



## Fortification of W/O Emulsions by Demilitarized Explosives. Part I. Use of TNT

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**Abstract:** This paper presents a study of the relationship between detonation velocities,  $D$ , and relative explosive strength (RS) of fortified W/O emulsion explosives, on the one hand, and the grain size and quality of the demilitarized TNT used for this fortification, on the other. It has been found that demilitarized TNT having a grain size below 400  $\mu\text{m}$  could give acceptable results both in terms of initiation and detonation velocity and the RS values. Demilitarized TNT, however, is not a reliable sensitizer even in a micronized state, and therefore using TNT necessitates simultaneous sensitizing of the final W/O explosive with microballoons and it has no significantly better influence on the performance of the resulting explosives. The small-scale cook-off test of such a W/O mixture fortified by 50% by wt. of TNT has shown that such a mixture is a relatively safe explosive.

**Keywords:** demilitarized explosives, detonation, emulsion, TNT

## Introduction

Water-in-oil type (W/O) emulsion explosives are suitable matrices for introduction of brisant explosives that have been eliminated from ammunition (demilitarized explosives, DEs) [1-7]. DEs are usually available in the form of a mixture of the energetic material and a binder (e.g. petroleum jelly,

polyisobutylene, polyurethanes, waxes etc.) in various forms (cylinders, powders, flakes, amorphous particles etc.) and various sizes (ranging from  $10^{-4}$  to  $10^{-2}$  m). All these parameters are important in the process of reworking to obtain industrial explosives. This incorporation of DEs into emulsion explosives [1-5] is economically effective, safe and, at the same time, environment-friendly.

There exists a critical amount of high explosive (HE) required as an admixture in W/O emulsion to be sufficient for a reliable initiation of resulting mixture; this content represents at least 30% by wt. of HE [8-10]. The Refs [8-10] mentioned, and others, describe studies of the effects upon the detonation and performance parameters of emulsion explosives observed after their fortification with multi-perforated (seven times perforated) fine-grain powders [8, 9] and with RDX and PETN [9, 10]. The older papers [8, 9] show that incorporation of HEs into the matrix of W/O-type emulsion explosives increases their performance (working ability) to such an extent that the fortified W/O-type emulsions shift from being the equivalent of loose-material ammonium nitrate explosives to approach the level of dynamites.

A somewhat different situation is encountered in the case of incorporating TNT into the W/O matrix; in this context the published studies [4, 5] describe the use of micro-ground TNT with simultaneous sensitizing by addition of microballoons (MBs). However, no description is presented about the behavior of W/O-type mixtures fortified by addition of “current-quality” TNT (from dismantling of ammunition), with 50% by wt. filling of the emulsion matrix, without sensitization by MBs. Also, the behaviour of such a modified emulsion in cook-off tests has not yet been studied. These issues are dealt with in the current paper.

## Experimental

### Preparation of samples

The details of the W/O emulsion explosives studied are shown in Table 1; mixtures marked as A, and B (i.e. without fortification) are called Blasting Agents. The matrices were prepared using a simple apparatus consisting of a thermostat and a container equipped with a stirrer. The solution of oxidizers was heated to a temperature of 95-100 °C and then slowly added to the container in which a preheated mixture of the fuels (paraffin and wax) with the emulsifier (95 °C) was agitated with the stirrer at the speed of 2000 rpm. After adding the full amount of the solution, agitation was continued for about two minutes to obtain fine particles of the emulsion; the composition of the final W/O-type

mixture is presented in Table 1. The basic emulsion matrix is designated as Mark A (the oxidant is a mixture of ammonium and sodium nitrates). The final explosive mixtures were prepared by mixing the emulsion matrices with TNT at a temperature of 20-25 °C; in Table 1 these fortified mixtures are denoted as Mark B; for several specifications of detonation velocities the mixture B, containing Politol 2, was additionally sensitized by MBs to an amount of 0.2-3.0% by wt. (see data 8-15 in Table 3).

