



Explosive Properties of Erythritol Tetranitrate

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Abstract: The physical and explosive properties of erythritol tetranitrate (ETN) are described herein. Although the chemical structure of ETN is simple and its preparation is undemanding, this explosive is only briefly described in the literature. Nowadays, it is widely prepared by amateur chemists and by criminals as well. Therefore, it is necessary to describe its physical and explosive properties for pre-explosion and post-explosion forensic analyses. However, as a nitric acid ester, it can also be potentially useful for some industrial applications. ETN was prepared in 83% yield by the reaction of nitric acid/sulfuric acid with erythritol. The molecular structure of ETN was characterized by single-crystal X-ray diffraction. The structure of the ETN molecule is composed of a central carbohydrate chain and two pairs of facing coplanar ONO_2 groups. The crystal density of ETN is 1.827 g cm^{-3} . It is a non-hygroscopic compound, which is

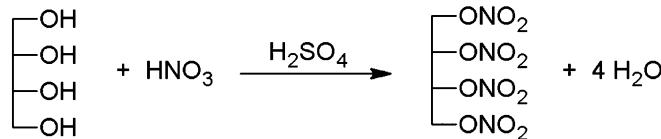
slightly soluble in water (the solubility in water was determined in a temperature range from 5°C to 80°C ; the solubility at 20°C is similar to that of PETN). The sensitivity of melt cast ETN to friction significantly differs from powdered ETN. Melt cast ETN is more sensitive to friction than PETN, whereas powdered ETN is less sensitive than RDX. The sensitivity of powdered ETN to impact is slightly lower than for melt cast ETN that is on the level of PETN. The detonation velocity of melt cast ETN is 7940 m s^{-1} at a density of 1.69 g cm^{-3} , which is slightly below the PETN level. The relative explosive strength was measured using the ballistic mortar method and value of 143% TNT was found, which is similar to that of PETN (145% TNT is reported in the literature). Additionally, the relative brisance was determined using the Hess test.

Keywords: Erythritol tetranitrate · ETN · Sensitivity · Explosive parameters · Properties

1 Introduction

Erythritol tetranitrate (ETN) is a simple nitrate ester structurally similar to nitroglycerine (see Scheme 1). This ester of nitric acid was first synthesized in 1849 by Stenhouse [1]. Although it is a structurally simple and easily synthesized powerful explosive, only a few authors have paid attention to it as an explosive in the past. The main reason was the high cost caused by limited availability of erythritol, which was obtained by costly extraction from seaweed, algae, or lichen [2]. Later, production of erythritol via acetylene chemistry did not significantly change anything; the price of ETN was still too high for industrial use [3]. However, a new technology for erythritol production using microbial methods from osmophilic yeasts has been recently developed [4]. The price of erythritol has been significantly reduced and its technological accessibility is no longer a limitation.

In recent years, ETN has become a popular amateur explosive since erythritol was first released into the market as a sweetener. For this reason the material is now available for criminals or terrorists and the risk of ETN misuse increases.



Scheme 1. Synthesis of ETN.

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es. Unfortunately, the properties of ETN are described only briefly in scientific literature [3, 5–7]. Research into the fundamental parameters of this improvised explosive is therefore important e.g. for forensic analysts and EOD. And last but not least, the sensitivity characteristics are important for anybody working with the compound. There have been plenty of articles concerning the medical effects and uses of erythritol tetranitrate (it is used as an effective cardiovascular medicine [5]), but only a few remarks have appeared in tertiary literature about its explosive properties. The subject was addressed two years ago by Oxley et al. [8], who published some analytical data for ETN, along with certain physical and explosive properties. The characterization of explosive properties of explosives based on ammonium nitrate sensitized with erythritol tetranitrate has recently been published by the current authors [9]. The thermal behavior and decomposition kinetics of pure ETN and its mixtures with pentaerythritol tetranitrate (PETN) and hexogen (1,3,5-trinitro-1,3,5-triazinane, RDX) have also been published recently [10]. We decided to study additional characteristics of this nitrate ester that, according to our best information, have not been published to date. The results of our research are summarized in this article.

2 Experimental Section

Caution: No problems have occurred during the synthesis and handling of erythritol tetranitrate, but the material is still an explosive. Laboratories and personnel should be properly prepared and safety equipment such as protective gloves, shields, and ear plugs should be used, even when working with small-scale reactions.

