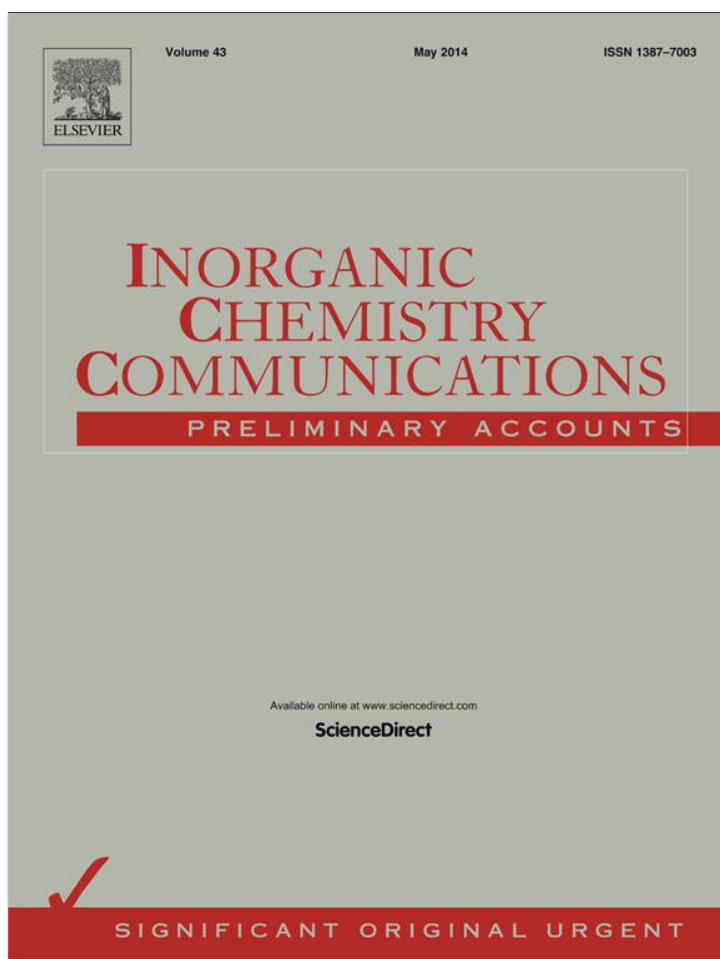


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# Synthesis, supramolecular structure, and energetic properties of the first metal–organic nitrotetrazolate, $[\text{Me}_3\text{Sn}(\mu\text{-OH})\text{SnMe}_3(\mu\text{-OH})\text{SnMe}_3(\text{H}_2\text{O})][\text{NT}]$ (NT = 5-nitrotetrazolate)

Gesine Stock<sup>a</sup>, Cristian G. Hrib<sup>a</sup>, Raik Deblitz<sup>b</sup>, Marcel Kühling<sup>a</sup>, Georg Plenikowski<sup>b</sup>, Frank T. Edelmann<sup>a,\*</sup><sup>a</sup> *Chemisches Institut der Otto-von-Guericke-Universität Magdeburg, 39106 Magdeburg, Germany*<sup>b</sup> *Nammo Germany GmbH, Wilhelm-Dümling-Str. 12, 39218 Schönebeck, Germany*

