

Spontaneous explosion of ammonium nitrate in a contact with an active chlorine-containing organic substance

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Abstract

Results of a complex investigation into behavior and consequences of chemical reactions between ammonium nitrate and an organic substance, disinfectant, sodium dichloroizocyanurate which is incompatible with ammonium nitrate are presented. This substance being dissolved in water releases active chlorine which easily enters into chemical reaction with ammonium nitrate. Interaction between ammonium ion and active chlorinated chemicals triggers a chain of chemical reactions at normal temperature, resulting in heating, formation of explosive gas NCl_3 and foaming. Chemical reactions controlling the interaction proceed at a rather low energy of activation, and their conversion level is restricted by attaining the temperature at which the released gas NCl_3 explodes (115°C). Mostly, only local explosions happen without involving the total mass of the mixture into the chemical conversion. If there are no conditions providing accumulation of explosive gas in rather large amount, its detonation cannot initiate ammonium nitrate. It seems that the buildup of explosive wave can only be due to the foam which contains solution of active reaction products and has the higher propensity to initiating stimuli in comparison to the pure ammonium nitrate.

Keywords : ammonium nitrate, sodium dichloroizocyanurate, spontaneous explosion

1. Introduction

The most destructive accidental explosions taking place in course of manufacturing and handling of explosive materials turn out to be connected with ammonium nitrate^{1,2)}. One can notice, however, an obvious contradiction between statistic of explosions which involved ammonium nitrate (AN) and its explosive properties determined with use of standard tests³⁾. Pure AN does not burn under normal conditions and has very low susceptibility to common mechanical and thermal stimuli. There was no detonation observed in AN, except if it has been initiated by direct action of detonation of other high explosive, and there was no well-grounded scenario proposed to connect explosion picture with conditions and

properties of AN, even for the most famous explosions happened in Oppau and Texas-Sity¹⁾.

The causes of accidental explosions having involved large masses of AN seem to be concerned to an unexpected change of its usual behavior. This change can be due to various chemical or physical factors, in particular, to presence of active additives⁴⁾. There are materials which can strongly reduce chemical stability of AN being in contact with it⁴⁾. Some of these materials (mineral acids, sulfur, sulfides and several active chlorinated compounds) can initiate self-supporting chemical interaction with AN even under room temperature. These reactions have high exothermic effect and produce a real hazard of spontaneous explosion. The

rules regulating safety during handling of AN require eliminating any contact with these substances.

Catastrophic explosion of a warehouse with 300t off-grade ammonium nitrate at AZF plant at Toulouse, France took place on September, 21, 2001. The TNT equivalent was estimated 30–40t, 30 people died, ~3000 injured, 600 buildings were destroyed, the losses were estimated to cost 2.1 billion Euro. The key idea connects this explosion with sodium dichloroizocyanurate (DCCNa, chemical formula is $C_3N_3O_3Cl_2Na$) which is a granulated disinfectant of the new generation. Some amount of this chemical seems to be brought during regular unloading together with defective portion of AN stored in the entrance section of the warehouse. Really, DCCNa makes part of the group of chemicals recognized to be incompatible with AN due to the hazard of a spontaneous explosion. Contact at the AZF plant between DCCNa and AN could happen due to a negligence, that was a direct violation of the plant instructions. Before the Toulouse accident, there were no investigations into explosion in this pair and of other dangerous compositions. After explosion, several papers have been published^{(5)–(7)} considering different aspects of explosive behavior.

The purpose of this work is to get the detailed information on the behavior and consequences of interaction between AN and DCCNa, which releases active chlorine in aqua solution. Results of investigation obtained shed light on mechanisms of interaction and spontaneous explosion and can be used to estimate hazard of contacts of AN with domestic chemicals.

2. Object and methods of investigation

French-fabricated industrial AN at loose-packed density of $\sim 700 \text{ kg}\cdot\text{m}^{-3}$, consisting of porous spherical granules 1–2 mm in average diameter, and DCCNa as a polydisperse powder with a mean particle size of $\sim 0.8 \text{ mm}$ have been studied. Chemicals were used as supplied, or sieved after crushing into fractions with particles 0.63–1.25 mm and 0.25–0.63 mm in size. Before firings the chemicals were carefully dried up. Mixture was prepared at the mass fraction 1 : 1, that approximately corresponds to stoichiometric composition. In addition, the layered samples were also studied in order to diminish explosion hazard of kinetics tests. In this case the chemicals were placed one over another by layers of identical thickness varied depending on the sample mass and sizes of the test vessel. Wetting the samples was implemented by different ways as follows. In the first method, water was injected directly into the ready dry sample using either a syringe or a special injector. Another method consists of keeping the separate chemicals, before the sample preparation, in moist air, and the amount of moisture absorbed by the sample was determined by the sample mass increment. The most part of procedures especially related to wetting the samples have been carried out remotely.

