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## Author Manuscript

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# Bis-(1,2,4-Oxadiazolyl) Furoxan: A Promising Melt-Castable Eutectic Material of Low Sensitivity

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**Abstract:** : A scalable synthesis of bis-(1,2,4-oxadiazolyl) furoxan, C<sub>6</sub>H<sub>2</sub>N<sub>6</sub>O<sub>4</sub>, its physical properties, and its theoretical performance values are described. Previous attempts to synthesize this compound required expensive reagents, and/or time consuming synthesis processes and low overall yields. In addition to disclosing a streamlined synthesis of bis-(1,2,4-oxadiazolyl) furoxan, we report its molecular configuration and crystal structure, as well as its correct melting point. Bis-(1,2,4-oxadiazolyl) furoxan exhibits a very insensitive behavior to impact, friction and electrostatic discharge, with a calculated detonation pressure 20% higher than TNT. Given its physical properties and theoretical performance values, this material can be classified as a promising ingredient in the development of melt-castable eutectic technology.

**Keywords:** Energetic Materials, Explosives, Heterocycles, Process Chemistry, Synthesis

## INTRODUCTION

Melt-castable explosive technology is an area of significant importance in civilian and military applications. Such a technology must display a high performance, a low sensitivity, and must possess specific characteristics. An energetic material that melts between 70–120 °C is typically defined as being in the melt-castable range<sup>1</sup>, with a melting point below 100 °C being ideal. This feature allows for the use of steam at ambient pressures during casting operations, which is critical in minimizing manufacturing costs.

There are two classes of melt-castable technologies; standalone and eutectic materials. A standalone melt-castable energetic material possesses melt-castable physical properties without the need to employ any other ingredients. TNT and its lower performing counterpart DNAN (dinitroanisole)<sup>2</sup> are the benchmark examples of a standalone melt-castable materials. However, recently, high-performing nitrate ester-based materials have been developed that also fall into the standalone melt-castable range.<sup>3</sup>

On the other hand, a melt-castable eutectic material typically has a melting point just north of 100 °C. Such a material cannot be melted alone using steam heating, but is still potentially useful in melt-castable technology when combined with other energetic

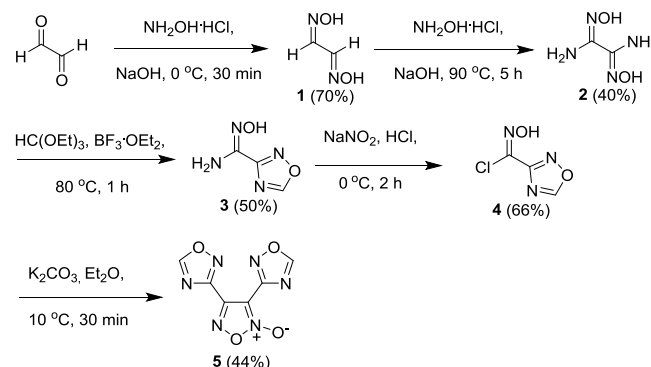
amenable to steam heating is the basic definition of melt-castable eutectic science. DEMN (a mixture of 34.9 wt. % diethylenetriamine trinitrate/33.4 wt. % ethylenediamine dinitrate/25.4 wt. % methyl nitroguanidine/6.3 wt. % nitroguanidine)<sup>4</sup> is an example of one such formulation in which an optimal melting point below 100 °C has been achieved, with a recent example of a potential melt-castable eutectic ingredient being bisoxazole tetrakis (methyl nitrate).<sup>5</sup> Despite the aforementioned research examples, the number of melt-castable materials available for practical use remains quite limited on the bases of cost, performance and sensitivity. Therefore, syntheses of melt-castable ingredients (eutectic or standalone) that have a high degree of scalability, with low sensitivities and high performance are still very much desired.

In this paper, we report the synthesis, crystal structure, and proper physical properties of bis-(1,2,4-oxadiazolyl) furoxan, (**5**). Further, we report its sensitivity to external stimuli and its calculated performance properties.

## RESULTS AND DISCUSSION

Upon reviewing the literature for melt-castable compounds, it was found that Yan *et al.* prepared bis-(1,2,4-oxadiazolyl) furoxan (**5**) from glyoxal in 5 steps in a low yield of 4% (Scheme 1).<sup>6</sup> A DSC of **5** was reported as having an onset temperature of melting of 98 °C, with a peak melting temperature of 114 °C, and an onset decomposition temperature of 208 °C. As this reported melting temperature range was quite broad, it indicates that an impurity is likely present in the final product.