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## ABSTRACT

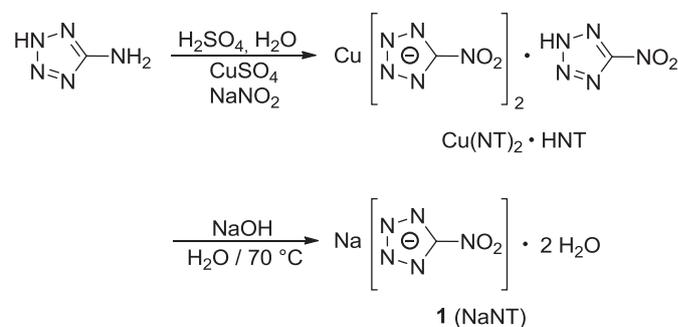
The first metal–organic 5-nitrotetrazolate,  $[\text{Me}_3\text{Sn}(\mu\text{-OH})\text{SnMe}_3(\mu\text{-OH})\text{SnMe}_3(\text{H}_2\text{O})][\text{NT}]$  (**2**), has been prepared by a simple metathetical reaction between  $\text{Me}_3\text{SnCl}$  and sodium 5-nitrotetrazolate dihydrate (= NaNT, **1**). A single-crystal X-ray diffraction study revealed the presence of the trinuclear  $[\text{Me}_3\text{Sn}(\mu\text{-OH})\text{SnMe}_3(\mu\text{-OH})\text{SnMe}_3(\text{H}_2\text{O})]^+$  cation to which the 5-nitrotetrazolate anion is coordinated via a ring-N atom. The NT anion is further engaged in four different O–H⋅N and O–H⋅N hydrogen bonds involving the remaining three ring-nitrogen atoms and one oxygen of the nitro group, leading to an extensive supramolecular hydrogen-bonded network in the solid state. Despite its very low N content of only 10.65%, compound **2** is highly impact-sensitive (<2.5 J) and can be classified as a primary explosive.

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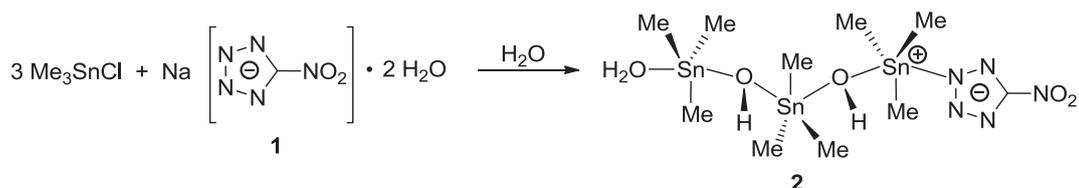
Primary explosives are highly sensitive explosive compounds which are used to initiate large amounts of secondary explosives such as 2,4,6-trinitrotoluene (= TNT) [1–3]. Historically, one of the first practical primary explosives was mercury fulminate,  $\text{Hg}(\text{CNO})_2$  (“Knallquecksilber”), which has been widely used for almost 100 years [4]. It was later replaced by lead(II) azide,  $\text{Pb}(\text{N}_3)_2$ , which is being generally used as primary explosive until today [1,5,6]. However, lead(II) azide inherits the severe disadvantages that highly toxic hydroazoic acid,  $\text{HN}_3$ , can be formed under certain conditions [5,6], and that its use is always associated with lead pollution of the environment [7]. For this reason, the search for environmentally acceptable (“green”) primary explosives is currently a hot topic in this field [8]. One of the most promising approaches is the development of nitrogen-rich energetic compounds based on tetrazole and tetrazine derivatives [9–11]. Of particular interest among these are explosive materials comprising the 5-nitrotetrazolate anion,  $[\text{CN}_4\text{NO}_2]^-$  (= NT). Various salts [12–16] and transition metal complexes [17–23] containing nitrotetrazolate have been prepared and tested. Perhaps the most promising candidate to come out of these research efforts is the recently reported copper(I) 5-nitrotetrazolate (= DBX-1) [24]. The easily prepared DBX-1 has been shown to be one of the best “drop-in” lead(II) azide replacements to date due to its high thermal stability and equivalent safety and performance properties. The preparation of DBX-1 involves treatment of copper(II) chloride with sodium 5-nitrotetrazolate dihydrate (= NaNT, **1**) in the presence of sodium ascorbate as reducing agent. The synthesis

of the important precursor NaNT (**1**) has been investigated in great detail [16,25–27]. To our best knowledge, no metal–organic main-group or organotransition metal derivatives of the NT anion have ever been reported in the literature. We describe here the synthesis, structural characterization, and energetic properties of the title compound,  $[\text{Me}_3\text{Sn}(\mu\text{-OH})\text{SnMe}_3(\mu\text{-OH})\text{SnMe}_3(\text{H}_2\text{O})][\text{NT}]$  (**2**), as the first example of a metal–organic main-group metal nitrotetrazolate.