**Table 1.** Composition and density of the W/O-type emulsions studied

Mark	A	B	Note
Component	in % of weight		
AN	72.0	36.0	Emulsifier ANFOMUL (sorbitane monoisostearate); fuel – oil and mixture of paraffin and wax
SN	9.6	4.8	
CN	-	-	
Fuel	4.4	2.2	
Water	11.0	5.5	
Emulsifier	3.0	1.5	
TNT	-	50.0	
Density (g cm <sup>-3</sup> )	1.26	See in Table 3	

The TNT used was Politol, a product of the demilitarization process carried out by the Poličské Strojírny Co., which, in some cases, was separated into fractions by sifting (Table 2). Samples from two batches of this TNT were used, designated as Politol 1 and Politol 2. Detonation velocity of the original Politol was 5200 m s<sup>-1</sup> at charge density 1.1 g cm<sup>-3</sup>. For comparison, we also used TNT in flake form (military grade) obtained from a Russian manufacturer with detonation velocity 5800 m s<sup>-1</sup> at charge density 1.48 g cm<sup>-3</sup>.

**Table 2.** Fractions of TNT (Pitolol) according to the size of particle used for fortification of W/O mixtures

Fraction number	1	2	3	4	5
Particle size (µm)	< 100	< 200	100-200	200 - 400	400-800
Fraction number	6	7	8	9	10
Particle size (µm)	800-1600	1000-2000	1600-4000	> 4000	original mixture

### Measurement of velocity of detonation (VoD)

The prepared W/O-type emulsions were used to make cylindrical charges of 37 mm diameter (polypropylene artificial sausage skin). At least three optical sensors were placed in each charge. The measurement of VoD ( $D$ ) was performed by means of an EXPLOMET -2000<sup>®</sup> apparatus (producer Kontinitro AG). The results are summarized in Table 3.

### Relative explosive strength measurement

A ballistic mortar test was used for determination of the relative explosive strength of the samples studied, using “blasting gelatine” (BG) as reference [13, 14] (it contains 90.75% by wt. nitric acid esters, 8.05% by wt. nitrocellulose, 0.29% water, 0.51% by wt. calcium carbonate and 0.39% by wt. sodium tetraborate). A fixed amount of test explosive (10 g) was wrapped in polypropylene foil and inserted into the mortar enclosed by a steel projectile and then fired using a non-electric detonator (No. 8). The measurements are based on obtaining a calibration curve for the reference explosive at different masses, then the explosive strength of the tested explosive is expressed relative to the calibration curve of BG (% BG) [13]. For each measurement, a part of the non-electric detonator is inserted in the plastic sample and fired by match. Three measurements were made for each sample and the mean values are reported in Table 3.

### Thermal stability of the TNT used

A DTA 550 Ex apparatus was used for thermal analysis of the explosives [13]. The measurements were carried out at atmospheric pressure, with the test sample in direct contact with the air. The test sample (0.05 g) was placed in a test tube made of Simax glass, 5 mm in diameter and 50 mm long. The reference standard was 0.05 g aluminum oxide. A linear heating rate of 5 °C min<sup>-1</sup> was used. The results of these measurements were analysed using the software delivered with the DTA apparatus [11].

The thermal reactivity is expressed here as the slope (activation energy)  $E_a R^{-1}$  in the Kissinger relationship [12], which is used in a modified form for evaluation of results of non-isothermal differential thermal analysis.

$$\ln\left(\frac{\phi}{T^2}\right) = -\left(\frac{E_a}{R}\right) \cdot \frac{1}{T} + \ln\left(\frac{A \cdot R}{E_a}\right), \quad (1)$$

where  $\phi$  is the rate of temperature increase,  $T$  is the onset temperature of the exothermic decomposition (see Figure 1),  $E_a$  is the activation energy of the

reaction,  $R$  is the universal gas constant,  $A$  is a pre-exponential factor. The data required for calculations using eqn. 1 are presented in Table 4. The  $E_a R^{-1}$  values obtained, together with the corresponding  $r^2$  values (coefficients of determination), are shown in Figure 1.

