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Ethylene Glycol Dinitrate (EGDN): from Commercial Precursors, Physicochemical and Detonation Characterization*)

Hichem FETTAKA*, Michel LEFEBVRE

Royal Military Academy, Department of Chemistry,
Laboratory for Energetic Materials,
Av. de la Renaissance 30, 1000, Brussels, Belgium
** E-mail: h_fettaka@outlook.fr*

Abstract: Currently, liquid explosives pose a potential threat. An important phase in the assessment of this threat is to investigate the various synthesis paths leading to their manufacture and to evaluate the potential use of readily purchasable precursors.
The aim of this work was to assess the synthesis of Ethylene Glycol Dinitrate (EGDN), a liquid nitrate ester explosive, using commercially available precursors. The characteristics of the synthetic process (ease, yield), the chemical properties of the synthesized product (purity, spectra) and its explosive properties (sensitivities, detonability) were investigated. Comparisons are drawn between these products and the product obtained using laboratory ingredients.
Three ingredients have been used: 1) ethylene glycol, laboratory grade, 2) ready to use, commercial coolant fluid, and 3) ethylene glycol extracted from commercial coolant fluid.
The chemical composition and purity of the synthesized liquid explosive was analyzed by Gas Chromatography-Mass Spectrometry (GCMS), and infrared spectroscopy (IR). Differential Scanning Calorimetric (DSC) analysis allowed the heat of decomposition and activation energies to be assessed. The Ozawa and Kissinger models were used. The explosive properties of the pure synthesized products and comparable other explosives, have been tested. The potential use as a priming charge or as a main charge was assessed.

Keywords: EGDN, liquid explosive, precursor, activation energy, detonation tests

*) Some results included in this article have been orally presented and included in the Proceeding of the 17th NTREM Seminar, Pardubice the Czech Republic, 2014.

1 Introduction

Home-made explosives are synthesized from common precursors that are commercially available [1]. Hexamine, hydrogen peroxide and fertilizers are the most familiar precursors commonly used in manufacturing home-made explosives like RDX, TATP and Ammonium Nitrate, and are therefore subject to some regulations [2]. Other chemical precursors are accessible and available with little or inadequate regulation, and therefore constitute a potential threat.

Liquid explosives are synthesized from one or more liquid precursors and can be initiated through mechanical stimuli, friction or heat [3]. EGDN (Ethylene Glycol DiNitrate, also known as nitroglycol) belongs to the category of nitrate ester liquid explosives. This colourless and oily liquid can be obtained by nitration of ethylene glycol (di-alcohol) [4].

Except in some propellant formulations, EGDN does not actually have many military applications because of its high vapour pressure. It is 100 times more volatile than nitroglycerin. EGDN is sometimes used as gelatinous agent and/or is mixed with nitroglycerin in several kinds of dynamite to reduce its freezing temperature. EGDN is a secondary explosive with a neutral oxygen balance, therefore it has a large power index which makes it a powerful explosive [1, 3-5]. Exposure to a high concentration of this liquid can affect the blood circulation and health [6, 7].

Coolant fluids used in engines are in most cases composed of more than 30% of ethylene glycol, the other components being water and generally an anticorrosion agent. They constitute the main accessible source of ethylene glycol for EGDN synthesis [8, 9].

The present work consists of assessing the properties of EGDN synthesized from coolant fluid, and then comparing the characteristics of this synthesized explosive with those of two other samples synthesized respectively from a laboratory grade sample and a sample derived by distillation of the coolant fluid. The three precursors and their corresponding synthesized products were fully characterized. The impact sensitivities were also determined. Finally, the synthesized EGDN was tested for brisance and compared to the brisance of conventional explosives (TNT and C4).

2 EGDN Synthesis

EGDN is a nitrate ester (see Figure 1) and in this work was synthesized from three sources:

- The laboratory grade ethylene glycol, further referred to as (EG.Pur), provided by Fluka Chemika.
- Commercial coolant fluid that was used without any pretreatment (EG.Comp).
- Ethylene glycol extracted by distillation of the commercial coolant fluid (EG.Dist).

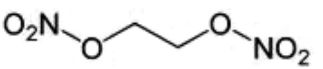

Figure 1. EGDN molecule.

The synthesis of the EGDN samples followed different procedures, using sulfonitric mixtures with appropriate control of the physical parameters, and following all safety precautions and good laboratory practice. EGDN was produced according to the following reaction:

$$C_2H_6O_2 + 2HNO_3 \xrightarrow{H_2SO_4} C_2H_4N_2O_6 + 2H_2O \quad (1)$$

After the synthesis, a short wash with distilled water was carried out. The products were then further washed with aqueous sodium carbonate solution in order to neutralize the residual acids. This operation was followed by further washing with distilled water until pH neutral. The product yield was between 60 and 64%. In total, three samples were obtained:

1. One prepared from laboratory grade ethylene glycol, called hereafter EGDN.Pur.
2. One produced from the untreated commercial coolant fluid, called EGDN.Comp.
3. One obtained with ethylene glycol distilled from coolant fluid, called EGDN.Dist.

