

## **Dimethoxycarbonyl Groups Surrounding a Symmetric** Diaminobistetrazole Ring: Exploring New Green Energetic Materials

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Abstract: A novel oxygen-containing dimethoxycarbonyl diaminobistetrazole (1) was synthesized via a facile strategy. The sodium salt (2) based on this ligand was prepared and these two compounds were fully characterized by using elemental analysis, IR and mass spectrometry and single-crystal X-ray diffraction. Their density, heats of formation, thermal stability and sensitivity, as well as the energetic properties from EXPLO5 code were investigated. These newly synthesized compounds possess high positive heats of formation and detonation heats. Compound 1 exhibits good detonation performance and acceptable stability, and might be a potential eco-friendly alternative of lead azide. The present study contributes to the development of tetrazole derivatives as new energetic materials.

Energetic materials play an important role in both military and civilian applications because they are well-known used as all kinds of explosives, propellants and pyrotechnics.<sup>[1,2]</sup> As typical examples of high nitrogen-containing energetic compounds,<sup>[3]</sup> the development of tetrazoles and their derivatives has been rapidly benefiting from the efforts of synthetic and theoretical researchers over the past few years.<sup>[4-9]</sup> These tetrazoles have characteristics of large number of NN and CN bonds, deriving most of their energy from the high positive heats of formation rather than from the oxidation of the carbon backbone in traditional energetic compounds. This high nitrogen content also leads to high densities, and the low amount of carbon and hydrogen, which allows for a good oxygen balance to be achieved more easily.<sup>[10]</sup> Compared to the mono-tetrazole, the bistetrazoles<sup>[11-18]</sup> have additional advantages owing to the higher

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thor(s) of this article can be found under:

https://doi.org/10.1002/asia.201901271.

Chem. Asian J. 2019, 00, 0-0

nitrogen content and heats of formation, thus exhibit better stability and explosive performance (Figure 1).

The methoxycarbonyl is an important kind of organic groups, and its derivatives can be used as intermediates in pesticides, medicines and organic synthesis.<sup>[19,20]</sup> In addition, the high oxygen content in the molecule could contribute to the good oxygen balance (OB),<sup>[21]</sup> which is a key indicator for evaluating the energy of an explosive. However, the chemistry of methoxycarbonyl introduced in energetic materials remains unexplored so far. Thus incorporating this oxygen-containing group into the bistetrazole skeleton to improve the overall performance will be of interest as well as challenge.

In a continuing effort to develop new eco-friendly energetic materials, we directed our attention to design and synthesis of tetrazole-based derivatives. In this work, a novel dimethoxycarbonyl diaminobistetrazole and its sodium salt were readily synthesized firstly. All new compounds were fully characterized by elemental analysis, IR and mass spectrometry and single-crystal X-ray diffraction analysis. And their physicochemical and energetic properties were studied as well. The present study contributes to the tetrazole system and provides a new insight in the design and synthesis of new energetic materials.

#### **Syntheses**

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The synthesis pathways of dimethoxycarbonyl diaminobistetrazole (1) and its sodium salt (2) are shown in Scheme 1. The commercially available methyl carbazate was reacted with glyoxal, NCS, and sodium azide in sequence at room temperature, then the obtained solid was stirred in diethyl ether with



Scheme 1. Synthetic pathway towards compound 1 and 2.

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Figure 1. Selected bistetrazoles as energetic compounds.

saturated HCl gaseous for three days to get compound **1**. The preparation of sodium salt **2** was achieved by straightforward metathesis reactions that used **1** with the corresponding base NaOH. These pure materials were slowly crystallized from water in a colorless block, and their structures were fully characterized by elemental analysis, mass spectrometry, IR spectrometry and single-crystal X-ray diffraction. The crystallographic data are given in Table S1 (Supporting Information).

#### **Crystal structures**

Compound 1 crystallizes in the monoclinic P2(1)/c space group (Figure 2a), with two molecular moieties in the unit cell and a density of 1.640 g cm<sup>-3</sup> at 298 K. Both of the tetrazole rings can be considered planar (N1-N2-N3-N4  $-0.8(2)^\circ$ , C1-N1-N2-N3  $0.5(2)^\circ$ ), while the methoxycarbonyl shows a certain distortion of the plane. The C1–C1 bond length of the linkage connecting the two tetrazole moieties is 1.450(4) Å and the CO bond lengths of the substituted group are 1.317(2) (O1–C2), 1.444(3)

(O1–C3) and 1.184(2) Å (O2=C2), respectively. The extensive hydrogen bonded network starts from N5 atom of the imino, respectively linked to neighboring O2 and N3 atom of the methoxycarbonyl and the tetrazole (N5-H5--O2: 126°, H--O: 2.835(2) Å and N5-H5--N3: 130°, H--N: 3.134(3) Å) (Figure 2b). These hydrogen bond interactions form a reticular framework of 1, which could be a key factor for the good thermal stability.