**Table 3.** Detonation velocity ( $D$ ) and relative explosive strength ( $RS$ ) of the W/O-type emulsions fortified with TNT

Data No.	Grain size of TNT and/or information about charge	Charge density ( $\text{g cm}^{-3}$ )	$D$ ( $\text{m s}^{-1}$ )	RS (% BG)
1.1	<200 $\mu\text{m}$	1.45	5948	
1.2		1.45	a	86.5 <sup>b</sup>
2.1	200-400 $\mu\text{m}$	1.42	5887	89.0 <sup>c</sup>
2.2		1.41	5935	
2.3		1.41	a	84.2 <sup>b</sup>
3.1	400-800 $\mu\text{m}$	1.39	5727	
3.2		1.45	a	83.2 <sup>b</sup>
4.1	800-1600 $\mu\text{m}$	1.43	5697	83.2 <sup>c</sup>
4.2		1.47	5687	
4.3		1.45	a	82.3 <sup>b</sup>
5.1	1600-4000 $\mu\text{m}$	1.40	5452	85.5 <sup>c</sup>
6.1	>4000 $\mu\text{m}$	1.40	5549	85.5 <sup>c</sup>
6.2		1.48	5809	
6.3		1.47	a	84.0 <sup>b</sup>
7.1	Emulsion A from Table 1 + 3% MBs	1.13	5165	89.0
7.2		1.13	5157	89.0
8	Emulsion B <sup>d</sup> from Table 1 + 0.2% MBs	1.45	a	
9	+ 0.3% MBs	1.43	a	
10	+ 0.4% MBs	1.42	5554 <sup>e</sup>	
11	+ 0.5% MBs	1.41	5741 <sup>e</sup>	
12	+ 1.0% MBs	1.37	5851.5 <sup>e</sup>	
13	+ 1.5% MBs	1.30	5830 <sup>e</sup>	
14	+ 2.0% MBs	1.26	5488 <sup>e</sup>	
15	+ 3.0% MBs	1.17	4577 <sup>e</sup>	

Note: a) extinction of detonation;

b) fortified by Politol 2 (from the second batch) used for fortification;

c) fortified by Politol 1 (from the first batch) used for fortification;

d) fortified by Politol 2 having original grain size;

e) the averaged value of four measurements.

**Table 4.** Output from using DTA to specify the stability of the types of TNT used – data for use in Kissinger method of evaluation

Sample	Rate of temperature increase $\phi$ ( $^{\circ}\text{C min}^{-1}$ )	Peak temperature $T$ ( $^{\circ}\text{C}$ )
Politol 1	5	291.8
	10	301.2
	15	307.2
Politol 2	5	288.8
	10	304.2
	15	318.6
TNT flake	5	293.3
	10	308.5
	15	312.2

**The auto-ignition temperature**

This temperature was determined [13, 14] by heating a 100 mg sample of the given mixture at a heating rate of  $5\text{ }^{\circ}\text{C min}^{-1}$  until the point of auto-ignition of the sample was reached. The corresponding results are presented in Table 5.

**Table 5.** Results for the auto-ignition temperatures of the types of TNT used

Data No.	Auto-ignition temperature in $^{\circ}\text{C}$ for:		
	Politol 1	Politol 2	TNT flake
1	300	296	306
2	302	286	306
3	298	298	308
4	301	287	307
5	300	297	308

**Small-scale cook-off test**

The small-scale slow cook-off test setup was developed in our Institute [15]. The procedure follows UN standards [16] or STANAG 4491 [17]. The only change to the original procedure is a reduction in the sample dimensions. The steel tube (90 mm length, 21 mm in inner diameter, thickness of wall 2.7 mm, closed by steel caps at both ends – see Figure 6) was filled with approximately 40 g of the sample. The filled and insulated steel bomb was placed in the explosion chamber and held for five hours at a constant temperature of  $50\text{ }^{\circ}\text{C}$  below the assumed ignition temperature to obtain a homogeneous temperature profile. After

five hours, a heating rate of  $3.3\text{ }^{\circ}\text{C h}^{-1}$  was set until the ignition point. The bomb with the sample was heated directly with a heating wire.

Three measurements were obtained with mixture B from Table 1 and are shown in Table 6 – where  $T_1$ ,  $T_2$  and  $T_3$  are the temperatures on the particular thermocouples (they read the temperatures),  $T_i$  is the initiation temperature and  $\sigma$  is one standard deviation. Figure 7 shows a fragmented (“opened”) pipe after decomposition of its content.