2.1 Preparation of Erythritol Tetranitrate

Erythritol tetranitrate was prepared according to the method patented by Bergeim [11] and described by us recently in detail [9]. Erythritol [a meso form (2R,3S)-butane-1,2,3,4-tetraol] with declared purity of 99.5% was obtained from a local pharmacy (trade name Extra-Linie produced by FAVEA, Kopřivnice, Czech Republic). Other chemicals used were of analytical purity (p.a.).

Each batch used 100 g of erythritol. Crude erythritol tetranitrate (m.p. 56–58 °C) was purified by re-crystallization from ethanol. We obtained 218–221 g of crystallized products in one batch (88–89%). Melting point of the crystallized product was 59–60 °C (literature [7], 61 °C). The crystal size and shape of the obtained product were determined by optical microscopy and are presented in Figure 1.

Elemental analysis: calcd. C 15.90, H 2.00, N 18.55%; found C 16.66, H 2.48, N 17.78%.

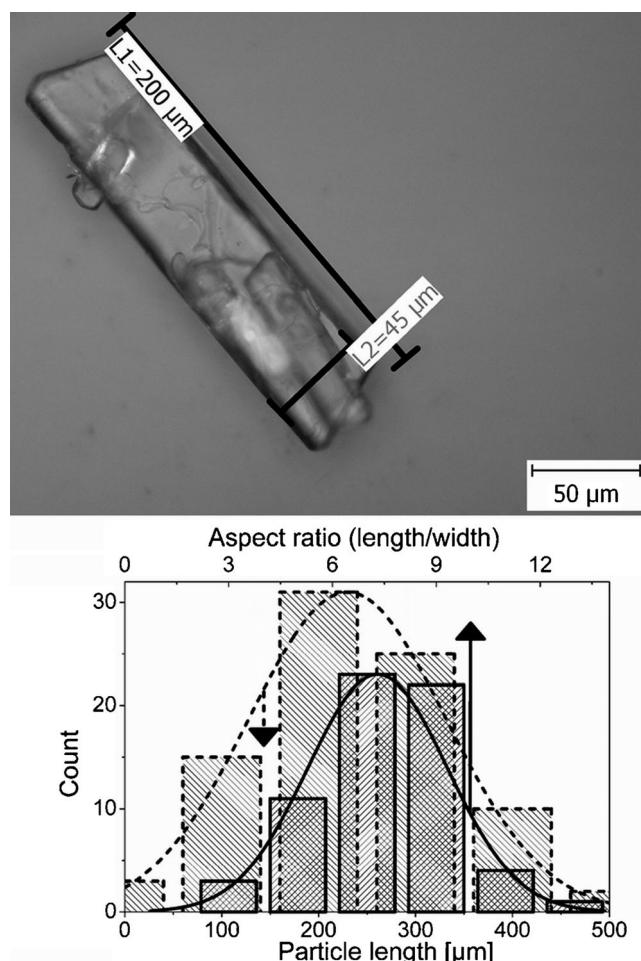


Figure 1. Crystal shape of the ETN particles and histogram of the diameter (dotted curve) and aspect ratio (length vs. width) of the particles (block curve).

2.2 Characterization of Comparative Explosives

Mercury fulminate (MF), pentaerythritol tetranitrate (PETN), and hexogen (RDX) were used as standard explosives for comparison of ETN sensitivity. Brown mercury fulminate was prepared using the process described in the literature [12]. The crystal size and shape were determined by electron microscopy and have been published in a previous paper [13]. PETN with particles smaller than 200 μm was provided by the Explosia a. s. company (Czech Republic) under the trade name "Pentrit NS" and RDX was provided by the Chemko Strazske company (Slovak Republic).

2.3 Analysis of ETN

A FLASH 2000 apparatus from Thermo scientific with HCNS Analyzer was used for performing elemental analysis. Nicotinamide (for samples having nitrogen content up to 25%) and melamine (for substances having nitrogen content up to 66%) were used as standards.