3 Physicochemical Characterization of the Precursors

3.1 GCMS Analysis

The Gas Chromatograph (GC) used was an Agilent 6890 series connected with an Agilent 5973 network Mass Spectrometer (MS). The column was DB5, having dimensions 30 m × 0.32 mm, and helium flowing at a rate of 1 mL/min was used as the carrier gas.

Prior to GCMS analysis, a quantity (0.5 µL) of each sample was dissolved in dichloromethane (2 mL) and a sample (1 µL) of the resultant solution was introduced using the splitless mode. The injection port temperature was kept at 180 °C. The initial oven temperature was 35 °C for 3 min, and was then heated at a linear rate of 20 °C/min until 250 °C, and finally kept at this temperature for 5 min.

The MS quadripol temperature was set at 150 °C and the MS source temperature was 230 °C. The solvent delay time was 2.2 min. The MS was operated in the scan mode (30-300 amu).

The chromatograms of the three different precursors are given in Figures 2(a)-(c**)**. These results shown that the retention times of ethylene glycol were 3.85, 3.83 and 3.84 min for EG.Pur, EG.Dist, and EG.Comp, respectively.

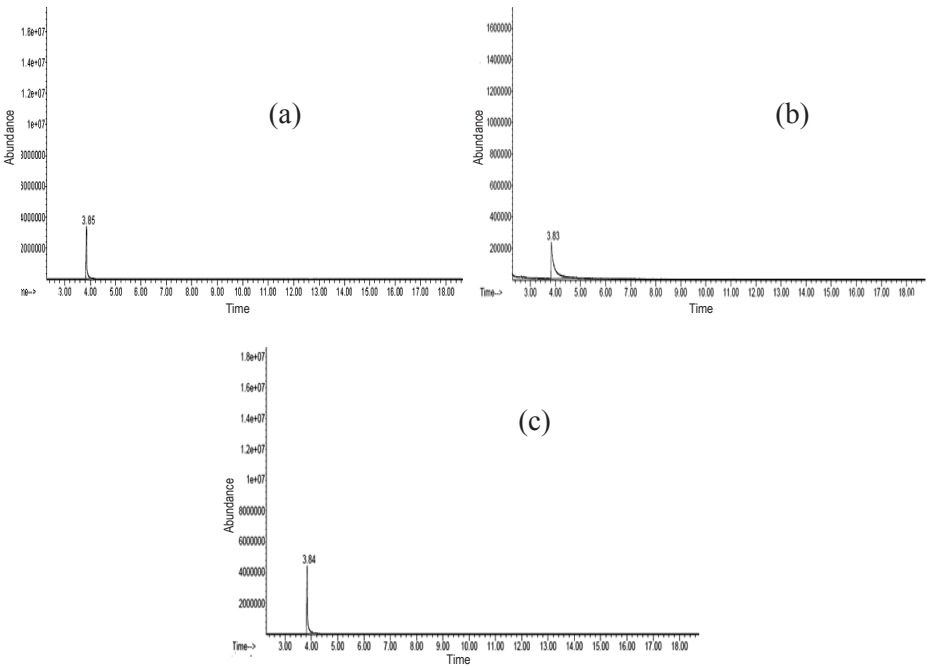

Figure 2. Chromatograms of ethylene glycol: (a) laboratory grade ethylene glycol, (b) distilled ethylene glycol, (c) commercial coolant fluid.

According to Figures 2(a) and (b), it is clear that the laboratory grade ethylene glycol and the distilled one were pure compounds. There were no other significant peaks, accounting for the quasi absence of impurities. The EG.Pur and EG.Dist peaks obtained are very similar in both their overall shape and intensity. As the

untreated commercial coolant fluid chromatogram (Figure 2(c)) does not show any impurities either, we may speculate that the other molecules present in the precursor cannot be detected using the stated operating conditions.

Figures 3(a)-(c) show the mass spectra of the three precursors. The first observation was that the entire mass spectra are similar. The base peak in the mass spectra of the samples was at m/z 31, corresponding to CH_3O. Other peaks appeared at m/z 43 and 62, which can correspond to C_2H_3O and the $C_2H_6O_2$ (EG without fragmentation) respectively.