**Table 6.** Results of small-scale cook-off test (temperatures in  $^{\circ}\text{C}$ )

$T_1$	$T_2$	$T_3$	Average of the three measurements	$T_i$	$\sigma$
164.3	163.4	163.4	163.7	<b>162.5</b>	1.23
161.0	160.9	161.1	161.0		
162.4	163.9	162.5	162.9		

### Calculation of Detonation Parameters

The calculation of theoretical detonation characteristics (detonation velocity,  $D$ , detonation pressure,  $P$ , heat of detonation,  $H_{\text{det}}$ ) for a W/O-type mixture with TNT but without sodium nitrate (SN) was carried out by means of the EXPLO5 code [18] (see in Table 7) – for detonation velocity determination, the mixture was sensitized by 1% by wt. of MBs; the composition of the W/O-type mixture and results of the calculations are presented in Table 7.

**Table 7.** Results of the detonation parameters of the fortified W/O mixture without SN calculated by means of EXPLO5

Component	Mass %	$H_{\text{det}}$ ( $\text{kJ kg}^{-1}$ )	$P$ (GPa)	$D_{\text{teor}}$ ( $\text{m s}^{-1}$ )	$D_{\text{exp}}$ ( $\text{m s}^{-1}$ )	$V_0$ ( $\text{dm}^3\text{kg}^{-1}$ )
$\text{NH}_4\text{NO}_3$	40.8	-4488.5	18.5	6894	6794	807.1
TNT	50.0					
Water	5.5					
Oil	3.7					
Density ( $\text{g cm}^{-3}$ )	1.36					

Further calculations were performed according to the European Standard [19] using the code **ZMWCyw** [20, 21]. Comparing the results from both these methods shows, in the case of mixtures from Table 6, that results for the thermal effect of detonation and the volume of gaseous products are relatively similar.

**Table 8.** Results of the detonation parameters calculated by means of the ZMWCyw code [20, 21]

Explosive	$Q_V$ (J/g)	$f$ (J/g)	$V_g$ (dm <sup>3</sup> kg <sup>-1</sup> )	$T_V$ (K)	$P_V$ (GPa)	CO/CO <sub>2</sub>	$\rho$ (g cm <sup>-3</sup> )
Mixture A from Table 1	3232.8	740.2	961.1	2102.7	6.042	0.0270	1.26
Mixture B from Table 1	4325.6	770.1	776.7	2707.3	8.107	0.0523	1.45
Mixture from Table 7	4267.1	802.6	815.2	2688.1	7.128	0.0814	1.36

where:

$Q_V$  – heat of explosion in a constant volume (a constant-volume explosion),

$f$  – specific force ( $f = n_g R T_V$ ),

$V_g$  – gas volume occupied by the explosion products in standard conditions,

$T_V$  – temperature for constant-volume explosion,

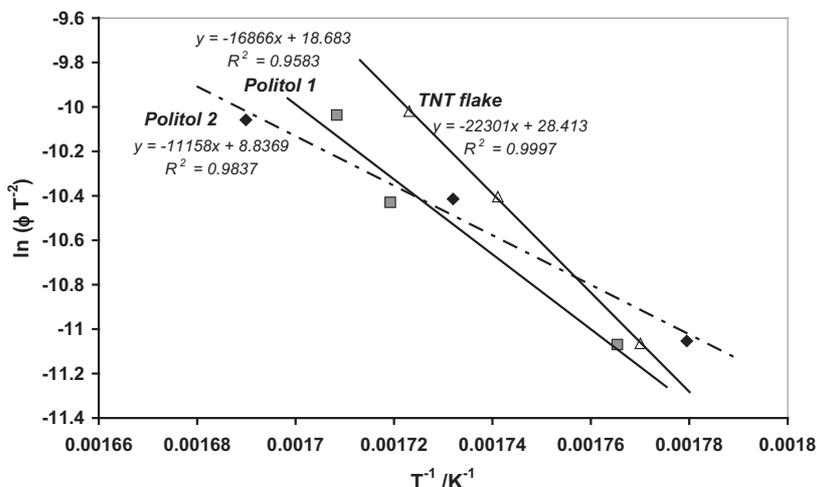
$P_V$  – pressure for constant-volume explosion,

CO/CO<sub>2</sub> – relation of moles of CO to that of CO<sub>2</sub> in the explosion products.