The liquid chromatographic system Agilent 1200 series was used for checking the purity of ETN. Isocratic separation using a solvent mixture consisting of 60% acetonitrile and 40% water with a flow rate of 1 mL min^{-1} was used. The injection volume was $10\text{ }\mu\text{L}$, column material was BDS Hypersil C-18 with $5\text{ }\mu\text{m}$ particle size and 12 nm pore size, column dimensions were $250\text{ mm} \times 4.6\text{ mm}$, temperature 30°C . The chromatograms were evaluated from the absorbance at 210 nm .

The sample of re-crystallized ETN was visualized by an Olympus BX60 optical microscope in reflex mode (magnification $200\text{--}500\times$) on the HCl etched microscope slide.

2.4 X-ray Crystallographic Study

The X-ray data for colorless crystals of ETN were obtained at 150 K using an Oxford Cryostream low-temperature device on a Nonius KappaCCD diffractometer with $\text{Mo}-K_\alpha$ radiation ($\lambda=71.073\text{ pm}$), a graphite monochromator, and using the ϕ and χ scan mode. Data reductions were performed with DENZO-SMN [14]. The absorption was corrected by integration methods. [15] Structures were solved by direct methods (Sir92) [16] and refined by full-matrix least-square based on F^2 (SHELXL97) [17]. Hydrogen atoms were mostly localized on a Fourier difference map, however, to ensure uniformity of treatment of crystals, all hydrogen atoms were recalculated into idealized positions (riding model) and assigned temperature factors $H_{\text{iso}}(\text{H})=1.2 U_{\text{eq}}(\text{pivot atom})$. Hydrogen atoms in the methylene and methine moieties were placed with distances C–H of 97 and 98 pm , respectively.

$R_{\text{int}}=\sum|F_o^2-F_{o,\text{mean}}|^2/\sum F_o^2$, $\text{GOF}=[\sum(w(F_o^2-F_c^2)^2)/(N_{\text{diffs}}-N_{\text{params}})]^{1/2}$ for all data, $R(F)=\sum|F_o|-|F_c|/\sum|F_o|$ for observed data, $wR(F^2)=[\sum(w(F_o^2-F_c^2)^2)/(\sum w(F_o^2)^2)]^{1/2}$ for all data.

2.5 Density

密度的测定使用了水中的浮沉法在 22°C 进行。ETN从甲醇中结晶,通过溶剂蒸发得到无色的椭圆晶体,没有可见的缺陷。气泡被用温和的超声波移除。

2.6 Hygroscopicity

三个ETN样品被分别称重,样品重量在5到6g之间,然后在五氧化二磷中干燥2d。之后,样品被称重。

样品被快速称重,直到0.1mg,并放入一个装有18.6%硫酸的干燥器中(相对湿度90%被获得)。样品被在24到72h的时间间隔内称重。

2.7 Solubility in Water

ETN在水中的溶解度通过HPLC测定。三个ETN样品被用于测量,每个温度下三个样品。ETN在水中的溶解度因此在5,20,40,60和 80°C 下测定。

ETN被放在装有10mL水的烧杯中,以形成饱和溶液,并加入过量的固体ETN(ETN在水中的悬浊液)。烧杯被放入恒温箱中,并在常温下搅拌。之后,固体ETN被过滤掉(仍然在给定的温度下在恒温箱中)并4mL的ETN溶液被引入6mL的乙腈中,用吸管帮助。

该溶液被用于HPLC测量。ETN的溶解度通过ETN溶液在60/40乙腈/水的流动相中测定。测量条件和HPLC装置与2.3节中所述相同。

2.8 Friction Sensitivity

摩擦敏感性通过FKSM-08 BAM设备由OZM Research提供。BFST-Pt-100S类型的测试板和BFST-Pn-200研杵被使用,所有产品由OZM Research生产。每个样品被在五个能量水平上测量,每个水平上有十五次试验。Probit分析再次被用于评估获得的数据,并用于敏感性曲线的构建。ETN在粉末和熔融铸造形式下被检查。为此,熔融铸造ETN样品被直接滴入粗糙的测试板上,使其固化。

2.9 Impact Sensitivity

冲击敏感性通过Kast fallhammer由Reichel and Partner生产。两者都是由OZM Research生产的。Piston(BFH-SR)和cylinders(BFH-SC)被生产。

0.5kg锤子被用于MF,1kg锤子被用于PETN和ETN,2kg锤子被用于RDX。Probit分析被用于评估数据并构建敏感性曲线。每个样品被在五个能量水平上测量,每个水平上有十五次试验。ETN被在粉末和熔融铸造状态使用。粉末ETN被在体积为 40 mm^3 的条件下使用。熔融铸造ETN样品被准备成小颗粒的形式,通过将熔化的ETN滴入冷瓷砖上——颗粒大小和形状如图2所示。一个重量约20mg的颗粒被用于每次试验。



Figure 2. Shape and size of powdered ETN (left) and melt cast ETN pellets (right). Melt cast ETN was prepared by dropping of molten ETN onto a cold tile. This form was used for sensitivity measurement (melt cast ETN).