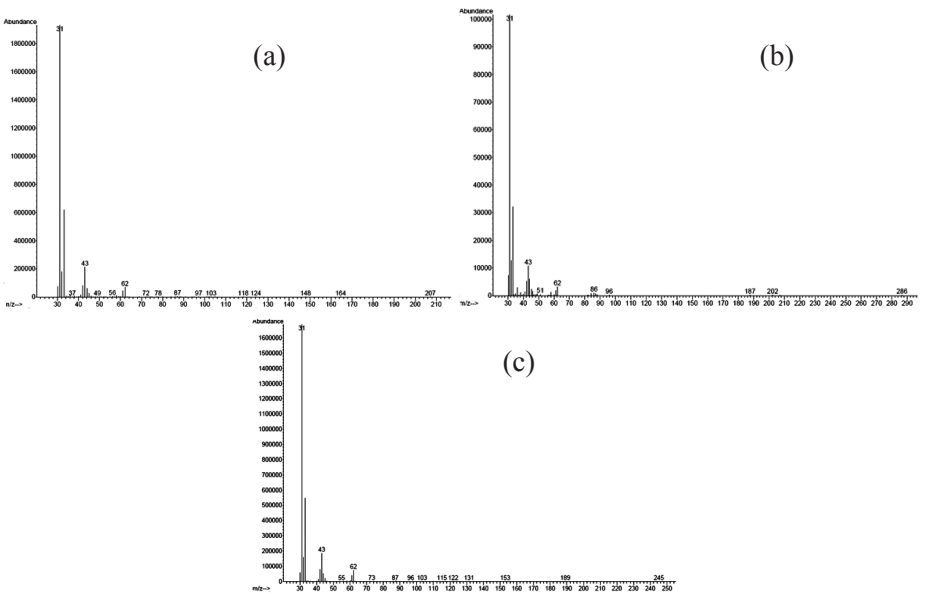

Figure 3. MS spectra of ethylene glycol: (a) EG.Pur, (b) EG.Dist, (c) EG.Comp.

3.2 FTIR spectroscopy

Fourier Transform Infrared Spectra were recorded with a Metler Tolido FTIR by averaging 60 scans, in the 4000-400 cm^{-1} spectral range, at a resolution of 1 cm^{-1}. A quantity (1 µL) of each sample was analyzed in the ATR mode. FTIR spectra provide qualitative and quantitative information. In this investigation, it gave information about the composition of each precursor (Figure 4). The perfect overlap of the three spectra indicates the same composition of the precursors.

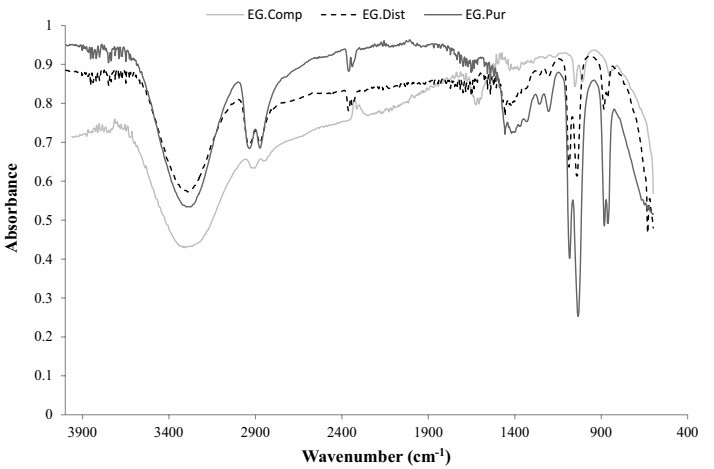

Figure 4. FTIR spectra of the three precursors.

All spectra show a broad absorption band at 3000-3600 cm^{-1} with a maximum at about 3370 cm^{-1}, which is attributed to the O–H stretching of hydroxyl groups and probably some water. The bands around 2943 and 2923 cm^{-1} are due to the stretching vibration of C–H and bands around 1460 are related to the bending vibration of C–H. Three peaks at 1215, 1084, and 1037 cm^{-1} correspond to C–O bond of alcohol and other bonds [14, 15].

The use of the same volume for all samples allowed the absorbance intensity of the three precursors to be compared. We noted a progressive decrease in the spectrum intensity, especially for absorbances below 1500 cm^{-1}. Pure laboratory grade ethylene glycol exhibits the highest absorbance intensity, followed by EG.Dist. The untreated commercial coolant fluid presents the lowest absorbance intensity. This is consistent with the EG-content of each sample. Nevertheless, a band at about 1600 cm^{-1} only appears in the EG.Comp spectrum; this band could not be identified. It is probably related to another compound from EG.

4 Physicochemical Characterization of the Products (EGDN)

4.1 GCMS analysis

The synthesis of EGDN also gives water molecules (Reaction 1) which are well mixed with the EGDN. The separation of the two substances is a difficult step because the density of EGDN is close to that of water (1.48 g/cm^3 [4]). The full production of EGDN results in a rather low yield (60 to 64%).

According to the results shown in Figures 5(a)-(c), the three samples EGDN.Pur, EGDN.Dist and EGDN.Comp were found to elute at 8.44, 8.46 and 8.44 min, respectively. The GCMS pro-analysis of a standard of ethylene glycol dinitrate (EGDN.Std), purchased from Accu-Standard. It shows that the retention time of the EGDN molecule is 8.43 min. The difference between the synthesized EGDNs and the standard is negligible.

