

Explosive Properties and Thermal Stability of Urea-Hydrogen Peroxide Adduct

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Abstract: The adduct of urea and hydrogen peroxide (UHP) is industrially produced material on a large scale. Although UHP is widely used as a bleaching and oxidizing agent, its properties as energetic material are generally overlooked. In this work we report comprehensive characterization of UHP explosive and thermal properties. We found that UHP is a compound with a negative value of standard enthalpy of formation ($-565.1 \text{ kJ mol}^{-1}$). It is not sensitive to impact and friction. However, we demonstrated that UHP ($\rho = 0.93 \text{ g cm}^{-3}$; packed into a steel pipe with inner diameter of

206 mm) detonates with experimental velocity of detonation (VOD) of 3780 m s^{-1} . Moreover, for UHP with maximal theoretical density ($\rho = 1.43 \text{ g cm}^{-3}$), the calculated VOD reaches 5219 m s^{-1} . Based on our findings, we recommend that present regulations regarding the handling, storage and transportation of the UHP should be revised, especially in cases, where UHP is kept on a large scale, under confinement and at places where the temperature can reach above 60°C .

Keywords: Urea hydrogen peroxide · Explosive properties · Thermal stability

1 Introduction

Chemical compounds containing both fuel and oxidizing functional groups in their molecular structure often belong to the family of hazardous energetic materials. The reaction between fuel and oxidizing parts of molecule can be very exothermic and can lead to explosion under certain conditions. For example, ammonium salts of oxidizing acids such as nitric, chloric and perchloric acids are well known representatives of this type of hazardous explosive materials [1–4]. Ammonium nitrate and perchlorate are stable chemical compounds, with very low sensitivity to mechanical stimuli, whereas ammonium chlorate is unstable and sensitive material that may undergo violent decomposition even at room temperature.

Certain uronium salts (salts of urea) of strongly-oxidizing acids are also known to be explosive. Although urea nitrate and urea perchlorate were suggested as components for industrial explosives, due to their problematic properties (such as high acidity, corrosivity, and chemical incompatibility) these urea salts have never been extensively used [1, 5].

Another related substance that contain urea and hydrogen peroxide is urea peroxohydrate (urea-hydrogen peroxide adduct or UHP). UHP is industrially produced on a scale of several hundred tons per year and is commonly used in the cosmetics and pharmaceutical industry, as a disinfecting and bleaching agent [6, 7]. For example, UHP is a stable and convenient source of hydrogen peroxide for tooth whitening [8–10]. Also, in the recent years, UHP became very popular and frequently used “green” oxidizing agent in organic synthesis [11–14]. It was utilized as a selective

and efficient oxidizing agent for oxidation of sulfides to sulfoxides [15–19], thiols to disulfides [20, 21], secondary alcohols to corresponding carbonyl compounds [22, 23], oxidant for epoxidation of olefins [24–28] and for many other oxidation reactions.

Despite the broad utilization of the UHP, as well as its storage and transportation on a large scale, only few reports were published regarding thermal stability of this compound. For example, pyrometry studies showed that UHP starts to decompose at 67°C , while manometric method revealed that the decomposition process takes place already above 55°C (at standard pressure) and even at 40°C (at a lower pressure of 2666.45 Pa) [29]. In contrast, report based on thermogravimetric analysis indicated that beginning of UHP decomposition was observed only at 74°C [30].

Material Safety Data Sheet (MSDS) of UHP classifies this material as an oxidizing and corrosive agent. Despite the

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fact that presence of both fuel (urea) and oxidizing components (hydrogen peroxide) in the UHP strongly indicates plausible explosive properties of this material, no experimental data regarding such properties were reported until now. In this work, we clearly exhibit that UHP has explosive properties. Even in its uncompressed form (as-manufactured crystalline form), without mixing it with any additional compounds, in a series of smaller and larger scale detonability tests, we discovered that UHP is detonable material. In addition, we determined UHP's sensitivity to impact, friction, and conducted meticulous and comprehensive thermal decomposition studies.