**Scheme 1.** Synthesis of **5** by Yan *et al.*



Two years prior to the aforementioned synthesis, **5** was synthesized by Fershtat *et al.* utilizing a scandium triflate-mediated double cyclization reaction of bis-amidoxime **7** and trimethyl orthoformate (Scheme 2).<sup>7</sup> Their synthesis was not only simpler than the route by Yan *et al.*, but was also higher yielding overall. By the methodology of Fershtat *et al.*, **5** was afforded in 28.5% overall yield. While the authors did not report any decomposition temperature for **5**, they listed a melting point of 84–86 °C. This melting point discrepancy was significantly different than the melting point reported for **5** by Yan *et al.* While it was not clear as to why such a difference in melting points for **5** existed when examining the two reported methods, the material appeared to be a possible melt-castable material. In fact, a melting point of 84–86 °C would fit the definition of a standalone melt-castable material.

Despite the simpler synthesis and increase in overall yield of **5** by Fershtat *et al.*, the cyclization step, while produced under mild conditions, is still problematic from the scale-up perspective. The reason centers on the use of scandium triflate. Scandium is a rare-

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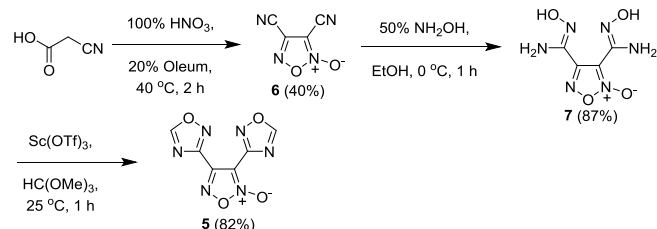
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materials. This practice of combining two or more ingredients in a formulation to achieve an overall melting point that becomes

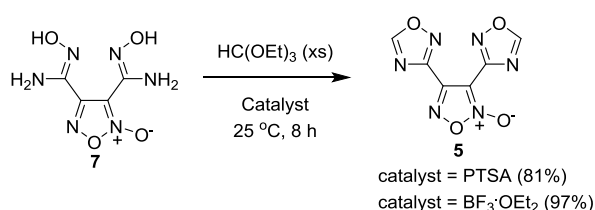
earth metal of relative low abundance, and as such, the price of scandium-based reagents is high. Therefore, in developing a more cost-efficient route for **5** that is amenable for scalability, a cheaper cyclization method from **7** was desired.

**Scheme 2.** Synthesis of **5** by Russian researchers.



Intermediates **6** and **7** were synthesized in equivalent yields as the procedure by Fershtat *et al.* outlined in Scheme 2.<sup>8,9</sup> Instead of relying on Sc(OTf)<sub>3</sub> to promote cyclization to give **5**, we chose to employ some cost-efficient catalysts in the presence of an excess of triethyl orthoformate (Scheme 3). When *p*-toluenesulfonic acid monohydrate (PTSA) was used, *bis*-amidoxime **7** was converted to *bis*-1,2,4-oxadiazolyl furoxan (**5**) in 81% yield. We also examined the effectiveness of boron trifluoride diethyl etherate in this reaction. Although this latter catalyst is commonly used to synthesize monosubstituted 1,2,4-oxadiazoles from amidoximes, such a transformation often requires temperatures of 70 °C or higher.<sup>10,11</sup> Unfortunately, when heat was applied to this cyclization reaction, the reaction decomposed and failed to yield any product. Fershtat *et al.* reported that BF<sub>3</sub>·Et<sub>2</sub>O was not an effective catalyst in the presence of trimethyl orthoformate to convert amidoxime-substituted furoxans to the monosubstituted 1,2,4-oxadiazole products at room temperature.<sup>7</sup> In our case however, BF<sub>3</sub>·Et<sub>2</sub>O proved to be a very effective catalyst at room temperature, converting **7** to **5** in the presence of triethyl orthoformate in near quantitative yield. Hence, the overall yield of **5** from cyanoacetic acid was further improved to 34% by this latter method. The BF<sub>3</sub>·Et<sub>2</sub>O-mediated double cyclization reaction detailed in Scheme 3 has been carried out on 25 g scale without difficulty. No exothermic event was observed throughout the course of the reaction.