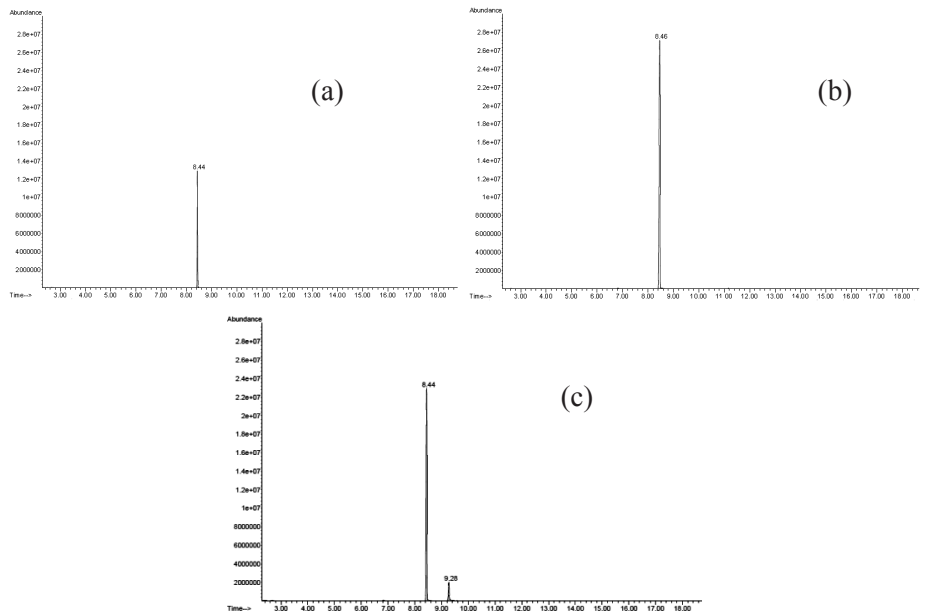

Figure 5. Chromatograms of EGDNs: (a) EGDN.Pur, (b) EGDN.Dist, (c) EGDN.Comp.

According to these results, the same peak appeared for the three synthesized samples. The mass spectra, represented in Figures 6(a)-(c), show the characteristic fragmentations corresponding to EGDN. It seems that all of the synthesized ethylene glycol dinitrate samples, EGDN.Pur, EGDN.Dist, and EGDN.Comp, exhibited the characteristic fragmentations of the EGDN molecule, which are m/z: 30 (NO, CH_2O), 46 (NO_2), and 76 (CH_2NO_3) [16].

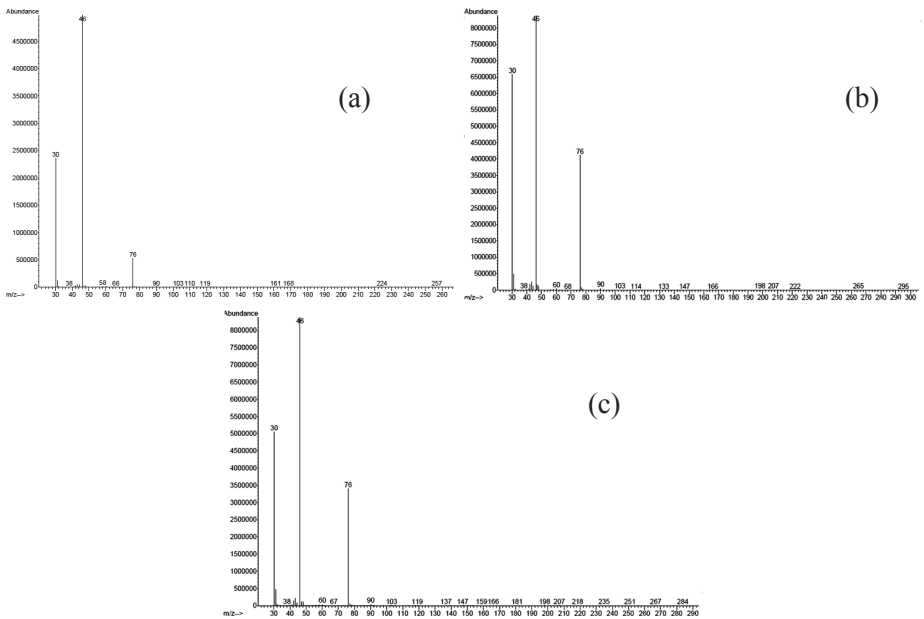

Figure 6. MS spectra of EGDN samples: (a) EGDN.Pur, (b) EGDN.Dist, (c) EGDN.Comp.

Another observation was the presence of a second peak which appeared in the case of EGDN.Comp, with a retention time of 9.27 min (Figure 6(c)). The mass spectrum corresponding to this peak corresponds to 2-ethylhexanoic acid. This substance is an anticorrosion agent in commercial coolant fluids [8, 17]. This agent was detected in the EGDN chromatogram but not in the precursor one, despite the fact that the same operating conditions were used. This is probably due to the presence of a strong Hydrogen Bond between the OHs of the di-alcohol and the O of the carboxylic acid when all OH bonding sites are consumed (through the production of EGDN), the anticorrosion agent is released and observed in the product.

4.2 FTIR spectroscopy

Figure 7 shows the FTIR spectra of the EGDN samples. The first observation is the complete absence of the band corresponding to the hydrogen bonded OH of the alcohol at 3000-3600 cm^{-1}.