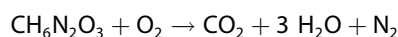
2 Experimental Section

2.1 Sensitivity Measurements

The drop hammer (Reichel and Partner) was used for determining impact sensitivity of compounds, utilizing a 10 kg drop hammer. Both cylinders (BFH-SR) and guide rings (BFH-SC) were produced by OZM Research. Sensitivity to impact tests included a series of 15 dropping trials at 100 J. Sensitivity to friction of compounds was determined by using a BAM (Bundesanstalt für Materialforschung und -prüfung) type friction apparatus FSKM-08 (OZM Research). Porcelain plates (BFST-Pt) and porcelain pegs (BFST-Pn) were produced by OZM Research. Sensitivity to friction tests included a series of 15 trials at 360 N.

2.2 Thermochemical Measurements and Calculations

A water calorimeter (static jacket) equipped with an oxygen bomb was used for the calorimetric measurements. Pressed UHP samples of approximately 2.0 g were loaded in open quartz crucibles and initiated with an electrically heated wire. The calorimeter was calibrated by combusting a benzoic acid reference standard (1.0 g) in an oxygen atmosphere at a pressure of 3.0 MPa. The reported value is the average of three single measurements. The enthalpy of formation was calculated at 298.15 K by using a Hess thermochemical cycle, with an assumption that the combustion products contain solely carbon dioxide, water, and nitrogen:



The CHEETAH code [31] was used to calculate the detonation parameters of UHP. The BKW EOS (Becker-Kistiakowski-Wilson equation of state) was assumed to characterize the physical properties of the gaseous products. The BKWC (CHEETAH's version of BKW EOS) set of parameters $\alpha = 0.5$, $\beta = 0.40$, $\kappa = 10.86$ and $\Theta = 5441$ [32], as well as the experimental densities and the value of the enthalpy of formation (ΔH_f) were obtained from the results of calorimetric experiments and were used in our calculations.

2.3 Detonability Measurements

Initially, the detonability of pressed UHP charges was tested. The charges of 25×100 mm (diameter \times length) or 36×100 mm were confined in copper tubes with wall thickness of 2.7 and 3.6 mm, respectively. They were fixed vertically on a 4 mm thick steel plate and their detonation was initiated with a 20 or 40 g booster (respectively) made of pressed HMX (octogen, 1,3,5,7-tetranitro-1,3,5,7-tetrazocane)/wax (95/5). The average density of aforementioned UHP charges was approximately 1.34 g cm^{-3} .

In the subsequent larger-scale tests, solid UHP was poured into large steel pipes. The material was used as-manufactured (at a bulk density of approximately 0.93 g cm^{-3}), without any pressing. The inner diameter of the first steel tube was 206.4 mm, the wall thickness 6.3 mm and the length 1 m. The inner diameter of the second tube was 124.0 mm, the wall thickness 8.0 mm, and the length 0.75 meter. The bottom of each tube was welded up with the steel disk with the thickness 10 mm. PETN (pentaerythritol tetranitrate) based plastic explosive PI Np10 (Explosia a.s.) was used as a booster. Boosters of 0.5 kg and 1 kg were used for the tubes of 124.0 and 206.4 mm, respectively.

The result of Velocity of Detonation (VOD) measurement is given as an average value from the eight individual results given by the individual probes located along each charge. VOD was measured by the optical method by using eight equidistant optical fibers. Each fiber was on the bare (sensing) end equipped by small air void, made by the thin aluminum foil, in order to ensure sufficient optical signal obtained from the passing detonation wave. Optical signals were processed by VOD 812 detonation velocity measuring system (OZM Research). Each charge was instrumented by eight optical channels. The distance between probes was 100 mm for the larger charge and 80 mm for the smaller one. Additionally each charge was instrumented by the continual VOD measurement Microtrap VOD/Data Recorder from MREL Group. The 1 m long HS type of probes were used for measuring. The scheme of the charges and probe location is shown at Figure 1.

