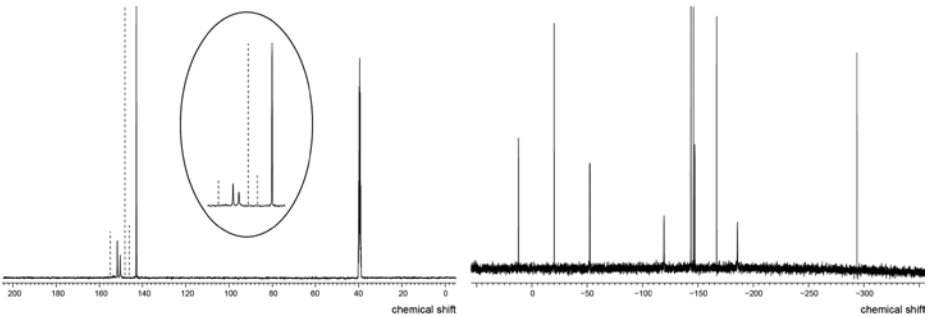


Figure 3. Left: ^{13}C NMR spectrum of **5** (the dashed lines represent the shifts of AzTT); right: ^{15}N NMR spectrum of **5**; recorded in DMSO-d_6 at room temperature.

The most striking result was the thermal stability, which is practically identical to that of AzTT and AzTTO at, a rather low, 152 °C. The DSC plots of the three compounds are presented in Figure 4. The two endotherms are loss of crystal water (AzTT; measurement was performed with the dihydrate) and melting (AzTTO). This could indicate that the weakest link is indeed the azidotriazole unit and neither the type nor the linkage of the tetrazole ring on the second carbon atom really matters. The nitramine analogue, 1-(5-nitramino-1*H*-1,2,4-triazol-3-yl)tetrazole, reportedly decomposes already at 91 °C [7].

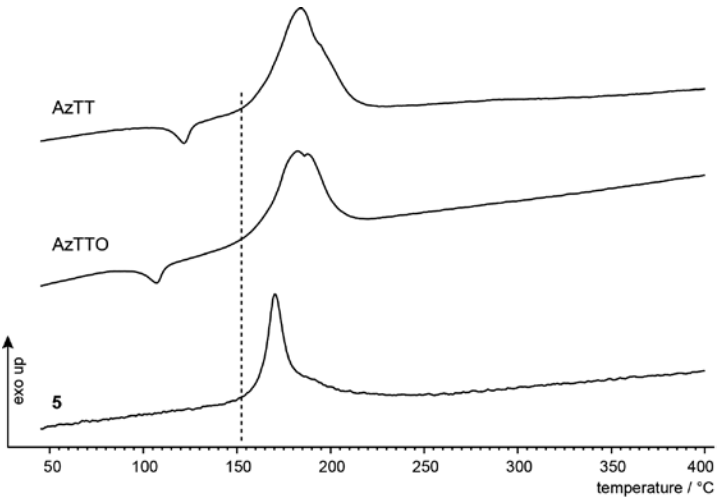


Figure 4. DSC plots for AzTT dihydrate, AzTTO and **5** at a heating rate of 5 °C min^{-1}. The dashed line indicates the decomposition onset for AzTT and **5**.

Regarding the sensitivities, **5** is rather sensitive to impact (4 J) and extremely sensitive to friction (10 N). These values are comparable to AzTT (1 J, 20 N) and AzTTO (4 J, 120 N), although AzTT is surprisingly more sensitive to impact, while AzTTO is much less sensitive to friction.