Compound **2** crystallizes in the monoclinic *P2/c* space group, with two molecules in the unit cell and the asymmetry unit is composed of two sodium ions and one dimethoxycarbonyl diaminobistetrazole anion (Figure 3 a). In its anionic structure, the tetrazole rings show a dihedral angle N1-N2-N3-N4of 172.6(7)°, and the bond length of connecting bond (C2– C2) is 1.480(17) Å. The substituted group is also twisted out of the tetrazole plane (N1-N2-C2-N5 –171.8(8)°). The sodium center atom is six-coordinated by two oxygen atoms from the dimethoxycarbonyl with Na–O bond length of 2.423(6) Å and four oxygen atoms from the water molecule with bond lengths



Figure 2. Crystal structure of the compound 1: (a) molecular unit (b) unit cell view along b axis with hydrogen bonds.



Figure 3. Crystal structure of the compound 2: (a) molecular unit (b) coordination octahedron.

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© 2019 Wiley-VCH Verlag GmbH & Co. KGaA, Weinheim **K** These are not the final page numbers! ranging from 2.371(7) Å to 2.451(7) Å (Figure 3 b). This sodium salt shows a 1D chain structure with alternately assembling units and an adjacent Na…Na distance of 3.176(3) Å (Na1-Na1).

#### **Thermal behavior**

The thermal behaviors of newly synthesized compounds were determined using differential scanning calorimetry measurements (DSC) at a heating rate of  $5^{\circ}$ Cmin<sup>-1</sup>. And the summarized DSC plot is depicted in Figure 4. Melting points are not observed both in compound 1 and 2, indicating their direct exothermic decomposition without the melting process. Compound 1 has an exothermic peak at 191°C, but compound 2 exhibits a lower decomposition temperature of 102°C. This result shows that the thermal stability of sodium salt decreases relative to the neutral compound, which is caused by the strong intermolecular H-bond interactions in 1 compared to 2.



Figure 4. DSC curves at a heating rate of 5 °C for compound 1 and 2.

#### **Energetic Properties**

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The energetic Properties of the synthesized compounds were investigated and the results are summarized in Table 1. Impact and friction sensitivities were measured by standard BAM techniques and classified according to the "U.N. Recommendations on the Transport of Dangerous Goods".<sup>[22]</sup> Compound 1 (IS=1 J, FS = 14 N) can be classified as very sensitive and the sodium salt 2 (IS=40 J; FS=360 N) can be classified as insensitive. Compounds 1 and 2 have high nitrogen content (exceeding 40%) and negative oxygen balances. The neutral compound 1 exhibits good thermal stability with a decomposition temperature of 191 °C, whereas the metal salt 2 has a higher density of 1.65 g cm<sup>-3</sup>. The results of heats of formation calculated based on isodesmic reactions indicate that they have positive heats of formation (325.5 and 232.8 kJ mol<sup>-1</sup>) and are significantly greater than that of TNT. The detonation parameters were calculated using the EXPLO5 code<sup>[23]</sup> using a calculated heat of formation and density. Both of compounds 1 and 2 show good detonation heats (3905 and 4762 kJ kg<sup>-1</sup>), which are higher than that of Pb(N<sub>3</sub>). The calculated detonation velocity (7403 and 7041 m s<sup>-1</sup>) and detonation pressure (20.1 and 17.5 GPa) imply that these new compounds outperform commonly used explosive TNT. Thereinto, the dimethoxycarbonyl diaminobistetrazole (1) is comparable to the primary explosive lead azide

Table 1. Physiochemical and energetic properties of compounds 1 and 2.							
Compound	1	2	TNT <sup>[24]</sup>	Pb(N <sub>3</sub> ) <sup>[16]</sup>			
/S [J] <sup>[a]</sup>	1	40	39	2.5–4			
FS [N] <sup>[b]</sup>	14	360	353	0.1-1			
$arOmega$ co $_2$ [%] <sup>[c]</sup>	-67.5	-53.6	-74.0	-11.0			
N [%] <sup>[d]</sup>	49.2	42.6	18.5	28.9			
T <sub>dec</sub> [°C] <sup>[e]</sup>	191	102	295	315			
$ ho$ [g cm $^{-3}$ ] <sup>[f]</sup>	1.64	1.65	1.65	4.8			
$\Delta_{ m f} {\cal H}_{ m m}^{\circ}$ [kJ mol $^{-1}$ ] <sup>[g]</sup>	325.5	232.8	-67.0	450.1			
$\Delta_{ m f} U^\circ ~ [ m kJkg^{-1}]^{[ m h]}$	1023.0	888.5	-415.2	1574.9			
EXPLO5 6.01:							
$-\Delta_{\scriptscriptstyle \! E\!x}\!H^\circ[ m kJkg^{-1}]^{[i]}$	3905	4762	5042	1569			
$T_{det}^{[j]}$	2862	3148	-	3401			
P <sub>CJ</sub> [GPa] <sup>[k]</sup>	20.1	17.5	19.5	33.8			
$V_{\rm det}[{ m m  s^{-1}}]^{[l]}$	7403	7041	6881	5920			
$V_0[L kg^{-1}]^{[m]}$	774	522	825	252			