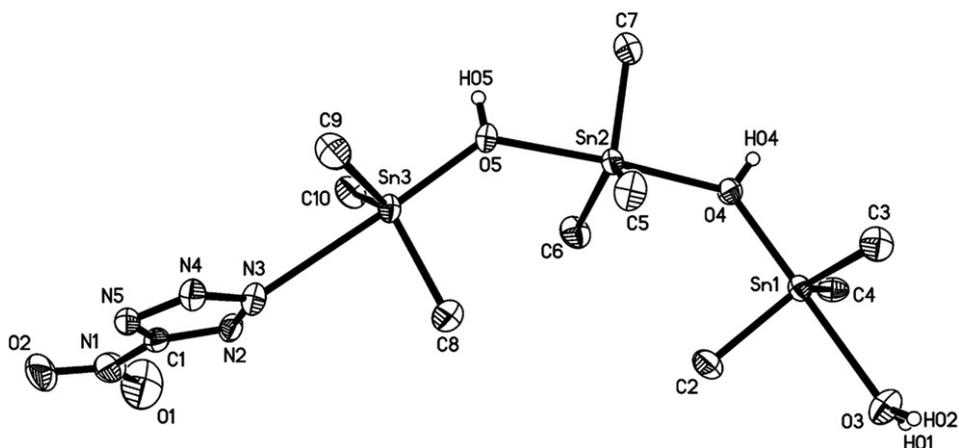
Sodium 5-nitrotetrazolate dihydrate (= NaNT, **1**), was prepared by a modified literature method according to Scheme 1 [16,28,29]. The preparation involves a modified Sandmeyer reaction (diazotation) of 5-aminotetrazole in the presence of copper(II) sulfate. In the first step, a pale blue, very explosive (especially when dry) intermediate of the composition  $\text{Cu}(\text{NT})_2 \cdot \text{HNT}$  is formed, which is kept in a wet state and

Scheme 1. Preparation of the starting material NaNT (**1**).

\* Corresponding author. Tel.: +49 391 6758327; fax: +49 391 6712933.  
E-mail address: [frank.edelmann@ovgu.de](mailto:frank.edelmann@ovgu.de) (F.T. Edelmann).



**Scheme 2.** Preparation of  $[\text{Me}_3\text{Sn}(\mu\text{-OH})\text{SnMe}_3(\mu\text{-OH})\text{SnMe}_3(\text{H}_2\text{O})][\text{NT}]$  (**2**).



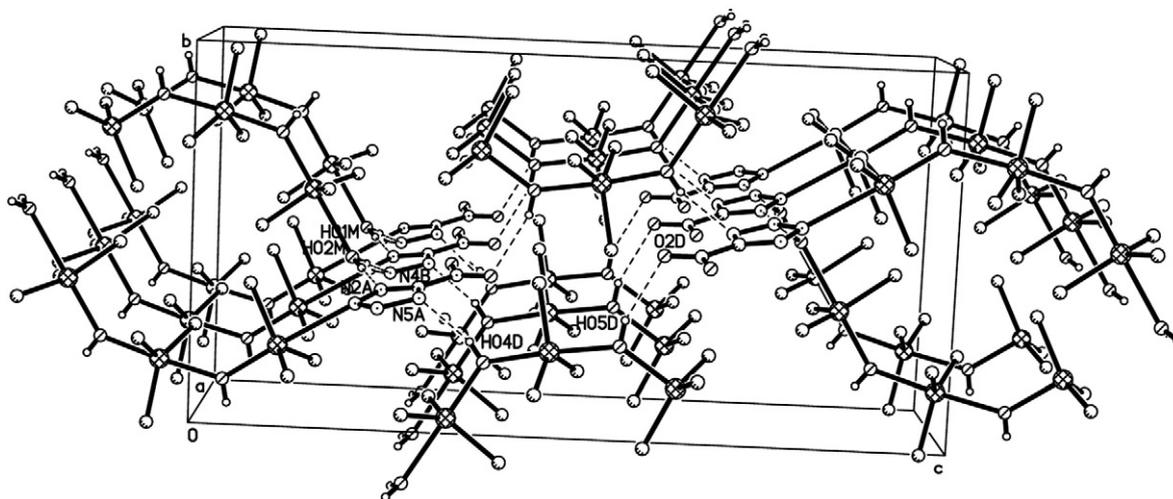
**Fig. 1.** Molecular structure of **2** in the crystal showing the atom-labeling scheme. Displacement ellipsoids are drawn at the 50% probability level.

subsequently treated with warm sodium hydroxide solution to afford **1** as the dihydrate. While the dihydrate of NaNT is fairly insensitive and can be handled with the appropriate care, the anhydrous material is dangerously explosive [16,28,29]. Most recently, after the present study had been finalized, a significantly improved preparation of high-purity NaNT has been reported, which avoids the handling of the potentially dangerous  $\text{Cu}(\text{NT})_2 \cdot \text{HNT}$  intermediate [27].