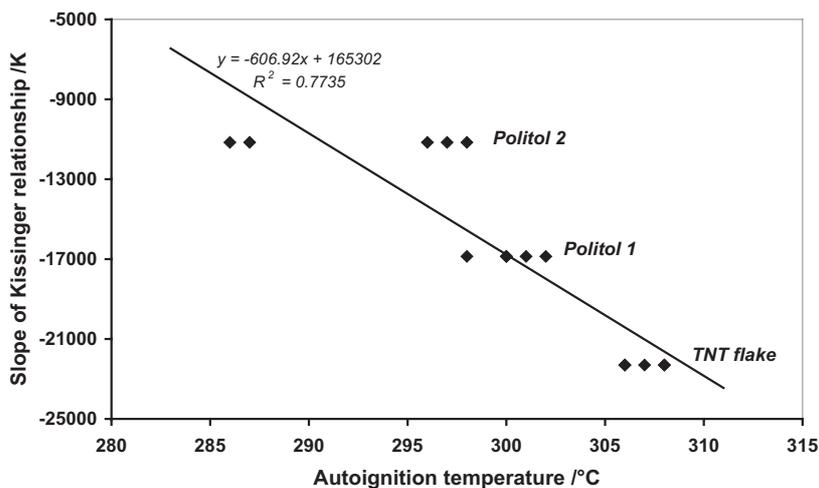
## Results and Discussion

Military high explosives after dismantling (demilitarization) suffer from a particular stability problem: there is a potential contamination with components from the protective coatings of the inner walls of the corresponding objects, particularly tar [22-24] and metals and/or corrosion products from the construction materials of these objects [25]. Incorporation of tar into re-melted TNT has a negative effect on its shock sensitivity and thermal stability [22, 24]. Therefore, it is clear that individual batches of dismantled TNT will differ, particularly in their thermal stabilities. This also applies to the Politol batches used in the current work. The evaluation of DTA outputs of the TNT samples (see Table 4) gave the results shown in Figure 1.

Figure 1 shows that the decomposition of Politol 1 is similar to that of TNT flakes, while the results for Politol 2 differ significantly from that of the previous two samples. This difference is also seen in the values of auto-ignition temperatures in Table 5 and Figure 2: here Politol 2 exhibits the lowest values of this temperature and the worst reproducibility of results.

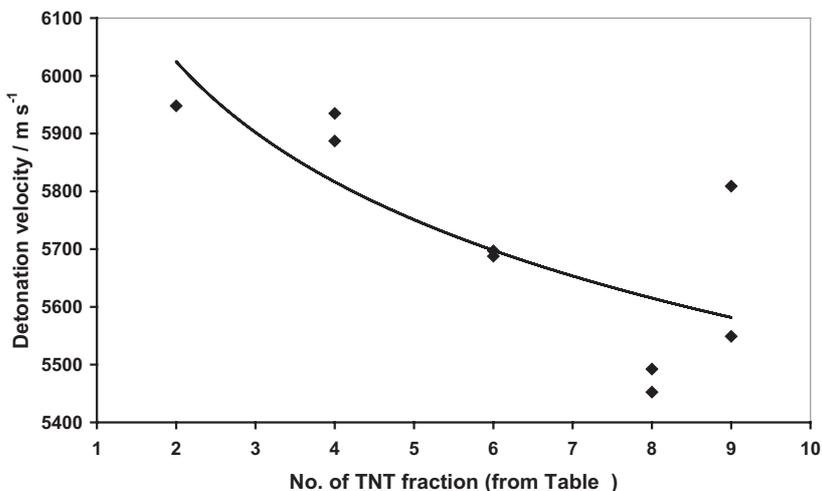


**Figure 1.** Evaluation of the DTA outputs by the Kissinger method; stability increases from right to left.



**Figure 2.** Relationship between plots of the Kissinger relationships (from Figure 1) and auto-ignition temperatures of the types of TNT used.

In classic slurry explosives, optimum fortification and sensitizing are achieved by addition of TNT having a coarser grain (2-4 mm) [26]. In the case of reversed emulsions (W/O-type mixtures) the opposite is true, as can be seen in Figure 3 (relationship between detonation velocities and TNT grain sizes).