2.4 Thermal Properties Measurements

The thermal decomposition of UHP was studied by several thermoanalytical methods, including Differential Scanning Calorimetry (DSC), Thermogravimetry (TG), and Accelerating Rate Calorimetry (ARC). DSC measurements were carried out with a Maia F3 (Netzsch, Germany). In a typical measurement, a sample of material (4.18 mg) was enclosed in an aluminum pan with pierced lid, while an empty pan was used as a reference. For comparison, a reference sample of urea (3.95 mg) was measured. The heating rate of 5 K min^{-1} in a dynamic nitrogen atmosphere (flow rate of 40 ml min^{-1}) was used in all DSC analyses. TGA measurements were carried out on the Tarsus F3 (Netzsch, Germa-

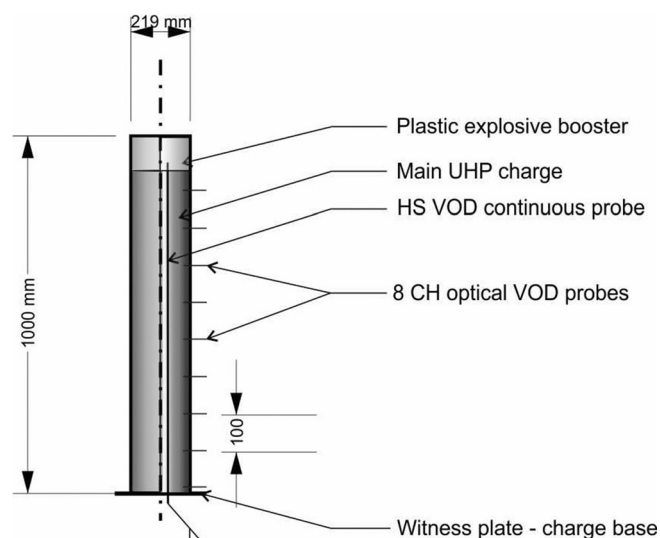


Figure 1. Scheme of the charges and VOD probes arrangement.

ny). In a typical measurement, a sample of material (4.06 mg) was placed into an alumina crucible. For comparison, a reference sample of urea (4.06 mg) was measured. The heating rate of $5\text{ }^{\circ}\text{Cmin}^{-1}$ under the dynamic nitrogen atmosphere (flow rate of 50 mlmin^{-1}) was used in all TGA analyses. ARC measurements were carried out on the Es-ARC (THT, UK). The standard heat-wait-search mode was utilized ($5\text{ }^{\circ}\text{C}$ per step, $0.02\text{ }^{\circ}\text{Cmin}^{-1}$ self-heating sensitivity). The hastelloy bomb (14 g) with internal volume of about 9 mL was filled with 200 mg of a sample and attached to pressure line of the calorimeter. Thermocouple was clamped to the outside bottom of the bomb. High Phi-factor was used to prevent the bomb fragmentation upon initiation of the sample.

3 Results and Discussion

3.1 Explosive Properties

In our experiments we confirmed that UHP is not sensitive compound to impact and friction (Table 1). We did not get any positive test (activation) of UHP by impact of fall hammer with energy 100 J and by friction force 360 N. For the comparison sensitivity of TNT to friction is lower than 360 N (which is the upper limit of BAM apparatus). The sensitivity to impact of TNT is higher than the sensitivity of UHP, as we obtained only one activation out of 10 trails with impact energy of 65 J [33].

Table 1. Physical properties of UHP.

	$\text{CH}_6\text{N}_2\text{O}_3$
Molecular mass [g mol^{-1}]	94.07
Oxygen balance [%]	-34.0
Heat of combustion [J g^{-1}]	7300 ± 70
Standard enthalpy of formation [kJ mol^{-1}]	-565.1

Despite of the observed and calculated thermodynamic stability, the oxygen content in the UHP is high enough to indicate that this molecule should have explosive properties. By using CHEETAH software, we calculated VOD to be above 3900 ms^{-1} for as-manufactured bulk UHP with density of 0.93 gcm^{-3} . For pressed UHP to the density of 1.43 gcm^{-3} , the calculated that VOD was significantly increased and was found to be higher than 5200 ms^{-1} (Table 2).