**Scheme 3.** Double cyclization of **7** to give **5**.



Interestingly, a DSC of **5**, synthesized by the cyclization method described in Scheme 3 showed an onset temperature of decomposition of 227 °C, which is significantly higher than the 208 °C value reported by Yan *et al.* The onset melting temperature of **5** was determined to be 114.3 °C, with a peak melting point of 117.8 °C. Such a sharp melting point range is indicative of a material of high purity. Hence, the melting point of **5** is quite different than the broad melting point of 98–114 °C observed by Yan *et al.*, as well as the sharp melting point of 84–86 °C reported by Fershtat *et al.* Although the 114.3–117.8 °C melting point range falls outside of the standalone melt-castable range, the melting point range of **5** is such that classifies it as a potential melt-castable eutectic ingredient. Hence, it could be mixed with other energetic materials to depress the melting point below 100 °C so that the resulting formulation can be used in casting operations.

The physical properties and explosive performances of **5** are provided in Table 1.<sup>12</sup> **5** outperforms TNT in oxygen balance, density, detonation velocity, and heat of formation by significant margins. **5**

also has a predicted detonation pressure that is 20% higher than that of TNT.

The sensitivity of **5** toward impact, friction, and electrostatic discharge (ESD) were determined and compared to TNT, which is considered the benchmark insensitive melt-castable explosive (Table 1). **5** was found to be more insensitive than TNT to impact and friction, while exhibiting an equivalent ESD sensitivity.

**Table 1.** Physical properties, calculated performance and sensitivities of **5** compared to TNT.

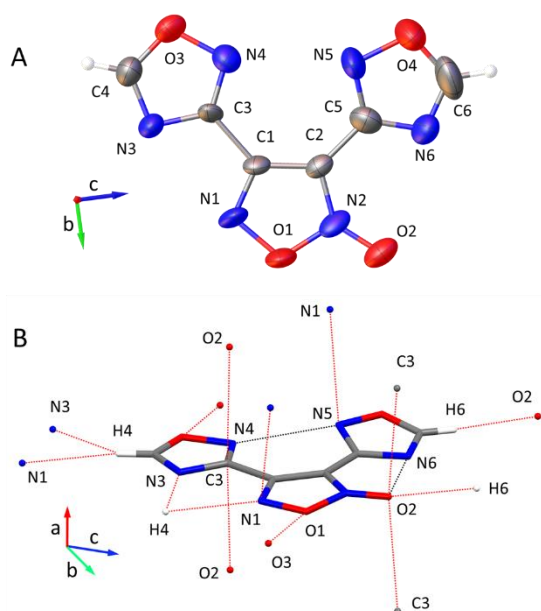
Data category	<b>5</b>	TNT
T <sub>m</sub> [°C] <sup>[a]</sup>	114.3	80.4
T <sub>dec</sub> [°C] <sup>[b]</sup>	227	295
Ω <sub>CO2</sub> [%] <sup>[c]</sup>	-64.9	-74
Ω <sub>CO</sub> [%] <sup>[d]</sup>	-21.6	-24.7
ρ [gcm <sup>-3</sup> ] <sup>[e]</sup>	1.79	1.65
P <sub>dj</sub> [GPa] <sup>[f]</sup>	24.1	20.5
V <sub>det</sub> [ms <sup>-1</sup> ] <sup>[g]</sup>	7577	6950
I <sub>sp</sub> [s] <sup>[h]</sup>	220	-
Δ <sub>r</sub> H° [kJ mol <sup>-1</sup> ] <sup>[i]</sup>	357	-59.3
IS <sup>[j]</sup> [J]	>34.7	15
FS <sup>[k]</sup> [N]	>360	240
ESD <sup>[l]</sup> [J]	>0.25	0.25

[a] T<sub>m</sub> = onset temperature of melting; [b] T<sub>dec</sub> = onset temperature of decomposition; [c] Ω<sub>CO2</sub> = CO<sub>2</sub> oxygen balance; [d] Ω<sub>CO</sub> = CO oxygen balance; [e] ρ = derived density from X-ray data; [f] P<sub>dj</sub> = detonation pressure; [g] V<sub>det</sub> = detonation velocity; [h] I<sub>sp</sub> = specific impulse; [i] Δ<sub>r</sub>H° = molar enthalpy of formation; [j] IS = impact sensitivity; [k] FS = friction sensitivity; [l] ESD = electrostatic discharge sensitivity