A number of techniques, common or specially designed, were used in this study. Kinetics of chemical interaction was analyzed with a help of thermogravimetric (ATV-17 M), and calorimetric (DAK-1-1) devices. Reaction products

were determined with a use of mass-spectrometry (MKh-1201-V with the energy of ionizing electrons 50 eV and the accelerating voltage 5 kV), and release of explosive gases was simply visualized. The shock sensitivity was monitored by using the interface deceleration technique with electromagnetic device⁽⁸⁾. Explosion of unconfined charges (up to 25 kg in mass) was studied visually and with a help of thermocouples. Explosion and detonation properties of confined charges were studied using steel and plastic tubes equipped with photo, piezo- and contact gauges to monitor the wave propagation. Besides, to clarify physical details of explosion, numerical modeling has been involved.

3. Investigation results

3.1 Kinetics of chemical interaction

When the mixed samples (up to 300 mg in mass prepared of carefully dried components) were kept under the temperature ranging from the room temperature up to 64°C, no visible traces of exothermic interaction were observed, at least, during several hours. However, both components are hygroscopic materials, and at a normal state they can contain 0.2–2% moisture. Behavior of the small mass samples with this moisture does not differ from that of dry samples. Under wet conditions the picture drastically changes. Samples exposed to air of 100% humidity absorb moisture rather rapidly. For instance, at 35°C, the mass of 30 mg sample of AN increases by 25 % in 3–4 h, and the sample of DCCNa does the same in 16–17 h. Visual observations and video filming show that wetting the samples results in release of gases, formation of a foam, change of the sample color and marked increase of the volume occupied by the sample. Observations of dynamics of sample heating with a help of thermocouples shows that it depends on a few factors, including the contact surface area of components, the sample mass, initial temperature, wetting way and amount of the injected water. In the case of the layered samples, the temperature increase due to the interaction does not exceed 25°C. In the case of mixed samples of small mass (2–5 g), injection of water in amount more than 5 wt. % results in the 40–70°C temperature increment. For the mixed 20 g and heavier samples, an injection of water causes the quick rise of temperature (during 30–70 s) up to 100°C. Thereafter an explosion happens resulting in a destruction of the cup containing the sample and a discharge of contains of the cup into the atmosphere.

The calorimetric study under isothermal conditions performed with the wetted 250 mg layer samples placed into the long glass unsealed ampoules has shown that the heat release has a sharp rate peak at the first minutes after preparation of the wetted sample. Thereafter the heat release rate drops rapidly. Experimental data on the maximum heat release rate, in spite of the marked scatter, demonstrate evident effects of the humidity and initial temperature. The highest values of the heat release rate are observed at 3–5% of the water contents. With increase of initial temperature in a range from 20°C to 74°C, the heat release rate grows following the Arrhenius plot with

a low activation energy (30–65 kJ·mol⁻¹) which is typically inherent in exchange or oxidation-reduction reactions.

Video filming of the process developing in the wetted mixture was conducted with chemicals of the 0.25–0.63 mm particle fraction. Samples ~2 g in mass were placed into a Plexiglas cup 10 mm in diameter. The dry mixture is easily ignited by electrically heating wire placed at the top of the sample, and burns in a flameless combustion mode with the average rate ~0.6 mm·s⁻¹, releasing a white smoke and leaving (after combustion) a grayish-white porous skeleton. Wetting of the sample by injecting 0.2–0.3 g of water initiates an intensive generation of foam (the volume occupied with the foamed material increases by 4–5 times). Expansion of the foam continues during 5–10 sec with an average rate 5 mm·s⁻¹.