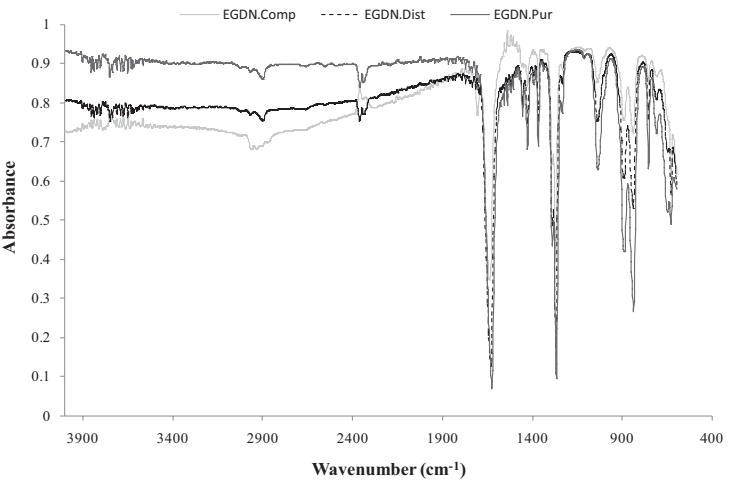

Figure 7. FTIR spectra of the EGDNs produced.

This phenomenon is accompanied by the appearance of two new bands at 1635 and 1270 cm^{-1}, related NO_2 groups [5].

It is clear that the FTIR spectra of EGDN.Pur and EGDN.Dist have almost the same intensity, especially for the bands characterizing the NO_2 groups. However, the EGDN.Comp spectrum is less intense; this could be related to impurities.

On the other hand, a peak at 1700 cm^{-1} was observed for EGDN.Comp. This is characteristic of the C=O stretching vibration of a carboxylic acid group. This result is consistent with the GCMS analysis, where a carboxylic acid used in the commercial coolant fluid as an anticorrosion agent was identified.

4.3 DSC analysis

Samples (1 mg) were tested under a nitrogen flow in cylindrical high pressure crucibles using DSC822 Mettler Toledo equipment, which had been calibrated with indium and zinc. The samples were heated from 50 to 280 °C at four different heating rates ($\beta = 1$, 3, 5 and 8 °C/min). The onset temperature was determined as the intersection point on the base line with the leading profile of the peak corresponding to decomposition.

The activation energies (E_a) were obtained by applying the Ozawa and Kissinger models, expressed by the following equations [10, 11]:

The Kissinger method:

$$\frac{2.303\,d\left[\log\left(\frac{\beta}{T_{\max}^2}\right)\right]}{d\left(\frac{1}{T_{\max}}\right)} = -\frac{E_a}{R} \tag{2}$$

where R is the universal gas constant, β is the heating rate, T_{max} is the temperature corresponding to the maximum in the DSC exothermic peak at a specific heating rate.

The activation energy can be calculated from the slope of the linear plot of $\log\left(\frac{\beta}{T_{\max}^2}\right)$ versus T_{max}^{-1}.

The Ozawa method uses the following equation:

$$\frac{2.15\,d[\log(\beta)]}{d\left(\frac{1}{T_{\max}}\right)} = -\frac{E_a}{R} \tag{3}$$

In this case, E_a can be calculated from the slope of the plot of $\log(\beta)$ versus T_{max}^{-1}.

The thermograms of Figure 8 show a significant increase in the onset temperatures as the heating rate is increased. This table also provides the heat of decomposition (ΔH).

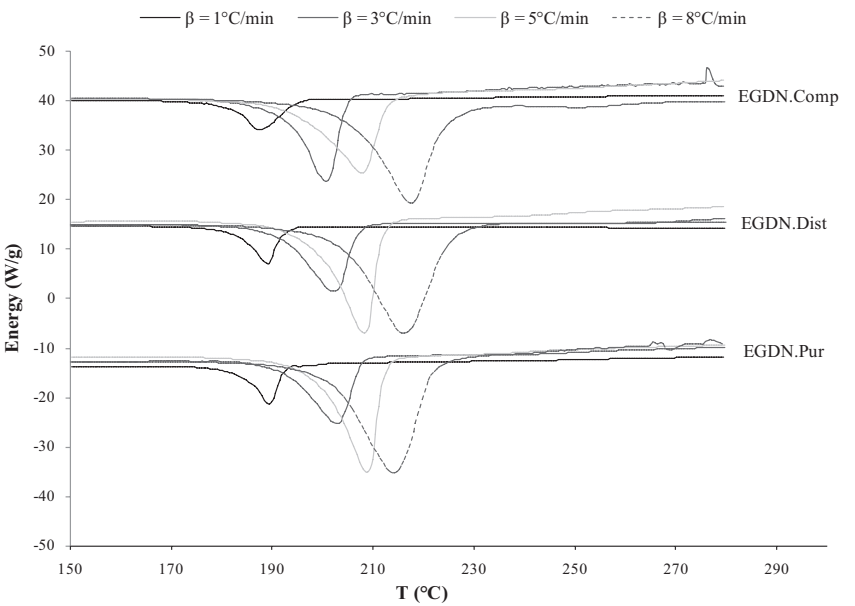

Figure 8. DSC thermogram of EGDN samples at different heating rates.