X-ray diffraction data analysis confirms the molecular structure of **5**, reveals its molecular configuration, and indicates key intra- and intermolecular interactions. The details of the data collection, cell constants determination, as well as the structure solution and refinement are provided in the supplementary material.<sup>13–15</sup> **5** presents one molecule in the asymmetric unit (Figure 1). Its bond length and angles are in the usual range, consistent with those reported for other oxadiazoles and their derivatives.<sup>3,16–22</sup> Overall, **5** is near planar [r.m.s. = 0.082 (5) Å] with a maximum out-of-plane deviation of 0.021 (2) Å for the N5 atom. The molecule comprises three near planar pentagonal oxadiazole rings [r.m.s. deviation = .0026 (2), 0.0060 (5), and 0.023 (4) Å for rings containing the atoms O1 (R1), O3 (R2), and O4 (R3), respectively]. The 1,2,5-oxadiazole-*N*-oxide ring presents an O2 atom appendant forming a near planar functionalized moiety, M1 [r.m.s. deviation = 0.014 (1) Å]. Plane M1 subtends dihedral angles of 3.8 (5)° and 11.2 (6)° in relation to the planes R2 and R3, respectively, whereas planes R2 and R3 subtend a dihedral angle of 9.9 (6)°. The R3 moiety twists slightly from the near planar R1 and M1 moieties perhaps because of the N4···N5 [2.83 (1) Å] and N6···O2 [2.91 (1) Å] intramolecular interactions.

Intermolecular interactions play a key role in the crystal packing and stability of **5**. Contacts between the H atoms and O or N atoms of adjacent molecules [H6···O2<sup>iv</sup> = 2.37 (1) Å or H4···N1<sup>ii</sup> = 2.56 (2) Å; symmetry codes: (i) 1-x, -1/2+y, -z; (ii) -2-x, -1/2+y, -2-z] dominate the intermolecular interactions. In addition, contacts between the O3 atoms and O1 atoms of adjacent atoms [O3···O1<sup>iv</sup> = 2.93 (1) Å; symmetry code: (iv) -x, -1+y, z], the N5 atoms and N1 atoms of nearby molecules [N5···N1<sup>v</sup> = 3.07 (2) Å; symmetry code: (v) -1-x, -1/2+y, -1-z], and the C3 atoms and O2 atoms of adjacent molecules

$[C3 \cdots O2]^{\text{iii}} = 3.19$  (1) Å; symmetry code: (iii)  $-2-x, -1/2+y, -1-z$ ) contribute to its crystal packing (see Fig. 3). The  $C \cdots O$  and  $N \cdots N$  interactions help link the molecules along the  $a$  axis, whereas the  $O \cdots H$ ,  $N \cdots H$ , and  $O \cdots O$  interactions contribute to its network of near planar sheets almost parallel to (010) (see Figs. 3 and 4). Based on its molecular mass and lattice constants listed in Table 2, we obtain a density of 1.79 g/cc at 297K.



**Figure 1.** (A) Molecular configuration and atom-numbering scheme for **5**. Non-hydrogen atoms are shown as 50% probability displacement ellipsoids; (B) Selected atom-atom intramolecular and intermolecular interactions for **5**, where the contacts are depicted as dashed lines.

In summary, an efficient, scalable route to *bis*-1,2,4-oxadiazolyl furoxan (**5**) has been developed, its crystal structure determined, and its explosive performance and sensitivity evaluated. It features a three step synthesis in an overall yield of 34%, which is an improved overall yield over previously existing syntheses. **5** was found to have a low sensitivity to impact, friction and ESD, and was estimated to be safer to handle than TNT. The characteristics and performance properties of **5** suggest that this material is not a standalone melt-castable as previously reported, but instead a potential high-energy melt-castable eutectic material. Combining **5** with other high-energy ingredients may depress the melting point below 100 °C, thus making it amenable for high-energy melt-casting formulations.

## Experimental Section

Chemicals and solvents were used as received from Oakwood Chemicals. Dicyanofuroxan (**6**) and *bis*-amidoxime **7** were synthesized according to the literature.<sup>8,9</sup> <sup>1</sup>H and <sup>13</sup>C NMR spectra of *bis*-oxadiazole **5** were recorded using an Anasazi 90 MHz and 22.5 MHz instrument, respectively. The chemical shifts quoted in ppm in the text refer to typical standard tetramethylsilane in CDCl<sub>3</sub> as the solvent. Infrared spectra were measured with a Bruker Alpha-P FTIR instrument. Melting and decomposition temperatures were measured at a heating rate of 5 °C/min using a TA Instruments Q10 DSC instrument. Single crystal X-ray diffraction (XRD) studies were performed with a SuperNova Dualflex diffractometer containing an

EosS2 charge-coupled device detector and a molybdenum Mo-Kα ( $\lambda = 0.71073$  Å) radiation source.