Foaming of the wetted mixture evidently occurs due to the gas release. Placing the electric wire at a distance 30–50 mm above the top level of the expanded foam and switching on the voltage, induces an explosion in gas which produces the subsequent explosion in the foam and a series of the weaker local bursts at the cup bottom. The cup often remained intact after the test. However, in a few tests an explosion of the foam appeared to be much more powerful: it caused the destruction of the cup and left a dent in the steel plate supporting the cup, at the level where the position of the interface between the gas and foam was at the explosion instant, according to the video filming. In order to determine temperature at which explosion in the gas is initiated, we have carried out the same tests with the elevated electric wire to which the thermocouple junction was glued. These tests have shown that the gas released in the damped mixture is not ignited until the temperature of the wire reaches 115°C. At higher temperatures the gas decomposes with explosion.

The chemical mechanism of the interaction considered is very complicated and here we make only a few remarks concerning its key stages. In aqua solution, AN partially dissociates producing ammonium cation and anion of nitric acid, and hydrolysis of DCCNa produces a whole “bouquet” of active chlorinated substances. They include hypochlorides, chlorine, Cl₂O, etc. The following, most probable, step is interaction of chlorinated substances with ammonium ion, generating, as intermediate reaction products, a set of chloramines: NH₂Cl, NHCl₂ and nitrogen trichloride NCl₃. It is well known⁹ that chloramines are extremely active substances: mono-chloramine explodes at -40°C, and nitrogen trichloride which can be extracted as a liquid or vapor (at room temperature) decomposes with explosion following the branching chain mechanism at the temperatures over 100°C. Exothermic decomposition of chloramines and generation of gaseous NCl₃, which can accumulate while its temperature does not exceed 100°C, seems to be a rational interpretation of observations considered above.

In order to confirm this mechanism, we have implemented additional tests in which AN was replaced by the powdery ammonium chloride. Aqua solution of ammonium chloride contains ammonium cation, but, obviously, no nitric acid anion. There was no exothermic

reaction observed in the dry 1 : 1 mixture of DCCNa with ammonium chloride up to 190°C. However, the same mixture being wetted demonstrates intense reaction accompanied with foaming and heating immediately after introducing water. This response looks similar to the behavior of the moistened AN + DCCNa samples studied above. Finally, the other series of tests has been conducted, when AN was replaced by sodium nitrate to exclude the presence of ammonium cation, but save nitric acid anion. In this case there was no interaction observed, both in dry and wetted mixtures. Thereby, reactions between ammonium ions and active chlorine triggering spontaneous explosions in the pair of AN + DCCNa can take place also in some other pairs of chemicals resulting in almost the same consequences and making them potentially dangerous.

3.2 Shock sensitivity and detonability of dry AN + DCCNa mixture

Only dry mixture of components as supplied was studied in this series of tests. Shock sensitivity was studied with use of the contact boundary deceleration technique⁸. The experimental assembly comprises the booster charge 80 mm in diameter that generates a plane shock wave, the plate with an air gap 10–15 mm wide, the paraffin disc 15–20 mm thick, and the charge of studied sample confined in a plastic casing 60 mm in diameter and 20 mm thick, with an electromagnetic gauge to monitor the contact-surface velocity. The intensity of the magnetic field was 340 Oe. The booster charge was fabricated of TNT or TNT/talc mixture, its composition and density were varied to provide the desired shock wave amplitude. The shock wave propagating through the plate with air gap and paraffin disc generates a rectangular profile at the paraffin/sample interface during time interval of ~4–5 μs. The electro-magnetic gauge monitored both the velocity of this interface and the wave velocity in the sample. Tests were conducted in a test field. If the sample tested does not react, the recorded signal looks like a horizontal line. In a reacting sample the contact-surface velocity reduces due to an exothermic reaction that releases gaseous products and raises the pressure. It results in a decaying contact-surface velocity profile. The earlier the declination from a horizontal line starts the faster is the exothermic reaction.

The shock wave 0.6 GPa in amplitude (the value determined in the paraffin disc) produces only weak traces of reaction in the mixture of AN with DCCNa. This amplitude seems to correspond to the threshold of initiation of a self-propagating reaction. The 1 GPa shock or stronger in amplitude triggers the marked exothermic reaction. The 1.5 GPa shock or stronger induces a violent decomposition of the samples with a delay not exceeding 1.5–2 μs. The same tests with the pure AN were used for comparison. It follows that the AN + DCCNa mixture is slightly less sensitive than pure AN. This ex-fact unexpected result can be explained supposing that under these conditions there is no chemical interaction between AN and DCCNa.