Nitrogen-rich highly energetic compounds tend to burn incompletely in bomb calorimetric measurements due to the tendency for explosion. Often the wrong enthalpies of combustion ($\Delta_c H$) and thus the wrong enthalpies of formation ($\Delta_f H°$) are obtained. Therefore the enthalpies of formation of AzTT and **5** have been calculated at the same level for a better comparison. All calculations were carried out using Gaussian 09 (revision C.01) [13]. The enthalpies (H) were calculated using the complete basis set (CBS) method described by Petersson and co-workers in order to obtain very accurate values. In this study the modified CBS-4M method (M referring to the use of minimal population localization) was applied, which is a reparameterized version of the original CBS-4 method and also includes some additional empirical corrections [14]. According to the calculations, the tautomer with the proton being on the nitrogen atom closer to the tetrazole ring has a lower energy than the tautomer where the proton is on the nitrogen closer to the azide (see AzTT or AzTTO), although the difference is practically negligible (0.49 kJ·mol^{-1}). Thus the latter was used for the following calculations and the numbering of the atoms (see Figure 2). The molecule itself shows a completely planar structure (both CBS-4M and B3LYP/aug-cc-pVDZ), similar to AzTT, with a C1–N4 single bond (1.401 Å) connecting the two rings. The aforementioned 1-(5-nitramino-1*H*-1,2,4-triazol-3-yl)tetrazole on the other hand consists of two twisted rings in the crystal structure [7]; however the torsion angle was not given.

The enthalpies of the gas-phase species M were calculated by the atomization energy method according to Equation 1 [15], using literature values for the atomic $\Delta_f H°_{(g,A)}$ values [16].

$$\Delta_f H°_{(g,M)} = H^{298}_{(g,M)} - \sum H^{298}_{(g,A)} + \sum \Delta_f H°_{(g,A)} \quad (1)$$

The solid-state enthalpy of formation for neutral compounds can be estimated using Trouton's rule according to Equation 2 [17], where T is either the melting point or the decomposition temperature (in K) if no melting occurs prior to decomposition.

$$\Delta_f H°_{(s)} = \Delta_f H°_{(g)} - \Delta_{sub} H = \Delta_f H°_{(g)} - (188\ \text{J·mol}^{-1}\text{·K}^{-1} \times T) \quad (2)$$

Finally, the solid-state molar enthalpies of formation ($\Delta_f H°$) were used to

calculate the solid-state energies of formation ($\Delta_f U°$) according to Equation 3, with Δn being the change in the number of moles of the gaseous components.

$$\Delta U = \Delta H - \Delta nRT \quad (3)$$

The results of the calculations are compiled in Table 1. As expected, the C–N linked isomer **5** shows a higher enthalpy of formation (819.0 kJ·mol^{-1}) than the C–C linked isomer AzTT (779.5 kJ·mol^{-1}).

Table 1. CBS-4M calculation results

M	PG [a]	$-H$ / a.u. [b]	$\Delta_f H°_{(g)}$, [kJ·mol^{-1}] [c]	$-\Delta n$ [d]	$\Delta_f H°_{(s)}$, [kJ·mol^{-1}][e]	$\Delta_f U°_{(s)}$, [kJ·kg^{-1}] [f]
AzTT	C_1	662.043029	859.5	6.0	779.5	4460.2
5	C_1	662.028006	898.9	6.0	819.0	4681.6
H		0.500991				
C		37.786156				
N		54.522462				
O		74.991202				

[a] Point group, [b] CBS-4M calculated enthalpy, [c] gas-phase enthalpy of formation, [d] change in moles of gaseous components, [e] solid-state enthalpy of formation, [f] solid-state energy of formation.

AzTT and AzTTO [18], together with other *C*-azido-1,2,4-triazoles, like AzNT [10] and 5-azido-1*H*-1,2,4-triazole-3-carbonitrile [19], are able to form ionic primary explosives when paired with metal cations like silver. Compound **5** could unfortunately not be tested in this regard, due to our failed attempts at preparing a silver salt, which always resulted in a brown slurry.

3 Conclusions

The goal of the present study has been the preparation and characterization of the energetic nitrogen-rich heterocycle 1-(5-azido-1*H*-1,2,4-triazol-3-yl) tetrazole (**5**). The potential for salt formation was tested with silver nitrate, but unfortunately this always resulted in a brown slurry. The ability to form ionic primary explosives was therefore not further investigated. It is nevertheless quite interesting to see that the linkage to the azide is apparently still weaker than that to the C–N bonded tetrazole, resulting in practically identical decomposition onsets for AzTT, AzTTO and **5** (around 150 °C), independent of the type and linkage

of the tetrazole ring. The sensitivities towards external stimuli are comparable for the three compounds.

4 Experimental Section

All chemicals were used as supplied (ABCR, Acros Organics, AppliChem, Sigma-Aldrich, VWR), if not stated otherwise.