[a] Impact sensitivity. [b] Friction sensitivity. [c] Oxygen balance for CaHbOcNd: 1600(c-2a-b/2)/MW. [d] Nitrogen content. [e] Decomposition temperature. [f] Density at RT. [g] Heat of formation. [h] Energy of formation. [i] Heat of detonation. [j] Detonation temperature. [k] Detonation velocity. [l] Detonation pressure. [m] Volume of detonation gases (assuming only gaseous products). MW=molecular weight.

and could be its potential alternative from an environmental point of view.

#### **Computational considerations**

To better understand the electronic properties, geometric structure, HOMO–LUMO orbitals and electrostatic potential (ESP) of **1** was calculated using the density functional theory (DFT) with B3LYP/6-31 +  $G^{**}$  method. Compound **1** shows a bitetrazole linked by the C–C bond and two dimethoxycarbonyl groups twisted out from the tetrazole plane with a torsion angle (Figure 5a). The optimized geometrical parameters are similar to the experimental values and tabulated in Table S4. The HOMO of the molecule is localized both on tetrazole rings and substituted groups, while the LUMO is distributed on the rings as well as the C=C bond (Figure 5b). The energy gap be-



**Figure 5.** DFT calculations for compound 1: (a) optimized structure (b) HOMO–LUMO (3) electrostatic potential.

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tween the HOMO and LUMO is 5.54 eV, which shows molecular stable nature. As can be seen from ESP map (Figure 5 c), the regions having the negative potential are over the electronegative oxygen and nitrogen atom, which are preferred sites for the electrophilic attack indications. The regions having the positive potential are over the hydrogen atom, which are preferred sites for the nucleophilic attack symptoms.

In summary, we prepared a novel dimethoxycarbonyl diaminobistetrazole in a straightforward manner. Computational analysis was used to reveal the stability and electronic properties of this new molecule. The sodium salt based on this ligand was also synthesized and these two compounds were fully characterized by elemental analysis, IR spectroscopy, mass spectrometry and single-crystal X-ray diffraction. Compound 1 shows a reticular network with hydrogen bond interactions, and compound 2 features a 1D framework structure benefiting from the characteristic coordination modes. Their thermal stabilities were determined by DSC, and the energetic properties were evaluated by the EXPLO5 code as well as the BAM test. Both of them exhibit high heats of formation (up to 325.5 kJ mol<sup>-1</sup>) and detonation heats (up to 4762 kJ kg<sup>-1</sup>). The detonation performance of 1 is higher than that of the primary explosive lead azide and might serve as its eco-friendly alternative.

## **Experimental Section**

#### **General procedures**

All chemical reagents and solvents were obtained from Sinopharm Chemical Reagent Co., Ltd. Decomposition point was determined by differential scanning calorimetry (DSC) on a model Pyris-1 at a heating rate of  $5\,^\circ\text{Cmin}^{-1}$ . Infrared spectra were recorded on a Bruker Equinox 55 spectrometer. Mass spectra of the described compounds were measured at a Agilent 500-MS. Elemental analysis was performed on an Elementar Vario El III analyzer. The impact and friction sensitivities were performed on a BAM fall hammer BFH-10 and a BAM friction apparatus FSKM-10, respectively. Collection of XRD data was performed on a Rigaku Saturn 724 + CCD diffractometer equipped with graphite monochromatized  $Mo_{K\alpha}$  radiation. The structure was solved by using direct methods and successive Fourier difference syntheses (SHELXS-97)<sup>[25]</sup> were refined by using full-matrix least-squares on F2 with anisotropic thermal parameters for all non-hydrogen atoms (SHELXL-97).<sup>[26]</sup> Hydrogen atoms were added theoretically and refined with riding model position parameters and fixed isotropic thermal parameters.

CCDC 1492730 (1) and 1492731 (2) contain the supplementary crystallographic data for this paper. These data are provided free of charge by The Cambridge Crystallographic Data Centre.