The detonation velocity of the mixture and pure AN (for

comparison) was measured using electric contact gauges in steel and plastic tubes of various diameters. The length of tubes was at least 7 tube diameters. An explosive mixture (AN with TNT of 79/21 in wt. %) of loose-packed density detonating at velocity $5\text{ km}\cdot\text{s}^{-1}$ was used as the booster. The booster charge diameter was not less than diameter of the tested sample. The tests have shown that the mixture confined in steel 80 and 146mm diameter tubes (5 and 7mm in wall thickness, accordingly) detonates steadily at velocity of 2.6 and $3.1\text{ km}\cdot\text{s}^{-1}$. In plastic 152mm diameter tubes the mixture also demonstrates the steady detonation at velocity of $2.1\text{ km}\cdot\text{s}^{-1}$. For comparison, pure AN confined in the same steel 146-mm diameter tubes detonates steadily at velocity $2.9\text{ km}\cdot\text{s}^{-1}$, which is $200\text{ m}\cdot\text{s}^{-1}$ smaller than the aforementioned detonation velocity in the mixture. However, the tests performed in the 80mm diameter steel tubes demonstrate a significant scatter in shock front velocity: the detonation either failed or its velocity leveled at the end of the sample at the velocity $\sim 1.3\text{ km}\cdot\text{s}^{-1}$. The pure AN behaved in the same way in plastic tubes. Namely, no detonation was observed in the 152mm diameter tubes and the process at the velocity $\sim 1.3\text{ km}\cdot\text{s}^{-1}$ at the end of the sample was observed in the plastic 190 mm tube.

Thus, the tests conducted with a dry mixture in steel (strong) and plastic (soft) casings and comparison with the similar tests with pure AN shows that under detonation conditions the interaction of mixture components has enough time to manifest itself resulting in higher detonation velocity. Nevertheless, the effect of interaction is rather moderate, and in the plastic 152mm diameter tube the same booster fails to initiate detonation both in the mixture and in pure AN.

3.3 Explosion and detonation of the wetted mixture in confinement

The field tests were implemented with the wetted mixture samples up to 25 kg in mass placed into the 35L plastic tanks. Mixing was carried out by the jet technique: the jets of components flowing out from separate containers have entered the rotating mixer simultaneously with the sprayed water jet. In all tests wetting the mixture resulted in spontaneous exothermic reactions and heating. Consequences of these reactions differed from test to test without any regular trend, covering the range from a weakly developing process which begins as a pop and ends with an ejection of the white smoke cloud with the subsequent fusion of chemicals, and up to the vigorous explosion resulting in destruction of device, crushing of the plastic container walls into the fine fragments and the crater formation. There were no any indications just before explosion, except of temperature which often increased up to 100°C or slightly higher. However, there was no valid detonation in this case. Indeed, (i) a large mass of non-reacted material was dispersed by an explosion and covered the ground within a 25m radius around the crater center, and (ii) the explosion had just a moderate blasting effect.

The evolution of the explosion has been considered in a few tests in steel or transparent plastic tubes. The mixture of 300 g in mass was prepared from the dry granulated components; water in amount of 10 or 20 g was introduced by injector through the closed bottom end of the tube. Plastic tubes were 35mm in internal diameter with 12mm walls and 260mm long. Steel 32mm diameter tubes with 7mm walls and 460mm long had a few small orifices for the photo recording. In order to intensify luminosity of reacting products flowing out of the orifices they were filled with fine grained PETN. The tubes were set up in vertical position. All the tests manifested explosion after the water injection with a delay 30–70 s.

In the case of plastic tubes the bright luminous process related to the chemical interaction arises at a point being away $\sim 110\text{ mm}$ from the tube bottom and propagates forwards and backwards along the charge. The wave propagating forwards, to the open tube end, is a decaying low velocity detonation. Its initial velocity is $\sim 1100\text{ m}\cdot\text{s}^{-1}$, thereafter the velocity reduces, luminosity drops, mainly, due to destruction of casing. In the case of steel tubes low velocity detonation arises at a distance of $\sim 140\text{ mm}$ from the tube bottom and propagates steadily along the charge at a velocity of $\sim 1500\text{ m}\cdot\text{s}^{-1}$. Pressure in this wave recorded in a special test with using the Kistler-type piezoquartz gauge is 0.5–0.8 GPa. Detonation has resulted in destruction of the casing into coarse elongated fragments specific of low velocity detonation in solids.