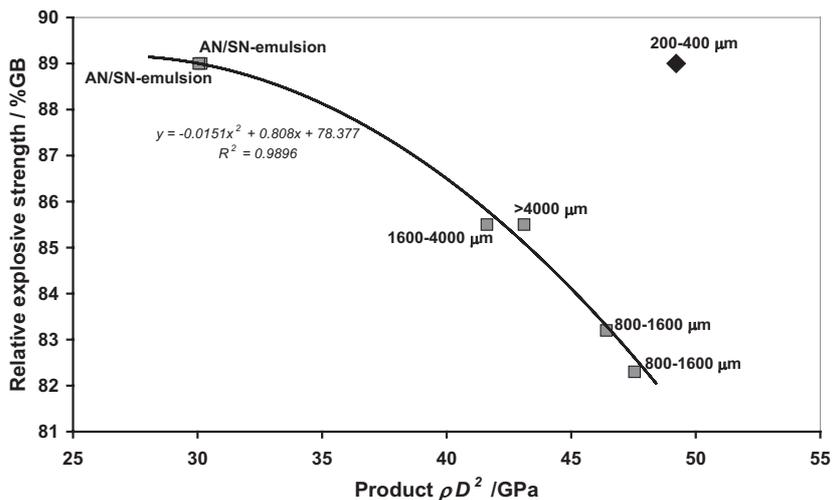


**Figure 3.** Relationship between the detonation velocity of the fortified W/O-type emulsions and the TNT fraction used and grain size of this TNT, expressed as a No. of fraction from Table 2.

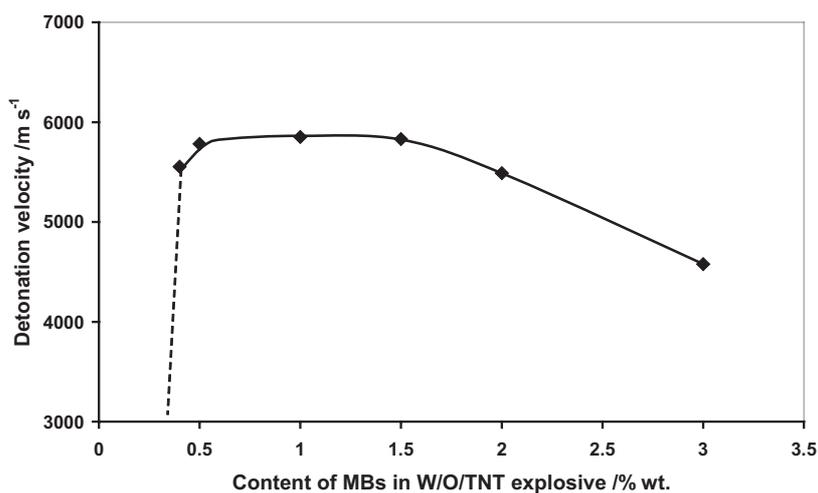
The addition of TNT to the matrix marked A (i.e. preparation of mixture B in Table 1) resulted in a density increase in the final explosive. Mixtures containing 50% by wt. of Politol 1 were possible to initiate in each case; however, in analogous mixtures with Politol 2, the detonation extinguished in all prepared charges. This shows that the detonation conditions of all the mixtures with TNT were far from ideal, which can also be seen in Figure 3, (relationship between detonation velocity and charge density).

In measurements of the relative explosive strength (RS) by ballistics mortar, the results obtained ought not to be affected by the density of the explosive and the degree of its homogeneity [27, 28]. However, the work done by the expansion of gaseous products of the explosive reaction in the mortar chamber is different, and decreases with increasing ignition delay [29] (i.e. with increase in the time which is needed for the reaction initiation in the mortar chamber). This might be the case for W/O emulsions fortified by Politol 1 with grain sizes below 400  $\mu\text{m}$  (see in Table 1 and partially from Figure 4) – here the explosive reaction might be faster in comparison with mixtures with higher grain sizes. However, it seems that for grain sizes over 1600  $\mu\text{m}$  there might be a further change to the higher RS values. Data in Table 1 also shows the increased RS values for mixtures with Politol 1 in comparison with those containing Politol 2. Figure 4 also shows the non-ideal nature of the explosives studied – here all fortified W/O mixtures, if they were close to being

ideal explosives, should have had a similar position in this Figure as data for a mixture fortified by Politol 1 with grain sizes 200-400  $\mu\text{m}$ .



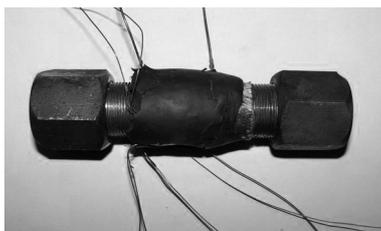
**Figure 4.** Relationship between the relative explosive strength and detonation pressure, expressed as a product of charge density, and the square of detonation velocity; numbers at points represent the grain size of the TNT used (Politol 1).