In order to understand the effect of the precursor on the heat of decomposition, we have calculated the ratios of the heat of decomposition:

$\Delta H1$: $\Delta H_{EGDN.Dist}/\Delta H_{EGDN.Pur}$

$\Delta H2$: $\Delta H_{EGDN.Comp}/\Delta H_{EGDN.Pur}$

$\Delta H3$: $\Delta H_{EGDN.Comp}/\Delta H_{EGDN.Dist}$

The results obtained are shown in Figure 9. The $\Delta H1$ is almost equal to 1 for all heating rate values, indicating that the heat of decomposition of EGDN.Pur and EGDN.Dist are very close. However, when comparing EGDN.Pur with EGDN.Comp, more fluctuations are found in the ratios. The ratio $\Delta H3$ also present the same qualitative variation. Nevertheless, no calculated ratios exceeded a value of 1.29 and we may assume that all of the synthesized samples have similar heats of decomposition.

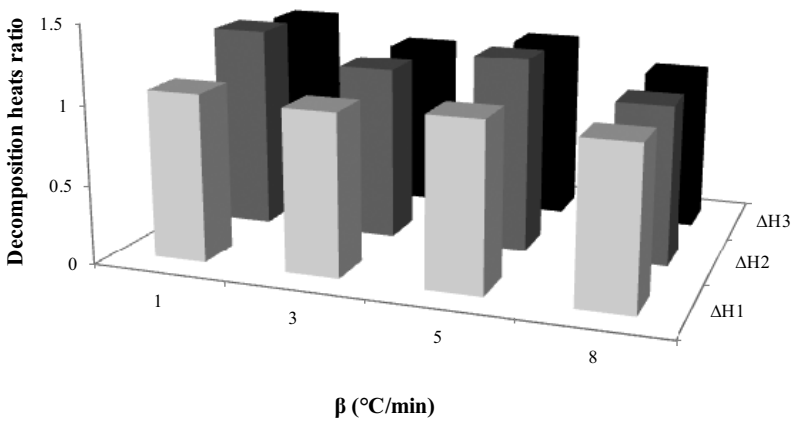

Figure 9. Effect of precursors on the heat of decomposition of EGDN samples.

For each type of EGDN, the Kissinger and the Ozawa models were applied for the determination of the activation energy. The results obtained are given in Table 1.

Table 1. E_a determination of EGDNs by the Kissinger and the Ozawa methods

Explosive	Ozawa		Kissinger	
	Correlation factor (R^2)	E_a [kJ·mol^{-1}]	Correlation factor (R^2)	E_a [kJ·mol^{-1}]
EGDN.Pur	0.999	145.06	0.999	148.04
EGDN.Dist	0.999	136.63	0.995	138.46
EGDN.Comp	0.988	123.46	0.986	124.36

The E_a of EGDN.Pur by the Kissinger method had exactly the same value of 148 kJ/mol as one given in the literature [18]. However, the E_a of EGDN.Dist and EGDN.Comp are significantly lower. This could be due to the presence of impurities, especially in the case of EGDN.Comp. Despite the differences in activation energies of the three explosives, all of the values were of the same order of magnitude.

Both methods (Kissinger and Ozawa) gave quite close quantitative results and lead to analogous conclusions.

5 Detonation Characterization

5.1 Sensitivity to impact test

A BAM Fall hammer device and the Bruceton test procedure were used to obtain the sensitivity to impact of the synthesized EGDN samples. The procedure consists of placing a volume (40 μL) of the sample under a cylindrical, fall hammer. A steel weight attached to a fixed, vertical rail was raised to a known height and released. For a given weight, the height of the hammer is varied. The Bruceton procedure allows some statistical data to be obtained from the test, namely the sensitivity to impact at 50% reaction and the standard deviation [12, 13].

For a steel weight of 1 kg, the tests were positive for all heights, so sensitive that a "non-standardized" drop weight was needed. Subsequently a weight of 0.534 kg, dropped at a height of less than 5 cm, gave six successive negatives. According to the results obtained following this impact test (Table 2), it seems that EGDN.Pur and EGDN.Dist had practically the same sensitivity.

Table 2. Impact energies of the explosives

Explosive	Weight [kg]	Distance [cm]	Energy [J]
EGDN.Pur	0.3542	5	0.177
EGDN.Dist	0.3542	5	0.177
EGDN.Comp	0.3542	5	0.106

In order to confirm the results of the impact test, the Bruceton statistical method has also been applied for all of the types of EGDN. The objective was to calculate a Dh_{50}. A series of 25 tests were conducted with the same instrument, the weight being fixed and the height changed for each positive or negative test. The E_{50} values, defined as a 50% impact height related to an event "go"

for positive tests and "no-go" for negative tests [13], for all of the samples are reported in Table 3. The Bruceton result details are mentioned in [19].