**Caution!** Although we did not experience any problems handling the compounds described in this paper, proper laboratory precautions should be taken. Laboratories and personnel should be properly grounded, and safety equipment such as heavy Kevlar/steel gloves, reinforced Kevlar coat, ballistic face shield, ear plugs, and blast shields are necessary for all energetic transformations, and in handling any material that is determined to be energetic.

### *Bis*-(1,2,4-Oxadiazolyl) Furoxan (**5**).

**Method A:** *Bis*-amidoxime **7** (505 mg, 2.50 mmol, 1.00 eq) was placed in a 10 ml round bottomed flask. Triethylorthoformate (1.45 g, 13.7 mmol, 5.48 eq) was then added to the reaction flask and the mixture stirred at ambient temperature. *p*-Toluenesulfonic acid monohydrate (47 mg, 0.25 mmol, 0.100 eq) was then added and the reaction stirred at room temperature for 8 h. After 1.5 hours, the mixture becomes a homogenous solution after which a precipitate began to form. The mixture was poured into water (10 mL), triturated, filtered, washed with water and air dried to provide 450 mg of *bis*-1,2,4-oxadiazolyl furoxan (**5**) (81 %) as an off-white solid. **Method B:** To a 1 L round-bottom flask equipped with a stir bar charged with 206 mL of triethyl orthoformate (183.5 g, 1.24 mol, 10 eq) was added *bis*-amidoxime **7** (25.0 g, 123.8 mmol, 1.00 eq). To the resulting stirred suspension was then added 1 mL of boron trifluoride diethyl etherate (1.41 g, 9.90 mmol, 0.08 eq). After 10 minutes, the solid went into solution, and the reaction mixture was stirred further at ambient temperature for 8 h. The reaction mixture was then poured onto crushed ice and stirred vigorously for 2 h, during which time the ice melted. The resulting white solid was collected by Büchner filtration, washed with cold two 500 mL portions of cold water, suction dried for 1 h, and was transferred to crystallization dish. Drying of the white solid overnight at 60 °C afforded 26.7 g (97%) of *bis*-1,2,4-oxadiazolyl furoxan (**5**) as an off-white solid.  $T_{\text{melt}} = 114.3$  °C (onset), 117.8 °C (peak);  $T_{\text{dec}} = 227.0$  °C (onset), 268.2 °C; <sup>1</sup>H NMR (90 MHz, DMSO-*d*6) 10.02 (s, 1H), 9.95 (s, 1H); <sup>13</sup>C NMR (22.5 MHz, DMSO-*d*6)  $\delta$  177.34, 177.04, 176.40, 176.10, 175.50, 175.20; IR (neat) 3108.99 (w), 1604.49, (m), 1578.19 (m), 1546.95, 1529.96 (m), 1270.35 (s), 1091.82 (w), 1017.00 (s) cm<sup>-1</sup>.

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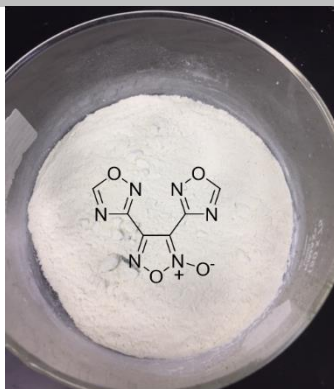
**Keywords:** Energetic Materials • Synthesis • Heterocycles • Explosives • Eutectic

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## COMMUNICATION

**A Melt-Castable Eutectic:** A scalable synthesis of *bis*-(1,2,4-oxadiazolyl) furoxan,  $C_6H_2N_6O_4$ , its physical properties, and its theoretical performance values are described. This material possesses a calculated detonation pressure 20% higher than TNT, and is very insensitive to impact, friction and electrostatic discharge. The material is classified as an insensitive explosive ingredient that could find application in melt-castable eutectic formulation science.



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***Bis*-(1,2,4-Oxadiazolyl) Furoxan: A**

**Promising Melt-Castable Eutectic**

**Material of Low Sensitivity**

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