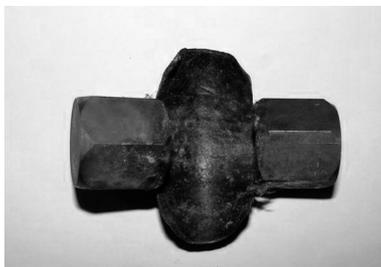
**Figure 5.** Relationship between detonation velocity and the content of MBs in W/O explosive fortified by TNT.

The facts mentioned above and the data in Table 3 demonstrate that demilitarized TNT is not a reliable sensitizer. Therefore, the use of TNT as a component of W/O explosives necessitates simultaneous addition of microballoons in order to make the final explosive more sensitive to initiation and propagation of detonation in a corresponding charge. From data 8-15 in Table 3 and Figure 5 it can be seen that optimal addition represents 1.0-1.5% by wt. of MBs; higher amounts lead to decreasing charge density and therefore also detonation velocity.

The problem mentioned above concerning reactivity of the W/O explosives (mixture B type) studied and the measurements using a ballistic mortar can also be seen from the results of small-scale cook-off test. As it follows from the previous chapter, the average initiation temperature of the W/O explosives containing TNT (mixture B from Table 1) studied was found by the small-scale slow cook-off test to be equal to 162.5 °C with a standard deviation of 1.2 °C. Because of a lack of homogeneity in the sample measured, the standard deviation is higher; but in spite of that, the results are sufficiently reproducible. The temperature mentioned agrees with the melting point of ammonium nitrate. The fragmentation of steel cylinders (bombs) in this test only consists in their opening (see Figure 7). In terms of the UN standard [16], the mixture B can be considered as EIDS – it only explodes or deflagrates under the conditions of the slow cook-off test.



**Figure 6.** The testing set-up of the small-scale cook-off test.



**Figure 7.** Fragment of the testing set-up (opened bomb) of the small-scale cook-off test after W/O mixture B testing.

As for the detonation characteristics calculated by means of EXPLO5 (ideal detonation), these are logically higher than those corresponding to reality. Comparison of results obtained from the calculations according to the European Standard [19-21] for mixture A, and its fortified version – mixture B (both from Table 1), with their RS values, shows that the relative similarity in the specific forces,  $f$ , of both these explosives corresponds to their almost identical RS values (see data 2.1, 7.1 and 7.2 in Table 3). That means that fortifying W/O emulsions only with TNT has no significantly better effect on the performance of the resulting explosives.

## Conclusion

The fortification and sensitizing of W/O-emulsion-type explosives by addition of demilitarized TNT could give some acceptable results if the grain size of TNT used does not exceed 400  $\mu\text{m}$  with a final proportion in the W/O explosive of 50% by wt. However, due to the diversity of demilitarized ammunitions it is impossible to achieve a precisely defined quality of demilitarized TNT; this fact causes initiation of the fortified W/O explosives where TNT may behave indifferently. That means that the using TNT necessitates simultaneous sensitizing of the final W/O explosive with microballoons in an amount of 1.0-1.5% by wt. Also, the relative explosive strength (RS) of the fortified W/O mixtures partly depends on the grain sizes of the TNT employed – also here the highest RS values correspond to explosives with TNT having grain sizes below 400  $\mu\text{m}$ . These maximum RS values are practically equivalent to the corresponding values of the original W/O matrix sensitized by addition of 3% by wt. of microballoons; in addition, the calculated specific forces of both these kinds of W/O explosives are similar. Thus the fortification of W/O emulsions by addition of TNT has no significantly better influence on the performance of the resulting explosives. The small-scale cook-off test of W/O mixture with 50% by wt. of TNT has shown that this explosive only deflagrates under the test conditions – this mixture is thus a relatively safe explosive.

## Acknowledgments

Authors are very indebted to Prof. Waldemar A. Trzciński from the Military University of Technology from Warsaw for disclosure of his ZMWCyw code for calculation of some detonation characteristics.

The work described in this paper was carried out under the auspices of the Ministry of Industry & Commerce of the Czech Republic as a part of its Research project No. FR-TI 2/639.

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