#### Dimethoxycarbonyl diaminobistetrazole (1)

The commercially available methyl carbazate (4.50 g, 50 mmol) was reacted with the glyoxal (1.45 g, 25 mmol) at room temperature to get the dimethoxycarbonylglyoxal bishydrazone. Then this compound was suspended in 300 mL DMF, added the NCS (20 g, 150 mmol) and the mixture was stirred overnight at room temperature and filtered to obtain the dichloro product. This dichloro product was suspended in 10 mL DMF and cooled to 0-5 °C with the sodium azide (650 mg, 10 mmol) added. The mixture was

stirred overnight at room temperature, then was diluted with 30 mL of ice-water and filtered to get the diazido product. This solid was suspended in 50 mL of diethyl ether with saturated HCI gaseous cooled with an ice bath at 5 °C. The flask was sealed and stirred at room temperature for three days. The solvent was removed and the solid recrystallized from methanol yielding new compound 1 as colorless plates.

**DSC** ( $5 \,^{\circ}$ Cmin<sup>-1</sup>): 191  $^{\circ}$ C (dec); **IR** (KBr):  $\tilde{\nu} = 3413$  (m), 3472 (w), 3220 (m), 3170 (m), 3133 (m), 3005 (w), 2956 (w), 2544 (w), 2326 (w), 2252 (w), 2158 (s), 2135 (s), 1704 (s), 1598 (s), 1528 (w), 1469 (s), 1345 (s), 1194 (w), 1153 (s), 1085 (s), 1014 (m), 881 (w), 786 (w), 764 (m), 683 (m), 663 (w), 608 (m), 574 (w), 547 (m), 511 cm<sup>-1</sup> (m); **MS** (ESI<sup>-</sup>): *m/z*: 285.08 (C<sub>6</sub>H<sub>8</sub>N<sub>10</sub>O<sub>4</sub>); **EA**: calc: C 25.36%, N 49.29%, 2.84%; found: C 25.75%, N 50.42%, 2.87. **BAM drop hammer**: 1 J; friction tester: 14 N.

#### Sodium dimethoxycarbonyl diaminobistetrazolate (2)

Powdered colorless 1 (0.28 g, 1 mmol) was dissolved in the deionized water, and sodium hydroxide (0.08 g, 2 mmol) was added. After the mixture had been stirred several minutes, the filtrate was slowly evaporated to leave the solid product **2** as colorless blocks. **DSC** (5 °C min<sup>-1</sup>): 102 °C (dec); **IR** (KBr):  $\vec{\nu}$  = 3440 (s), 3013 (w), 2955 (w), 2920 (w), 2850 (w), 2138 (m), 2108 (m), 1708 (s), 1581 (s), 1540 (s), 1446 (s), 1351 (w), 1313 (s), 1196 (w), 1157 (w), 1105 (w), 1072 (m), 1004 (m), 879 (w), 823 (w), 767 (w), 693 (w), 640 (w), 611 (w), 574 (w), 548 (w), 463 (w), 420 cm<sup>-1</sup> (w); **MS** (ESI<sup>-</sup>): *m/z*: 285.1 (C<sub>6</sub>H<sub>6</sub>N<sub>10</sub>O<sub>4</sub><sup>2-</sup>); **EA**: calc: C 21.96%, N 42.68%, 1.84%; found: C 21.93%, N 42.66%, 1.82%. **BAM drop hammer**: 40 J; friction tester: 360 N.

#### **Computational Details**

The geometric optimization and frequency analyses were carried out by using B3LYP functional<sup>[27-29]</sup> analyses with the 6-31+G\*\* basis set. The optimized structures were characterized as true local energy minima on the potential energy surface without imaginary frequencies. The electrostatic potential and HOMO–LUMO orbitals were calculated at the same level of theory based on the optimized structures. Single energy points were calculated at the MP2/ 6-311 + + G\*\* level. All computations were performed by using the Gaussian 09 suites of programs.<sup>[30]</sup>

### Acknowledgements

The supports of the Central South University Science Research Initial Fun and the opening project of State Key Laboratory of Explosion Science and Technology (Beijing Institute of Technology) (KFJJ19-07M) are gratefully acknowledged.

## **Conflict of interest**

The authors declare no conflict of interest.

**Keywords:** detonation performance · DFT calculations · dimethoxycarbonyl diaminobistetrazole · energetic materials

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Manuscript received: September 9, 2019 Revised manuscript received: September 20, 2019 Version of record online:

Chem. Asian J. **2019**, 00, 0–0

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# COMMUNICATION

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**An oxygen-containing** dimethoxycarbonyl diaminobistetrazole and its sodium salt were synthesized and fully characterized. Their properties were extensively investigated by a combination of experimental and computational methods. This compound exhibits good detonation performance and acceptable stability, and might be a potential ecofriendly alternative of lead azide.

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