Based upon our experience with the preparation of triorganotin(IV) pentacyanopropenides [30] and the recently reported azotetrazolate derivative  $[\text{Me}_3\text{Sn}(\mu\text{-C}_2\text{N}_{10})\text{SnMe}_3(\text{H}_2\text{O})]_n$  [31] we investigated the reaction of NaNT (**1**) with trimethyltin chloride in aqueous solution. Slow evaporation of the reaction mixture to dryness afforded well-formed, colorless, spear-like crystals of the product admixed with the cube-like crystals of the NaCl by-product. The latter could be redissolved in a minimum amount of cold water. The product was then carefully

dried in air. Spectroscopic characterization proved to be mostly uninformative (vide infra), but the crystals were found to be suitable for X-ray diffraction [32]. The X-ray structure analysis revealed the rather unexpected presence of the title compound,  $[\text{Me}_3\text{Sn}(\mu\text{-OH})\text{SnMe}_3(\mu\text{-OH})\text{SnMe}_3(\text{H}_2\text{O})][\text{NT}]$  (**2**). Scheme 2 illustrates the formation of **2**.

Compound **2** was found to crystallize in the monoclinic space group  $\text{P}2_1/\text{n}$  [33]. As can be seen in Fig. 1, the most notable structural feature is the presence of the trinuclear organotin cation  $[\text{Me}_3\text{Sn}(\mu\text{-OH})\text{SnMe}_3(\mu\text{-OH})\text{SnMe}_3(\text{H}_2\text{O})]^+$ . This cation is normally formed in reactions of  $\text{Me}_3\text{SnX}$  ( $\text{X} = \text{Cl}, \text{Br}, \text{I}$ ) with 2 equiv. of  $\text{Me}_3\text{SnOH}$  [34]. It has been frequently shown to be a highly useful component of interesting supramolecular structures [35–37]. As in the case of the rhenium cluster complex  $[\text{Me}_3\text{Sn}(\mu\text{-OH})\text{SnMe}_3(\mu\text{-OH})\text{SnMe}_3][\{\text{Me}_3\text{Sn}\}_3\{\text{Re}_6\text{Se}_8(\text{CN})_6\}]$ , the formation of the  $[\text{Me}_3\text{Sn}(\mu\text{-OH})\text{SnMe}_3(\mu\text{-OH})\text{SnMe}_3(\text{H}_2\text{O})]^+$  cation in **2** can be traced back to the fact that aqueous solutions of **1** have a



**Fig. 2.** Supramolecular crystal structure of **2** viewed along the *a* axis.

**Table 1**  
Energetic properties of **2**.

Compound	<b>2</b>
M (g/mol)	657.52
$\rho$ , g/cm <sup>3</sup>	1.946
Impact/J	<2.5
Friction sensitivity (N)	>288
Combustion test	Deflagration, smoke
N content (%)	10.65
Oxygen balance ( $\Omega$ )	−81.5