Table 3. Bruceton test results of EGDN

Explosive	E_{50}, [J]
EGDN.Comp	0.247
EGDN.Dist	0.136
EGDN.Pur	0.133
Literature [4]	0.200

E_{50}: energy relating to Dh_{50}

The energy E_{50} of EGDN.Dist and EGDN.Pur are practically the same. However, the E_{50} of EGDN.Comp exhibits a higher energy, contrary to the results of the six successive tests method. Indeed, EGDN.Pur and EGDN.Dist are relatively pure, and probably the presence of air bubbles creates a hot spot by decompression, increasing their sensitivity and therefore reducing their impact sensitivity values. The Bruceton test results are more accurate than the six successive tests, nevertheless, EGDN remains quite sensitive as an explosive.

5.2 Brisance test

Only EGDN.Comp has been assessed for its brisance index; it is the most interesting case because the untreated commercial coolant fluid was used in the synthesis.

The EGDN.Comp brisance was assessed according to the test described in Figure 10, in which a steel tube filled with the explosive was fixed onto an aluminum witness plate type Al-6082 ($50\times50\times10$ mm^3). A detonator was positioned above the tube. After detonation (see Figure 11), both the depth of the dent and the volume of the dent are representative of the brisance of the explosive. These are measured using two techniques:

- A digital probe for the depth,
- A K-Scan MMDX for the volume. The principle consists of laser scanning of the aluminum plate. The instrument was equipped with a camera in order to measure all metrological parameters such as volume and maxima of the y, x and z axes.

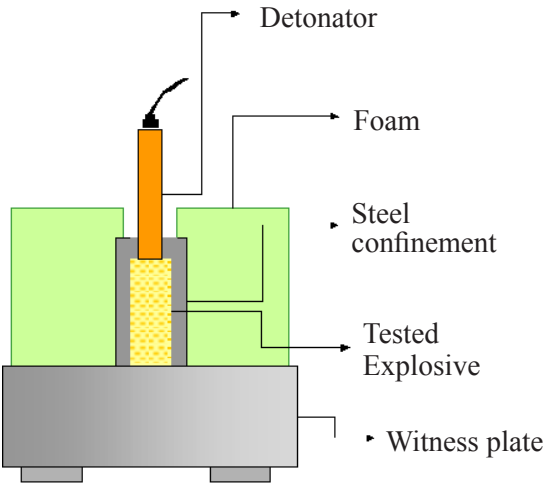

Figure 10. The steps followed to carry out a brisance test.

For the purposes of comparison, tests were also carried out with two other explosives, TNT and C4 (PBX). The densities of EGDN, TNT and C4 were 1.48, 1.61 and 1.68, respectively.

5.2.1 The Dent method

The measured brisances are given in Figure 15. We assume that at the intersection of the two linear parts, a steady detonation state has been achieved. According to the curves of Figure 12, EGDN.Comp, TNT and C4 reached a steady state detonation after 21 mm. The observed dent at these heights is thus assumed to be characteristic of the brisance of the sample. The EGDN.Comp, with a density of 1.48, is more brisant than TNT with a density of 1.61, as the scattering is proportional to the square of the velocity; this is associated with the fact that the detonation velocity of EGDN.Comp is higher than that of TNT. In addition, calculating the brisance index, defined as the average of the ratios (dents or volumes) on the witness plate, between the explosive and TNT, shows that EGDN.Comp was 1.15 times more brisant than TNT (Table 4).

Figure 11. Plate dent and fragments after explosion.

Table 4. Brisance test results of samples of different lengths

Tube dimensions (length × Φ_{inner} × Φ_{ext}) mm	TNT		EGDN.COMP		C4.PBX	
	h_{TNT}, [mm]	B_{TNT}	h_{EGDN}, [mm]	B^{*}_{EGDN}	h_{C4}, [mm]	B^{**}_{C4}
10 × 9.8 × 13.9	4.27	1	4.99	1.17	5.35	1.25
20 × 9.8 × 13.9	5.57	1	6.39	1.21	7.04	1.34
30 × 9.8 × 13.9	6.40	1	7.03	1.10	7.38	1.15
40 × 9.8 ×13.9	6.45	1	7.32	1.13	7.64	1.19
Average of B	1		1.15		1.23	

B: Brisance index; B^{*}_{EGDN}: h_{EGDN} / $h_{TNT,}$ B^{**}_{C4}: h_{C4} / h_{TNT}

As expected, the brisance of C4 is the highest, when compared with that of EGDN.Comp and TNT.

5.2.2 The Volume method

The volumes of the Al-witness plate craters after detonation are reported in Figure 13. The curves this case are similar to those obtained using dents (Figure 15). However, despite the fact that the detonation regime was not completely reached in the case of TNT and EGDN, compared to the dent method, the detonation steady state using the volume method is achieved after 27 mm. This can be explained by spherical propagation of the detonation wave, and therefore, in the tube, the axial detonation steady state is reached before the volume detonation steady state.