Table 2. Calculated detonation properties of UHP.

	Detonation parameters		
	Velocity VOD [ms^{-1}]	Pressure p_{CJ} [GPa]	Temperature T_{CJ} [K]
at 0.93 gcm^{-3}	3919	3.64	1628
at 1.43 gcm^{-3}	5219	7.95	1633

Following calculation studies, we conducted a series of detonation experiments with pressed and as-manufactured UHP. Photographs of pressed UHP charges with diameter of 36 mm, confined in a copper tube, witness plate and fragments of the tube collected after the detonability test, are presented in Figure 2.



Figure 2. Picture of the UHP charge (36×100 mm) with HMX booster, in a copper tube, prior to detonation (left); fragmentation of the copper tube after the detonation (middle) and stainless steel witness plate after the explosion of the charge (right).

For both confined charges of 25 and 36 mm in diameter, only fragments of copper tubes were recovered after tests, as well as the witness plates were dented (Figure 2). The produced dents were approximately 1 mm in depth and had diameters similar to that of the charges used. The pieces formed from the end part of the tubes (adjacent to the witness plate) were larger and had characteristic traces on the inner surface that can be interpreted as a symptom of explosion decay. These results evidently indicate that the UHP charges underwent explosive events, initiated by small HMX booster. It should be mentioned that under these experimental conditions, the detonation propagated with decreasing velocity, leading to formation only of a dent in a witness plate and not to the plate puncture. On the other hand, our results of the small scale detonability tests clearly

showed that much bigger and confined charges of UHP should be able to at least partial detonation.

Following the small scale detonation testing, we processed to the larger scale field experiments. The detonation velocities were calculated as slopes from the probe position-reaction time dependencies [34] (Figure 3). The results of VOD measurements of larger UHP charges that were confined in the thick-walled steel tubes are shown in Table 3.

Table 3. Summary of VOD results for larger scale UHP charges.

Charge number	Inner charge diameter [mm]	Density of UHP [g cm^{-3}]	VOD [m s^{-1}]
1	124	0.90	3517
2	124	0.93	3513
3	124	0.92	3236
4	206	0.93	3778

Good repeatability of VOD measurements strongly indicates a steady-state detonation of UHP under our experimental conditions. Any signs of lowering of VOD during the detonation cannot be seen in Figure 3.

Unfortunately, in our field tests of larger scale charges, the probes for the continuous VOD measurement did not provided any results. The possible explanation of these observations could be due relatively low detonation pressure (as indicated by our calculation results). The relatively low detonation order could also explain the steel tubes fragmentation in the form of elongated fragments and relatively small damage to the charge base (welded steel plate to the bottom of each tube), acting as the witness plate. The characteristic fragments from the both size of charges are shown at Figure 4.

Interestingly, we found that on many after-explosion fragments appeared characteristic parallel stretch marks that were located on the inner walls of steel tubes (Figure 5). Unfortunately, presently, the origin of these

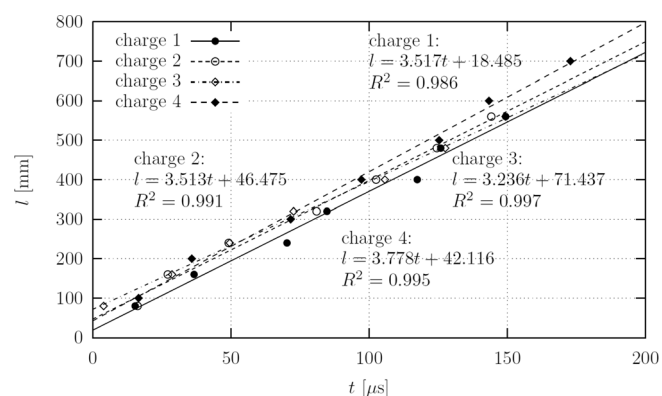


Figure 3. The detonation velocity of UHP. Positions of probes vs. their reaction time.