2.10 Detonation Velocity

The Detonation velocity of erythritol tetranitrate was measured using ionization probes and a digital Tektronix oscilloscope. Ionization probes were prepared using 0.1 mm twisted copper wire. Four charges of erythritol tetranitrate were prepared by filling polypropylene tubes having an internal diameter of 16.6 mm and wall thickness of 4.2 mm. Two of these tubes were filled by careful hand pressing of ETN powder. Fine powder was added in small increments in order to achieve a regular density distribution along the charge. The remaining two tubes were filled with molten ETN at 75 °C followed by cooling in a vertical position in order to obtain homogeneous charges without lumps.

2.11 Brisance

Hess's method of lead cylinder compression was applied for determination of brisance. Three charges were prepared from melt cast ETN and another three charges using powdered hand pressed ETN, each one having 50.0 g weight. A standard lead cylinder (40 mm diameter, 60 mm height) was placed on a 10 mm thick steel base plate resting on sand. It was covered by two 10 mm thick steel discs and the explosive charge was precisely positioned on the top. The lead cylinder, steel disks, and the charge were 40 mm in diameter. Initiation was conducted using a standard industrial electric detonator with aluminum cup. Powdered samples with quite low density were measured with a standard deviation of ± 0.40 mm.

2.12 Relative Explosive Strength

The relative explosive strength was determined using a ballistic mortar. Three charges were prepared from powdered ETN. The mortar was calibrated separately with powdered TNT charges, while each level of calibration was shot three times. All powdered samples were prepared by wrapping the powder in a thin polyethylene foil. Power function re-

gressions of mortar swift dependencies on the weight of trinitrotoluene charges were obtained and corresponding relative strength values were calculated using MS Excel. The explosive strength of erythritol tetranitrate was related to TNT.

3 Results and Discussion

3.1 Synthesis and Analysis of ETN

Erythritol tetranitrate was prepared by the reaction of nitric acid and sulfuric acid with erythritol (Scheme 1). The product was purified by re-crystallization. The yield of the recrystallized product was 88%. The purity of ETN was determined by using HPLC and elemental analysis. The peak area of ETN on a chromatogram is 99%. The results of C, H, and N analysis agreed with the calculated data. The structure of ETN was confirmed using X-ray crystallography (section 3.2).

The re-crystallized product forms needle like crystals (see Figure 1). The length and the aspect ratio (between length and the width) of the particles were analyzed and the histogram of these values is presented in Figure 1. Typical length of the particles is 150–350 μm . The length is typically 6 to 10-times greater than the width of each particle (see block line of the aspect ratio on the histogram).

3.2 X-ray Crystallographic Study

Crystallographic data for ETN: $\text{C}_4\text{H}_6\text{N}_4\text{O}_{12}$, $M=302.13$, monoclinic, $P2_1/c$, $a=1596.81(10)$, $b=519.40(4)$, $c=1476.09(12)$ pm, $\beta=116.238(6)^\circ$, $Z=4$, $V=1098.10 \times 10^6(15)$ \AA^3 , $D_c=1.827 \text{ g cm}^{-3}$ (-123°C), $\mu=0.189 \text{ mm}^{-1}$, $T_{\min}/T_{\max}=0.946/0.987$; $-20 \leq h \leq 20$, $-5 \leq k \leq 6$, $-16 \leq l \leq 18$; 7405 reflections measured ($\theta_{\max}=27.50^\circ$), 7364 independent ($R_{\text{int}}=0.0659$), 1686 with $l > 2\sigma(l)$, 181 parameters, $S=1.129$, $R_1(\text{obs. data})=0.0503$, wR_2 (all data)=0.0943; max., min. residual electron density=32.8, -30.8 e pm^{-3} .