NMR spectra were recorded using the spectrometers JEOL Eclipse 270, Eclipse 400 and JEOL ECX 400. The measurements were conducted in regular glass NMR tubes (Ø 5 mm) and, if not stated otherwise, at 25 °C. Tetramethylsilane (^{1}H, ^{13}C) and nitromethane ($^{14/15}$N) were used as external standards. As an additional internal standard the reference values of the partially deuterated solvent impurity (^{1}H) and the fully deuterated solvent (^{13}C) were used [20]. Infrared (IR) spectra were recorded on a PerkinElmer BX FT IR spectrometer equipped with a Smiths DuraSamplIR II diamond ATR unit using pure samples. Transmittance values are qualitatively described as "very strong" (vs), "strong" (s), "medium" (m) and "weak" (w). Raman spectra were recorded on a Bruker RAM II spectrometer equipped with a Nd:YAG laser (300 mW) operating at 1064 nm and a reflection angle of 180°. The intensities are reported as percentages of the most intense peak and are given in parentheses. Low resolution mass spectra were recorded on a JEOL MStation JMS-700. The determination of the carbon, hydrogen and nitrogen contents (EA analysis) was carried out by combustion analysis using an Elementar Vario EL. The determined nitrogen values are often lower than the calculated ones. This is common with nitrogen-rich compounds and cannot be avoided. Differential scanning calorimetry was conducted with a Linseis DSC-PT10 in closed aluminum pans, equipped with a hole (Ø 0.1 mm) for gas release, and at a heating rate of 5 °C min^{-1}. Melting points were checked with a Büchi Melting Point B-540 apparatus, in open glass capillaries.

The sensitivities to impact (IS) and friction (FS) were determined according to BAM [21] standards using a BAM drop hammer (100 cm maximum drop height; 1, 5 and 10 kg weights; compound contained between two steel cylinders held together by a steel ring) and a BAM friction apparatus (5 to 360 N range) [22]. The compounds were sieved to determine the grain size (< 100 µm, 100 to 500 µm, > 500 µm).

Caution! The title compound **5**, as prepared here, is an energetic compound sensitive to impact and friction. Although there were no problems in handling the compound, proper protective measures (ear protection, Kevlar® gloves, face

shield, body armor and earthed equipment) should be used.

1-Acetyl-3,5-diamino-1,2,4-triazole (1): Cyanoguanidine (85.0 g, 1.00 mol) and hydrazinium dichloride (105 g, 1.00 mol) were dissolved in water (200 mL) and stirred for four hours at 60 °C. The reaction mixture was neutralized with a solution of sodium acetate (90.4 g, 1.10 mol) in water (300 mL). Acetic anhydride (123 g, 1.20 mol) was then added dropwise at room temperature and the suspension was stirred for one hour. The precipitate was filtered off, washed with water and ethanol, and then suspended in ethanol and refluxed for five minutes. The precipitate was filtered off to yield a colorless powder (89.7 g, 636 mmol, 64%). **EA** ($C_4H_7N_5O$): calcd. C 34.04, H 5.00, N 49.62%; found C 33.89, H 4.81, N 49.59%. **^{1}H NMR** (DMSO-d_6): δ = 7.38 (s, 2H, NH_2), 5.66 (s, 2H, NH_2), 2.34 (s, 3H, CH_3). **^{13}C NMR** (DMSO-d_6): δ = 170.5, 162.1, 157.0, 23.5. **IR** (ATR): ν = 3415 (w), 3389 (m), 3297 (w), 3130 (m), 1710 (s), 1641 (vs), 1568 (s), 1449 (m), 1392 (s), 1366 (vs), 1337 (s), 1178 (m), 1117 (m), 1066 (m), 1044 (s), 973 (s), 839 (m), 758 (m), 699 (m), 670 (w), 656 (s) cm^{-1}. **MS** (DEI+): *m*/*z* = 141.2 (20, $[M]^+$), 99.2 (100, $[M-C_2H_3O]^+$). **DSC** (5 °C min^{-1}): T_r = 213 °C, T_d = 269 °C.