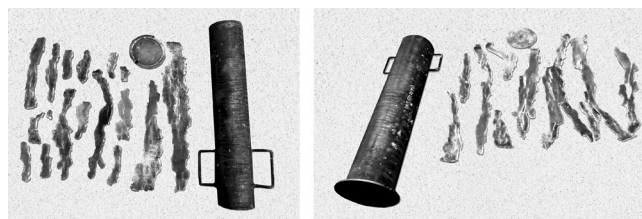


Figure 4. Fragmentation of a smaller steel tube after the explosion, identical tube is shown on a right side (inner diameter of 124 mm, the wall thickness of 8.0 mm and the length of 0.75 meter) (left). Fragmentation of a larger steel tube after the explosion, identical tube is shown on a left side (inner diameter of 206.4 mm, the wall thickness of 6.3 mm and the length of 1 m) (right).

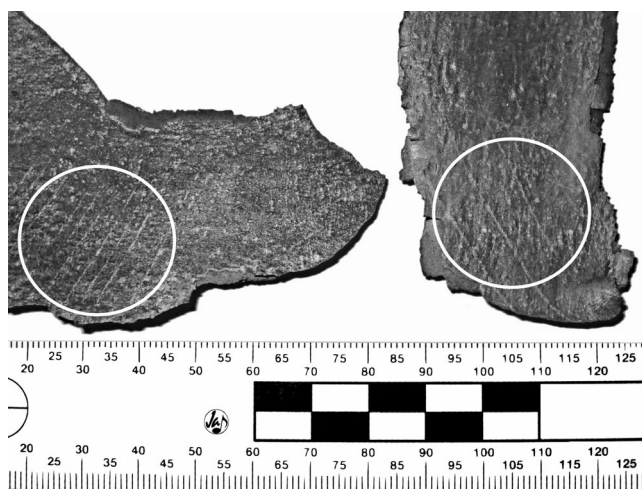


Figure 5. Steel tube fragments with characteristic stretch marks.

stretch marks is unclear and it will be a topic of further investigation.

3.2 Thermal Stability

The decomposition of UHP was studied by TG analysis and seems to take place in a four-step process (Figure 6). The first step (75–120 °C, with mass loss of about 38%) could be assigned to a release of hydrogen peroxide (theoretical mass loss of 32.5%) from UHP. It is possible that during the latter process, some urea is also evaporated or decomposed. The next three steps of the decomposition process correspond to the decomposition of urea via biuret and cyanuric acid [35–37], as could be seen in the corresponding mass-loss thermogram of the pure urea.

Further studies were focused on DSC and ARC analyses. The DSC thermograms of UHP and reference pure urea are shown in Figure 7. Two endothermic peaks in the UHP thermogram could be attributed to the release of hydrogen peroxide and melting of the compound, while the third strong exothermic peak is indicating the decomposition of UHP at below 100 °C. In a very good correlation with the

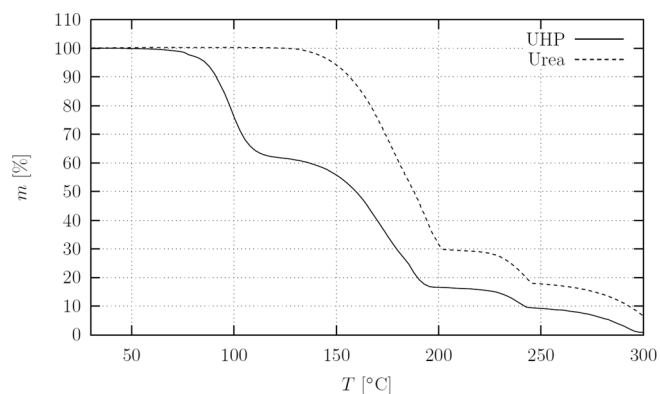


Figure 6. TGA thermograms of UHP (solid curve) and reference sample of pure urea (dashed curve).