ETN (Figure 3) crystallizes in the monoclinic space group $P2_1/c$ with four molecules within the unit cell – two independent ones. In both types of molecule, which are very similar from the point of view of inter-atomic distances and angles, the mixtures of R- and S-isomers are present in each molecule – a meso form as in the starting commercial erythritol [(2R,3S)-butane-1,2,3,4-tetraol]. The structure of the ETN molecule is composed of the central carbohydrate chain and two pairs of facing coplanar ONO_2 groups. Similar arrangement of the nitro groups is found as for other “sugar alcohol” derivatives as for example 3,4-dideoxy-3,4-dinitro-1,2,5,6-tetra-O-nitrohexitol [18], 1,2,3-propanetriol trinitrate [19], and pentaerythritol tetranitrate [20,21]. Significant similarities to the structure of the title compound were also found in the series of cyclic carbohydrates [22,23].

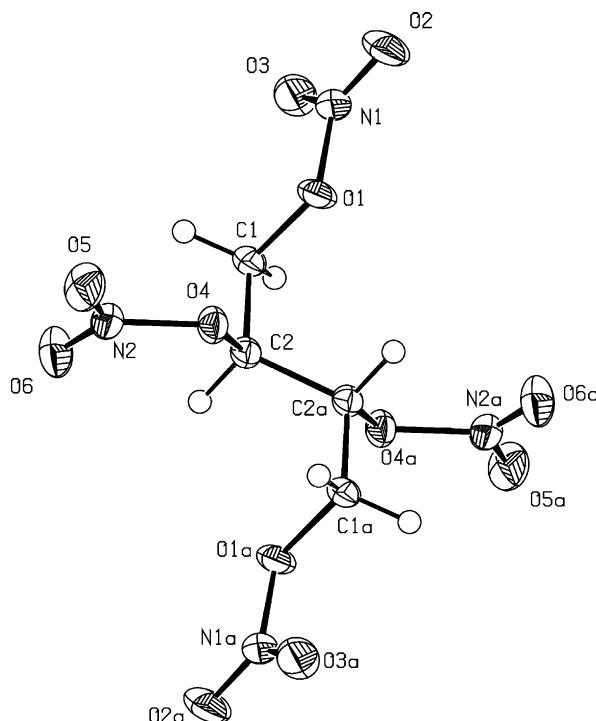


Figure 3. Molecular structure of ETN (ORTEP view, 50% probability level). The second molecule is omitted for clarity. Selected interatomic distances [pm] and angles [$^{\circ}$]: O1 N1 140.2(3), O2 N1 120.6(3), O3 N1 119.0(3), O1 C1 144.9(3), C1 C2 151.2(3), O4 N2 142.4(3), O5 N2 120.0(3), O6 N2 119.5(3), O4 C2 144.8(3), C2 C2a 153.4(4); O2 N1 O3 129.8(2), O1 N1 O2 111.9(2), N1 O1 C1 113.13(19), O1 C1 C2 104.49(19), C1 C2 O4 109.82(18), C2 O4 N2 114.65(17), O4 N2 O5 111.3(2), O5 N2 O6 130.3(2), C1 C2 C2a 114.3(2).

3.3 Density

The density of ETN determined using pycnometry was $1.716 \pm 0.005 \text{ g cm}^{-3}$, which is close to the recently published value 1.7219 g cm^{-3} [8]. The density of melt casted ETN was $1.61\text{--}1.71 \text{ g cm}^{-3}$.

3.4 Hygroscopicity

The hygroscopicity of ETN was determined at 90% humidity and 30°C . ETN was exposed to these conditions for one month. It was determined that ETN is non-hygroscopic, gaining $<0.1\%$ surface moisture over the month. The same results were obtained for PETN and RDX.

3.5 Solubility in Water

The solubility of ETN in water was determined for temperatures 5, 20, 40, 60, and 80°C . The results are summarized in Table 1.

The solubility of ETN in water is orders of magnitude lower than aqueous solubility of the lower nitroesters – methylnitrate (3850 mg per 100 mL water at room temper-

Table 1. The solubility of ETN in water.