5-Acetamido-3-amino-1*H*-1,2,4-triazole (2): A suspension of **1** (60.0 g, 425 mmol) in decalin (750 mL) was heated for seven hours at 180 °C without stirring. The precipitate was filtered off and washed with ethanol and diethyl ether to yield a very fine, pale brown powder (59.6 g, 422 mmol, 99%). **EA** ($C_4H_7N_5O$): calcd. C 34.04, H 5.00, N 49.64%; found C 34.23, H 4.87, N 49.46%. **IR** (ATR): ν = 3422 (w), 3250 (m), 2953 (w), 2869 (w), 2824 (w), 2166 (w), 1682 (s), 1597 (vs), 1582 (vs), 1450 (s), 1375 (m), 1360 (m), 1295 (s), 1267 (s), 1159 (w), 1080 (s), 1041 (w), 1024 (m), 1006 (m), 833 (w), 817 (w), 759 (w), 712 (s), 687 (m) cm^{-1}. **DSC** (5 °C min^{-1}): T_d = 273 °C.

1-(5-Acetamido-1*H*-1,2,4-triazol-3-yl)tetrazole (3): Acetic acid (350 mL) was added in one portion to a stirred mixture of **2** (35.3 g, 250 mmol), triethyl orthoformate (55.6 g, 365 mmol) and sodium azide (19.5 g, 300 mmol). A reflux condenser was attached and the resulting suspension was stirred first for 72 hours at room temperature, and then for 20 hours at 90 °C. Concentrated hydrochloric acid (30 mL) was added after cooling to room temperature (evolution of HN_3!) and stirred for one hour. The solvent was removed under reduced pressure, the residue was fully suspended in toluene (about 150 mL) and stirred for about one hour, then again evaporated again to dryness. This procedure was repeated once more. The residue was suspended in water (250 mL) and refluxed until the

solid was fully suspended. The precipitate was filtered off, washed with water and dried at 102 °C to yield a pale brown powder (35.1 g, 181 mmol, 72%). **EA** ($C_5H_6N_8O$): calcd. C 30.93, H 3.11, N 57.71%; found C 32.11, H 3.67, N 51.26%. **IR** (ATR): ν = 3315 (w), 3230 (m), 3107 (m), 1687 (vs), 1589 (vs), 1577 (vs), 1559 (vs), 1530 (s), 1492 (s), 1367 (s), 1324 (w), 1263 (s), 1248 (vs), 1188 (w), 1160 (w), 1117 (w), 1088 (s), 1068 (m), 1040 (w), 1014 (m), 1003 (m), 979 (m), 953 (w), 906 (w), 791 (m), 777 (w), 737 (s), 690 (w), 656 (w) cm^{-1}. **MS** (DEI+): *m*/*z* = 195.1 (2, $[M]^+$).

1-(5-Amino-1*H*-1,2,4-triazol-3-yl)tetrazole (4): Compound **3** (35.1 g, 181 mmol) and hydrazine hydrate (36.2 g, 724 mmol) were stirred for three hours at 100 °C, then cooled down. Water (30 mL) was added and the solution was acidified with hydrochloric acid (10%) to pH 6. The suspension was stirred for 30 minutes, after which the precipitate was filtered off and washed with moderate amounts of water, ethanol and diethyl ether, then finally dried at 102 °C to yield a pale brown powder (5.22 g, 34.3 mmol, 19%). **EA** ($C_3H_4N_8$): calcd. C 23.69, H 2.65, N 73.66%; found C 24.16, H 3.15, N 70.50%. **^{1}H NMR** (DMSO-d_6): δ = 11.88 (vbr, N*H*), 9.91 (s, 1H, C*H*), 6.66 (s, 2H, NH_2). **^{13}C NMR** (DMSO-d_6): δ = 157.6 (C2), 150.1 (C1), 142.7 (C3). **IR** (ATR): ν = 3385 (w), 3257 (w), 3160 (w), 3097 (w), 1650 (vs), 1570 (s), 1518 (vs), 1452 (m), 1423 (w), 1330 (w), 1275 (m), 1192 (w), 1163 (w), 1096 (m), 1078 (m), 1021 (w), 980 (s), 960 (w), 897 (w), 755 (w), 724 (w), 707 (w) cm^{-1}. **MS** (DEI+): *m*/*z* = 153.2 (3, $[M]^+$).