So, generation of the explosive gas and formation of the active foam are two factors which make the interaction between AN and DCCNa really hazardous. But the role of explosive gas should not be overestimated. At least, we can conclude that the explosion of this gas, most likely, does not initiate decomposition of pure AN under normal conditions. This conclusion is confirmed by the results of two parallel tests performed with the mixture of ground chemicals (fraction 0.25–0.63 mm). The charges 70 mm long were placed in steel tubes 15 mm i.d. and 250 mm long. Four orifices were made along the tube for the pressure gauges. The charge burning was initiated by igniter-cap mounted at the closed tube end. Difference between two tests was only that in one test the dry mixture was used, and in the other test a dry mixture was wetted by the water injection 30s before initiation of the mixture by the cap. During this 30s interval the interaction of wetted chemicals produces physical and chemical transformations drawing almost to self-ignition. The purpose of these tests was to study the effect of these transformations under conditions when due to the rather short length of the charge and possibility of generated gas to escape through the open tube end this explosive gas cannot accumulate in significant mass inside the reacting mixture.

As is seen from Figure 1, in the case of the wetted charge the wave with very steep front is formed already at an initial phase of the process (see pressure record at the distance 40 mm). Parameters of this wave are evidently related to explosive decomposition of the active gas released. The peak pressure value is twice as much as

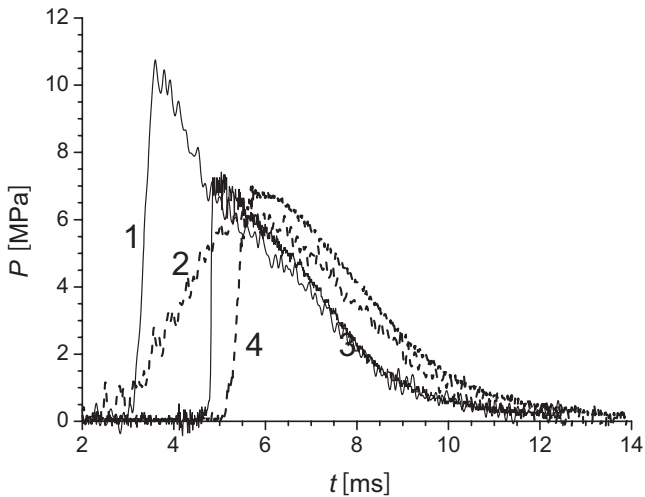


Figure 1 Pressure-time profiles recorded at two points along the charge at a distance 40 and 145 mm from the closed charge end in two tests with the dry (curves 2 and 4) and wetted (curves 1 and 3) mixtures initiated by the igniter-cap.

the maximum pressure recorded at this distance in the case of dry mixture. However, at the distance of 145 mm the difference vanishes, and the waves developed in two testes, become almost identical concerning both the front steepness and maximum pressure. Thereby, explosion of active gas, released in the wetted mixture in the considered test, has no active support from reactions in the condensed phase.

3.4 Numerical simulation

To shed light on this situation, we have involved numerical modeling. The computer code used here for simulation was developed earlier in quasi-1D approximation to analyze the burning-to-explosion transition in porous solid materials¹⁰. Initial model follows approach based on two-phase reactive mechanics and presented in¹¹. The process developing in the form of a burning wave is initiated and supported by hot gas combustion products which, being filtered through the charge pores, heat up and ignite outer surface of the material grains. Burning of grains produces hot gases and heat which, in turn, cause the pressure rise and burning wave acceleration.

The code has been modified to include the generation and explosion of active gas observed at wetting of the AN + DCCNa mixture. Considered is a charge of grained explosive material 800 mm in length, confined in the closed cylinder casing. The charge porosity and grain size reproduce properties of the AN prills + DCCNa mixture studied above. The grains being heated up to 800K start burning with the preset burning rate, generating the mixture burning products and combustion heat. The behavior caused by wetting the AN + DCCNa mixture is simulated in the charge section 200mm long where at the initial time instant the chemical reactions suppose to start on the grain surface generating the active gas and heat release. Active gas simulates properties of NCl_3 , which, according to thermodynamic estimations, detonates at