Temperature [$^{\circ}\text{C}$]	5	20	40	60	80
Solubility ETN [mg] in 100 mL water	1.53	3.02	9.33	26.9	39.9

ature [7]), ethylene glycol dinitrate (680 mg per 100 mL of water at 20°C [7]) and nitroglycerine (180 mg per 100 mL of water at 20°C [7]). The solubility is similar to that of PETN (10 mg per 100 mL of water at 50°C [3]).

3.6 Sensitivity to Mechanical Stimuli

The Sensitivity of ETN to friction was evaluated using probit analysis. The equation $Pr = 2.8988 \ln F - 9.3258$ ($R^2 = 0.9852$) was obtained using linear regression of probits to $\ln F$ dependence for powdered ETN. For melt cast ETN; the equation $Pr = 1.0285 \ln F + 1.0283$ ($R^2 = 0.9498$) was obtained. The resulting sensitivity curves for powdered and melt casted ETN are presented in Figure 4. We compared the friction sensitivity of ETN with the sensitivities of MF, PETN, and RDX (using the same method).

The form of ETN has a significant effect on its sensitivity. The experiments showed that sensitivity of melt cast ETN to friction is about three times higher than sensitivity for powdered ETN. Sensitivity of melt cast ETN exceeds the sensitivity of PETN. Sensitivity of powdered ETN is surprisingly low, even slightly lower than RDX. The values for 50% probability of initiation (taken from sensitivity curves) for all explosives measured are summarized in Table 2.

The Sensitivity of ETN to impact was also evaluated using probit analysis. The equation $Pr = 3.6709 \ln E + 0.6401$ ($R^2 = 0.9877$) was obtained using linear regression of probits to $\ln E$ dependence for powdered ETN. For melt cast ETN; the equation $Pr = 3.4092 \ln E + 0.4552$ ($R^2 = 0.9531$) was obtained. The resulting sensitivity curves for powdered and melt casted ETN are presented in Figure 5. The values for 50% probability of initiation (taken from sensitivity curves) for all explosives measured are summarized in Table 2. Fric-

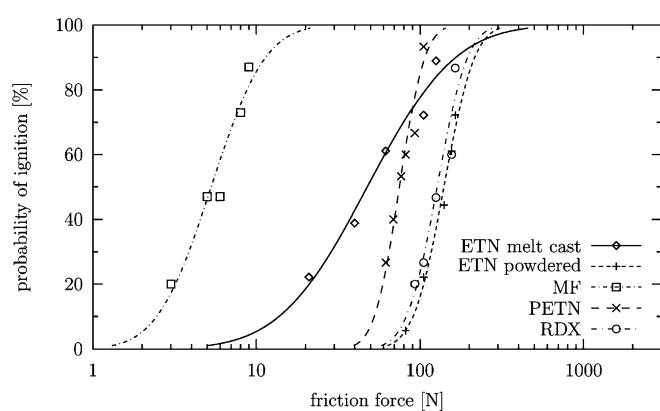
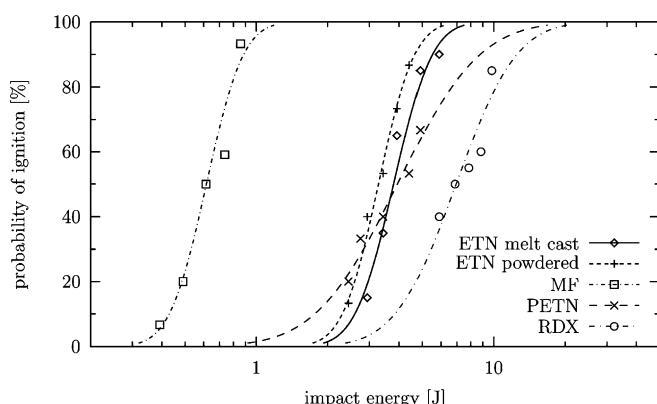


Figure 4. Comparison of friction sensitivity of ETN with that for MF, PETN, and RDX.

Table 2. Sensitivity to impact (Kast fallhammer) and friction (BAM type apparatus).

	Impact energy for 50% probability of initiation [J]	Friction force for 50% probability of initiation [N]
MF	0.62	5.3
ETN (powdered)	3.28	140
ETN (melt cast)	3.79	47.7
PETN	3.93	75.1
RDX	6.93	127

**Figure 5.** Comparison of impact sensitivity of ETN with that for MF, PETN, and RDX.

sensitivity of ETN was compared with sensitivities for MF, PETN, and RDX (using the same method).