1-(5-Azido-1*H*-1,2,4-triazol-3-yl)tetrazole (5): Sodium nitrite (414 mg, 6.00 mmol) in water (10 mL) was added dropwise at 0 °C to a suspension of **4** (761 mg, 5.00 mmol) in sulfuric acid (25%, 30 mL). The suspension was allowed to warm to room temperature, followed by stirring for 30 minutes at 40 °C. A small amount of urea was added after cooling to room temperature, followed by the dropwise addition of sodium azide (1.30 g, 20.0 mmol) in water (10 mL). The reaction mixture was stirred overnight, then extracted with ethyl acetate (3 × 75 mL). The combined organic phases were dried over magnesium sulfate, and then concentrated under reduced pressure. The residue was taken up in a minimal amount of ethyl acetate, mixed with *n*-pentane (about 200 mL) and the resulting precipitate was filtered off to yield a colorless solid (629 mg, 3.53 mmol, 71%). **EA** ($C_3H_2N_{10}$): calcd. C 20.23, H 1.13, N 78.64%; found C 20.63, H 1.71, N 73.66%. **^{1}H NMR** (DMSO-d_6): δ = 10.10 (s, C*H*). **^{13}C NMR** (DMSO-d_6): δ = 151.6 (C2), 150.4 (C1), 142.9 (C3). **^{14}N NMR** (DMSO-d_6): δ = −139 (N9). **^{15}N NMR** (DMSO-d_6): δ = 12.0 (d, $^3J_{NH}$ = 3.43 Hz, N6), −20.2 (N5), −52.4 (d, $^2J_{NH}$ = 12.11 Hz, N7), −119.4 (N2), −143.8 (N9/N10), −146.0

(N10/N9), −147.3 (d, $^2J_{NH}$ = 9.29 Hz, N4), −166.8 (N3), −185.7 (N1), −293.8 (N8). **IR** (ATR): ν = 3310 (m), 3167 (m), 3080 (m), 2932 (w), 2856 (w), 2237 (w), 2156 (vs), 1646 (w), 1572 (m), 1552 (vs), 1534 (vs), 1510 (vs), 1493 (vs), 1440 (m), 1411 (m), 1391 (m), 1353 (m), 1329 (m), 1312 (m), 1286 (m), 1266 (s), 1257 (s), 1191 (s), 1174 (m), 1160 (m), 1107 (m), 1088 (vs), 1043 (m), 1030 (m), 1006 (m), 982 (m), 975 (m), 965 (m), 917 (w), 870 (w), 792 (m), 721 (s), 682 (w), 667 (w) cm^{-1}. **Raman** (300 mW): 3168 (6), 2166 (15), 1572 (100), 1559 (10), 1535 (5), 1508 (11), 1461 (8), 1398 (22), 1330 (10), 1264 (14), 1255 (5), 1193 (5), 1175 (6), 1090 (7), 1035 (6), 1011 (29) cm^{-1}. **MS** (DEI+): *m*/*z* = 178.2 (2, $[M]^+$). **DSC** (5 °C min^{-1}): T_d = 152 °C. **Sensitivities** (grain size < 100 µm): IS 4 J, FS 10 N.

Acknowledgement

Financial support of this work by the Ludwig-Maximilian University of Munich (LMU), the U.S. Army Research Laboratory (ARL), the Armament Research, Development and Engineering Center (ARDEC), the Office of Naval Research (ONR) under grant no. ONR.N00014-12-1-0538, and the Bundeswehr – Wehrtechnische Dienststelle für Waffen und Munition (WTD 91) under grant no. E/E91S/FC015/CF049 is gratefully acknowledged. The authors acknowledge collaborations with Dr. Mila Krupka (OZM Research, Czech Republic) in the development of new testing and evaluation methods for energetic materials and with Dr. Muhamed Sućeska (Brodarski Institute, Croatia) in the development of new computational codes to predict the detonation and propulsion parameters of novel explosives. We are indebted to and thank Drs. Betsy M. Rice, Jesse Sabatini and Brad Forch (ARL, Aberdeen, Proving Ground, MD) for many inspired discussions. Stefan Huber is thanked for assistance during sensitivity measurements.

5. References

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