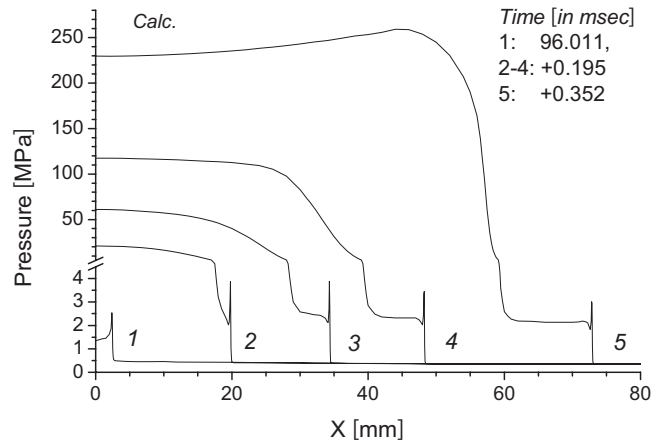


Figure 2 Computation of explosion development in the wetted AN+DCCNa mixture with reduced ignition temperature of the mixture. Evolution in time of the pressure profile.

normal pressure with the velocity $1450 \text{ m}\cdot\text{s}^{-1}$, the detonation pressure and temperature are 4MPa and 2500 K, respectively. The active gas released can partially accumulate in the 200mm “wetted” charge section providing the pressure rise and partially filtrate through the pores into the “non-wetted” charge section. Due to the heat release the temperature of the grain surface and gas monotonously increases. If temperature approaches 420K, the active gas starts reacting with a heat release following the Arrhenius reaction with the activation energy of $418 \text{ kJ}\cdot\text{mol}^{-1}$. We cannot enter here into details on input data of the model but only note that almost all of them including rates of release of active gas and heat were selected based upon the measurements described above.

The calculation results are as follows. By the instant when the temperature has reached 420K at a point in the “wetted” charge section, pressure of the gas filling the pores has attained 0.35 MPa. Approximately 16 ms later, a detonation-like pressure wave builds up in the gas. This wave propagates along the wetted charge section at the velocity $\sim 870 \text{ m}\cdot\text{s}^{-1}$ and along the other charge section at the velocity $710 \text{ m}\cdot\text{s}^{-1}$ with the peak pressure ranging between 3–4 MPa. However, transit of the wave produces only the 120–140K increment of the grain surface temperature which turns out to be insufficient to initiate burning of AN.

Thus, one can conclude that the active gas, being accumulated (to some degree) in the pores and exploded, does not involve AN grains into the chemical reaction. A real alternative, perhaps, is that the foam which contains a solution of active reaction products has the higher susceptibility to initiation as compared with AN. To simulate this alternative, the ignition temperature of the grained material is reduced from 800K to 550K. This has resulted in a drastic change of event shown in Figure 2: the wave of gas explosion, propagating along the charge with the same parameters as in the previous case, has caused ignition of the bed and followed (at some distance behind the primary front of the wave) by a vigorous pressure wave which amplitude quickly increased up to 250 MPa. Therefore, only combined effect of the active gas

and the elevated reactivity of the foam could trigger an explosion in the total mass of explosive.

4. Conclusion

Thus, this investigation has clearly manifested that the interaction inside the wetted mixture of AN and the organic disinfectant, DCCNa, which presents a couple of incompatible chemicals, considerably changes the explosion behavior of this mixture in comparison with normal explosive materials. The main conclusions can be stated as follows.

The interaction between ammonium ion and active chlorinated chemicals produced in the course of dissolution of AN and DCCNa in water at normal temperature, triggers a chain of chemical reactions resulting in a heating, a formation of explosive gas NCl_3 and a foam formation.

Chemical reactions which control the interaction rate have rather low activation energy, and conversion level is restricted by attaining the temperature at which the released gas NCl_3 explodes (115°C). Under the most conditions, there are only local explosions observed which can not involve total mass of the mixture into the explosive conversion.

If there are no conditions providing an accumulation of the explosive gas in rather large amount, its detonation can not involve AN into the chemical conversion. Only the foam which contains solution of active reaction products and seems to have the higher propensity to initiating stimuli in comparison to the pure AN can provide the buildup of explosive wave.

The experiments in which ammonium nitrate was

replaced by ammonium chloride have demonstrated that the reactions between ammonium ions and active chlorine triggering spontaneous explosion in the couple of ammonium nitrate with DCCNa can take place also in some other couples of substances resulting in almost the same consequences and making them potentially dangerous.

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