pH value of >7, which could cause intermediate formation of Me<sub>3</sub>SnOH. A notable structural difference to previously reported structures is that in **2** the [Me<sub>3</sub>Sn( $\mu$ -OH)SnMe<sub>3</sub>( $\mu$ -OH)SnMe<sub>3</sub>(H<sub>2</sub>O)]<sup>+</sup> cation adopts a U-shaped (*cisoid*) conformation with the H atoms of the  $\mu$ -OH groups pointing in the same direction, whereas in [Me<sub>3</sub>Sn( $\mu$ -OH)SnMe<sub>3</sub>( $\mu$ -OH)SnMe<sub>3</sub>]Br [36] and [Me<sub>3</sub>Sn( $\mu$ -OH)SnMe<sub>3</sub>( $\mu$ -OH)SnMe<sub>3</sub>][{Me<sub>3</sub>Sn}<sub>3</sub>{Re<sub>6</sub>Se<sub>8</sub>(CN)<sub>6</sub>}] [37] the configuration of the cation is Z-shaped (*transoid*) with the  $\mu$ -OH hydrogens pointing in opposite directions. It has been pointed out that the trinuclear [Me<sub>3</sub>Sn( $\mu$ -OH)SnMe<sub>3</sub>( $\mu$ -OH)SnMe<sub>3</sub>(H<sub>2</sub>O)]<sup>+</sup> is itself a supramolecular entity formed through dative O → Sn bonds connecting the Me<sub>3</sub>Sn groups [36,38]. In **2**, all three Sn atoms comprise a trigonal-bipyramidal coordination geometry. The coordination sphere of Sn1 is completed by a water molecule, while the 5-nitrotetrazolate anion is loosely coordinated to Sn3 via the ring-nitrogen atom N3 (Fig. 1). The length of the secondary Sn⋯N bond [38] is 2.809 Å. With 176.00(12)° the O4–Sn2–O5 angle at the central Sn atom is nearly linear (cf. 178.8(2)° in [Me<sub>3</sub>Sn( $\mu$ -OH)SnMe<sub>3</sub>( $\mu$ -OH)SnMe<sub>3</sub>]Br [36]). The 5-nitrotetrazolate anion is further engaged in four different O–H⋯N and O–H⋯N hydrogen bonds involving the remaining three ring-nitrogen atoms and one oxygen of the nitro group (Fig. 2). This leads to an extensive supramolecular hydrogen-bonded network in the solid state resembling e.g. those reported for the crystal structures of alkali and alkaline earth metal 5-nitrotetrazolates [16,22–27]. In good agreement with the solid state structure is the IR spectrum of **2** which shows bands typical for all major components of the molecule (OH, NO<sub>2</sub>, N–C–N, Sn(CH<sub>3</sub>)). In contrast, both the <sup>1</sup>H and <sup>13</sup>C spectra each show only a singlet attributable to Sn(CH<sub>3</sub>)<sub>3</sub>, indicating that the solid-state structure of **2** does not persist in solution (acetone-*d*<sub>6</sub>).

Initial test results of the energetic properties of **2** are listed in Table 1. In addition to a simple combustion test, the impact and friction sensitivity were tested according to established BAM methods [3] using the BAM drop hammer and BAM friction tester. The “flame test” revealed deflagration of **2** accompanied by strong evolution of smoke (SnO<sub>2</sub>). The material was found to be relatively insensitive to friction (>288 N). In contrast, the drop hammer test revealed that despite the low nitrogen content and negative oxygen balance, compound **2** is very sensitive to impact. The value of <2.5 J for **2** is comparable to those found for Pb(N<sub>3</sub>)<sub>2</sub> (3.0–6.5 J) [2] or lead(II) styphnate (2.5–5.0 J) [39], so that **2** can be classified as a primary explosive.

In summarizing the results reported here, the first metal–organic nitrotetrazolate, [Me<sub>3</sub>Sn( $\mu$ -OH)SnMe<sub>3</sub>( $\mu$ -OH)SnMe<sub>3</sub>(H<sub>2</sub>O)][NT] (**2**), was made via a simple metathetical reaction and was structurally elucidated by single-crystal X-ray diffraction. The supramolecular crystal structure is characterized by an extensive network of O–H⋯N and O–H⋯N hydrogen bonds. With an impact sensitivity of <2.5 J, compound **2** represents a new type of primary explosive. Being an organotin compound, it can certainly not be regarded as environmentally acceptable, but the results reported here will without doubt stimulate further work directed to the exploration of new metal–organic or organometallic (transition metals) 5-nitrotetrazolates.

## Acknowledgments

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## Appendix A. Supplementary material

Crystallographic data for the crystal structure reported in this paper can be obtained from the Cambridge Crystallographic Data Center, 12 Union Road, Cambridge CB21EZ, UK (fax: +44-1223-336-033; e-mail: deposit@ccdc.cam.ac.uk or <http://www.ccdc.cam.ac.uk/>) by referring to the CIF deposition code CCDC 977000 (**2**). Supplementary data to this article can be found online at <http://dx.doi.org/10.1016/j.inoche.2014.02.003>.