The form of ETN does not significantly affect sensitivity to impact unlike the friction sensitivity. Powdered ETN is only slightly more sensitive than melt cast ETN. Sensitivity of ETN to impact is about the same as that for PETN and higher than that for RDX. A similar result was obtained by Oxley et al. [8]. However, our absolute values differ from the values obtained by Oxley et al. The reason for this is the use of different apparatus and methods for measuring and evaluation. Therefore, it is always necessary to specify values for comparison using standard explosives.

The values of impact sensitivity of comparative explosives (PETN and RDX) in this study significantly differ from our previously published data [24]. The probable reason for this difference was the use of older types of pistons and cylinders in the previous measurements. Older types of pistons and cylinders were out of range of the diameter tolerance requested by STANAG 4489. Other conditions of measurement, methodology, and measuring apparatus were the same as in our previous study.

3.7 Explosive Characteristics

The detonation parameters for ETN have not been published to date, although the explosive has been known for over 150 years.

The detonation velocity of ETN was determined for powdered ETN and for melt cast ETN. Two identical values were obtained for both measured samples of melt cast ETN (density 1.69 g cm^{-3}). In the case of powdered ETN, a value of 4420 m s^{-1} (density 0.83 g cm^{-3}) was obtained for the first sample and 4630 m s^{-1} (density 0.86 g cm^{-3}) for the second sample. The values are summarized in Table 3 and com-

Table 3. Detonation velocities of ETN.

	Detonation velocity [m s^{-1}]		
	(density in parenthesis [g cm^{-3}])		
ETN	4420 (0.83)	4630 (0.86)	7940 (1.65)
ETN [25]	4760 (0.80)	5170 (0.88)	8100 (1.66)
Melt cast			

pared with values for PETN (taken from the literature for the closest densities). As can be seen from Table 3, the detonation velocities for ETN are a little bit lower than for PETN. The detonation velocity of ETN 7940 m s^{-1} (density 1.69 g cm^{-3}) is in agreement with a recently predicted value of 8206 m s^{-1} (density 1.7219 g cm^{-3}) that was calculated by Cheetah 6.0 [8].

The Hess test was used for determination of brisance. The result for powdered ETN is presented in Table 4 in comparison with TNT and Semtex 1A at identical conditions.

Table 4. Brisance of ETN.

	Density [g cm^{-3}]	Brisance according to Hess [mm]
ETN powdered	0.80	14.8
TNT	1.20	18.8
Semtex 1A	1.45	21.0

Melt casted samples at a density of $1.68\text{--}1.71 \text{ g cm}^{-3}$ failed to provide valid data because of excessive brisance leading to flower-like deformation of lead cylinders. In all cases, compression exceeded 26 mm.

The relative explosive strength of ETN was measured by the ballistic mortar method. We obtained 143% TNT for powdered ETN. This value is about the same as for PETN (145% TNT is reported in reference [26]) and nitroglycerine (140% TNT [26]).

4 Conclusions

Erythritol tetranitrate (ETN) belongs to the improvised explosives that are widely prepared by both amateur chemists and criminals nowadays. This nitric acid ester can also be potentially useful for some industrial applications.

The synthesis, analyses, physical, and explosive properties of ETN were described. ETN was prepared with an 88% yield by the action of nitric acid/sulfuric acid on erythritol. The purity of the product was clarified using elemental analysis and HPLC. Molecular structure of ETN was characterized by single-crystal X-ray diffraction. The crystal density of ETN provided by X-ray diffraction was 1.827 g cm^{-3} . ETN was found to be non-hygroscopic compound slightly soluble in water.

The sensitivity of melt cast ETN to friction significantly differed from powdered ETN. Melt cast ETN was more sensitive to friction than PETN, while powdered ETN was slightly less sensitive than RDX. Sensitivity of powdered ETN to impact was slightly lower than that of melt cast ETN that was on the level of PETN. Detonation velocity of melt cast ETN was 7940 m s^{-1} at a density of 1.69 g cm^{-3} , which is slightly below the PETN level. The relative explosive strength of ETN was 143% TNT that is also close to PETN.

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