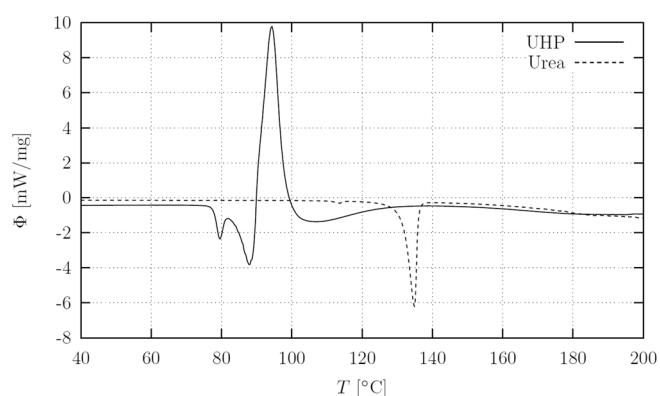


Figure 7. DSC thermograms of UHP (solid curve) and reference sample of pure urea (dashed curve).

TGA results, above temperature of UHP decomposition, no additional peaks could be detected in the DSC thermogram, strongly supporting the conclusion that most of UHP was consumed during the exothermic process. In contrast, pure urea undergoes melting 132 °C and according to TG analysis a significant non-exothermic mass loss begins in urea only above 150 °C.

The sample of UHP was also subjected for ARC analysis. The exotherm was observed already at 70 °C (Figure 8, solid curve), from the pressure curve (Figure 8, dashed curve) and it was possible to detect that the decomposition took place already at 60 °C. The temperature and pressure built-up rates are high, corresponding to the ignition of the sample.

4 Conclusions

The explosive properties and thermal stability of the urea hydrogen peroxide adduct (UHP) were studied in a series of laboratory and field tests. The following conclusions could be made:

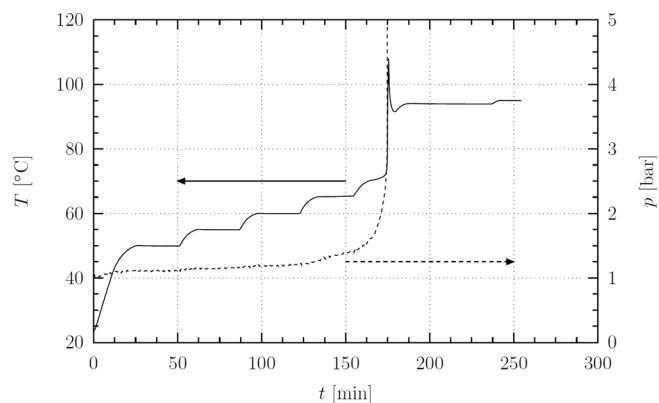


Figure 8. ARC thermogram of UHP (solid curve corresponds to temperature, dashed curve corresponds to pressure).

(i) UHP is practically a non-sensitive material to impact and friction, with sensitivity lower than of TNT.

(ii) Although a UHP is a non-ideal explosive, it can detonate in higher diameter vessels, under the strong confinement and using of a strong booster.

(iii) UHP cannot be regarded as very stable substance. Based on our observations, UHP decomposition begins already at 60 °C and with respect of enclosing, the decomposition reaction may turn to be violent and uncontrollable.

Based on our results, UHP should be considered as hazardous detonable substance with relatively low thermal stability. Therefore, we strongly recommend that special safety attention should be paid during large-scale transportation and storage of the UHP under confinement, especially when the temperature in the container could reach above 60 °C.

Acknowledgments

Dr. Qi Long Yan is acknowledged for his help with preparation of the manuscript.

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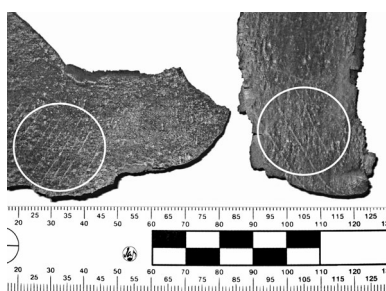
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Received: April 25, 2016

Revised: July 18, 2016

Published online: ■ ■ ■, 0000

FULL PAPERS



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