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- [31] R. Deblitz, C.G. Hrib, G. Plenikowski, F.T. Edelman, Inorg. Chem. Commun. 18 (2012) 57–60.
- [32] **CAUTIONARY NOTE:** The starting material Me<sub>3</sub>SnCl is highly toxic and has a stench. It should be handled only in a well-ventilated fumehood. Sodium nitrotetrazolate dihydrate (**1**) is an energetic material which is sensitive to various stimuli. Great care should be taken to not excessively dry this material, because anhydrous NaNT is much more sensitive than the dihydrate. The title compound **2** has been found to be sensitive to both impact and friction (cf. Table 1). Thus proper safety equipment is necessary and the compound should be handled with utmost care and only in small amounts. *Synthesis of [Me<sub>3</sub>Sn( $\mu$ -OH)SnMe<sub>3</sub>( $\mu$ -OH)SnMe<sub>3</sub>(H<sub>2</sub>O)][NT] (**2**):* Me<sub>3</sub>SnCl (360 mg, 1.8 mmol) was dissolved in water (10 ml) under slight warming to ca. 40 °C. 0.32 ml of the 80% stock solution of **1** (ca. 260 mg, 1.5 mmol) were added dropwise. The reaction mixture was left undisturbed in the fumehood until the water had completely evaporated. The NaCl by-product was redissolved in a minimum amount (ca. 5 ml) of cold water and the remaining spear-like crystals of **2** were isolated in 57% yield (225 mg) based on Me<sub>3</sub>SnCl. Analysis (C<sub>10</sub>H<sub>31</sub>N<sub>5</sub>O<sub>5</sub>Sn<sub>3</sub>, Mw = 657.52 g/mol): C 17.80 (calcd. 18.27), H 4.55 (4.75), N 9.98 (10.65)%. IR (KBr):  $\nu_{\max}$  3415 (s, OH), 2999 (m, CH<sub>3</sub>), 2922 (m, CH<sub>3</sub>), 1689 (m), 1550 (s, NO<sub>2</sub> asymmetric stretching), 1507 (m), 1455 (s), 1443 (s), 1419 (s, N–C–N symmetric

- stretching), 1385 (s), 1316 (s, NO<sub>2</sub> symmetric stretching), 1242 (s), 1194 (s), 1173 (s), 1152 (s), 1135 (m), 1066 (m), 1041 (m), 984 (m), 842 (s, N–C–N deformation), 773 (m), 671 (m), 547 (s), 464 (m) cm<sup>-1</sup>. <sup>1</sup>H NMR (20 °C, acetone-*d*<sub>6</sub>, 400 MHz): δ [ppm] 3.09 (s br, OH and H<sub>2</sub>O), 0.55 (s, SnCH<sub>3</sub>). <sup>13</sup>C NMR (20 °C, acetone-*d*<sub>6</sub>, 100 MHz): δ [ppm] –0.9 (SnCH<sub>3</sub>).
- [33] The X-ray diffraction data for compound **2** were measured at –120 °C on a Stoe IPDS diffractometer. Structure solution and refinement were carried out using the programs SHELXS-97 [40] and SHELXL-97 [41]. Crystal data for **2**: C<sub>10</sub>H<sub>31</sub>N<sub>5</sub>O<sub>5</sub>Sn<sub>3</sub>, M = 657.52, monoclinic, space group P2<sub>1</sub>/n, T = 153(2) K, a = 7.3722(15), b = 12.457(3), c = 24.446(5) Å, V = 2244.5(8) Å<sup>3</sup>, Z = 4, D<sub>c</sub> = 1.946 g cm<sup>-3</sup>, a total of 9100 reflections were collected, 3969 of which were unique (R<sub>int</sub> = 0.0561), final R<sub>1</sub> = 0.0264 for I > 2 σ(I), wR<sub>2</sub> = 0.0731 for all data, GOOF = 1.106. The crystal data and structure refinement are summarized in Table S1. The selected bond lengths and bond angles are listed in Table S2, and the hydrogen bond lengths and bond angles are listed in Table S3.
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