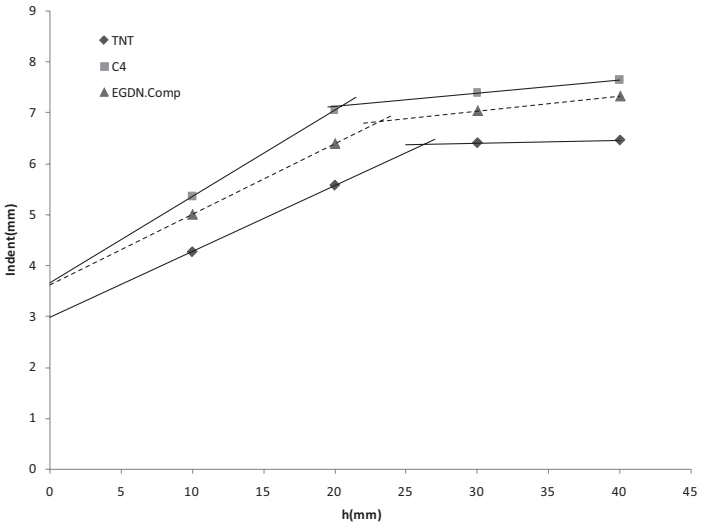

Figure 12. Brisance curves of EGDN.Comp, TNT, and C4, using the dent method

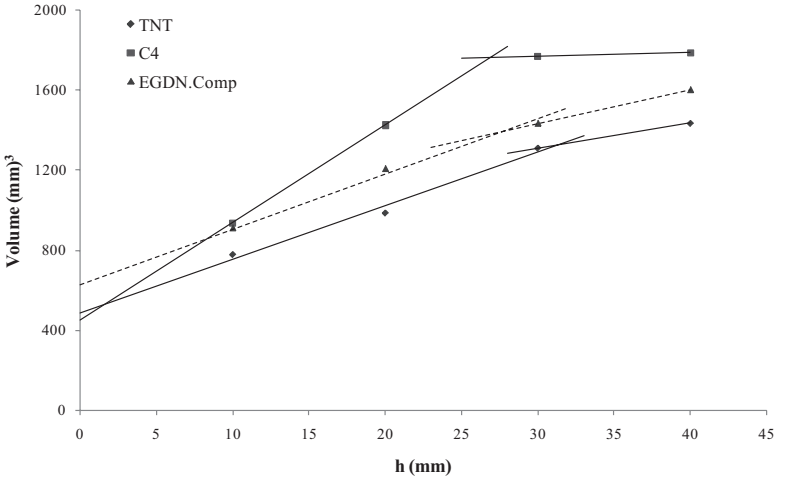

Figure 13. Brisance curves of EGDN.Comp, TNT, and C4, using the volume method.

The brisance index, calculated using the measured volumes show that EGDN brisance is 1.15, fully consistent with the dent measurement. Nevertheless the volume technique is still a more representative method because in the dent

technique, only one point on the Al-plate is selected while the whole crater is scanned in the volume technique.

According to Paul Cooper [20], the TNT-equivalent can be estimated using the following equation:

$$\text{Equivalent TNT} = D^2(\text{EGDN})/D^2(\text{TNT}) \quad (4)$$

where D is the detonation velocity.

The Table 5 shows literature information related to the TNT-equivalent of EGDN explosive.

Table 5. Equivalent-TNT of EGDN calculated using literature data

Designation	TNT	EGDN
Detonation velocity, [m/s] [4]	6900	7300
TNT-equivalent of EGDN	1.119	

The experimental brisance index determined by the witness plate method, irrespective of the assessment procedures (dent or volume), is consistent with the TNT-equivalent of EGDN reported in the literature.

6 Conclusions

This work has investigated the process of nitroglycol synthesis using a commercial source of ethylene glycol (coolant fluid).

A comparative study was performed using two other EGDN samples; one synthesized with laboratory grade ethylene glycol and the other with ethylene glycol obtained by distillation of the commercial coolant fluid.

The operating conditions of GCMS used in this work were not established to separate the different components of the coolant fluid but, these conditions were set to identify ethylene glycol and EGDN. The identification of traces of the anticorrosion agent after nitration (EGDN.Comp), and not in the initial ingredient (EG.Comp), although they were subjected to the same operating conditions, reflects the presence of a strong hydrogen bond. The latter is diminished by the nitration reaction, physically liberating the anticorrosion molecule, which can then be identified by GCMS.

The yield of EGDN from the coolant fluid was about 60%, which seems quite high taking into account that we limited this investigation to simple technology and synthetic procedures.

The EGDN made from the commercial coolant fluid has the same physicochemical properties as the two other explosives, despite the presence of 2-ethylhexanoic acid, as shown by the GCMS analysis. The presence of the anticorrosion agent did not affect the characteristics of the explosive. The calculation of the heat of decomposition and activation energies, using both the Kissinger and Ozawa models from the DSC analyses, shows a negligible difference in this parameter.

In addition, identical explosive sensitivity was obtained. The impact sensitivities of the three products are similar, with more confidence in the Bruceton method.

The EGDN synthesized from the commercial ingredient (EGDN.Comp) has been used for the witness plate test. It is more brisant than the conventional TNT; this result was confirmed by using two techniques and comparison with the calculated theoretical value.

Acknowledgments

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