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Under Construction-2  
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Distillation and Crystallization  
Recent Additions, Updates  
Compositions  
Less Energetic Basic Compounds  
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## Nitro Alkanes

NitroAlkanes are, in general, insensitive, and quite energetic when detonated. However, the secondary nitro compounds, whether the two nitro groups are on the same carbon or adjoining carbons, are thermally unstable. 2,2-dinitro propane has a melting point of 51.5degC, and is thermally unstable when warmed. At 75degC, it loses a 2/3rds of its weight after two days. It should be used soon after it is made, or be kept cold. 1,2-dinitroethane is described as being fairly reactive, but I am unsure as to how it is. Explosive Effects and Applications By Jonas A. Zukas

### Trinitromethane

Trinitromethane, while still giving off mildly poisonous fumes, is far less toxic than tetranitromethane. You would be well advised to avoid ever preparing tetranitromethane, which was once considered for use as a chemical weapon. (should it be desired to have the compound synthesized despite this, tetranitromethane is produced in high yield by the treatment of acetic anhydride with concentrated nitric acid)

A mixture of nitromethane and NaOH will form the salt of nitromethane, sodium 'nitromethanate'. Bubble in nitrogen dioxide into a solution of this salt, and trinitromethane can be obtained, because the intermediate aci- form of nitromethane, which is vulnerable to oxidation, is formed. The aci-form is probably CH2=NO2H. Alternatively, bubble mixed nitric oxides into a solution of sodium nitrite and nitromethane (which is sparingly soluble in water), then bubble in only nitrogen dioxide. Sodium nitrate and trinitromethane will form in solution.

Trinitromethane can form salts with a base, such as methylamine or ammonia, or it can dissolve in hydrocarbons. It can be mixed with either nitro methane or nitro ethane, to form a powerful explosive binary mixture. The one with EtNO2 is somewhat more powerful, but it also requires a higher ratio of HC(NO2)3, so if nitromethane is easily available in large quantities it may be preferable. (the 'Et' means the --CH2CH3 group)

Salts of trinitromethane are more stable (and somewhat less energetic) than perchlorates, although trinitromethane itself is fairly sensitive. The C(NO2)3(-) anion is very stable because of the resonances, there being four additional electrons to resonate around the six oxygen atoms. These salts are also known as nitroformates and are bright yellow in color. Ammonium nitroformate is much more stable than the sodium salt. Silver nitroformate slowly decomposes at room temperature.

Trinitromethane melts at 15°C, and begins slowly decomposing above 25°C

At room temperature nitroform is a colorless, or slightly yellow, oily liquid.

Moderately toxic by inhalation. Irritating to skin, eyes, and mucous membranes. When heated can cause headache and nausea. Causes mild narcosis. A very dangerous explosion hazard; explodes when inflated rapidly. Dissolution is exothermic and solutions of more than 50% can explode. Mixtures of 90% trinitromethane + 10% isopropyl alcohol in polyethylene bottles have exploded. Frozen mixtures with 2-propanol (10%) explode when thawed. Can explode during distillation. Mixtures with divinyl ketone can explode at 4°C. The boiling point is 175.1 °C so at least it is much less volatile than tetranitromethane.

**Trinitromethane is somewhat poisonous:** 800 mg/m<sup>3</sup>/2 hour Inhalation Mouse LC50. compare this with 1230ppm (for 36min, LC50) for tetranitromethane.

It can be seen that **tetranitromethane is far more toxic (it is highly recommended that the synthesis of this compound never be attempted)**, but nitroform should still be assumed to give off toxic vapor, and so should be handled outdoors or behind a windowed fumehood.

Trinitromethane oxidizes ferrous ions (Fe+2) to ferric (Fe+3). Dinitromethane can also oxidize the Fe+2 ion, but only if an alkaline solution of dinitromethane is added to an acidified solution of a ferrous salt. This is because the transient "aci-form" tautomer O2NCH=NO2H, which only exists for a few seconds, is more reactive.

### Trinitropropane

#### 1,2,2 trinitro propane

**Route 1:**

Nitrogen dioxide can be prepared far more easily than concentrated nitric acid. Nitrogen dioxide reacts with excess acetone to create NitroAcetone (and H2O and nitric oxide). This is because acetone is a ketone and there is a double bond between carbons in equilibrium. The double bond makes one of the hydrogen atoms come off easier. For example, acetone reacts with bromine at room temperature. This reaction is somewhat slow, unless the pH is lowered, but in this case that would ruin the reaction. First the NO2 has to stop being bubbled in. Then a 20% HCl solution is added. This causes the NitroAcetone to hydrolyze with water to hydroxylamine chloride and pyruvic acid. For example, NH2OH is made commercially from nitromethane and sulfuric acid, formic acid being the byproduct in that case.

Hydroxylamine will condense with more NitroAcetone to create an oxime =NOH. Ammonia, by contrast, only transiently forms an imine =NH so there is only a small condensation product in equilibrium. The industrial production of hydrazine sometimes uses acetone to form imines with the hydrazine product, to prevent further oxidation, improving yields.

4. CH3C(NOH)CH3 reacts with NO2 to make CH3C(NO2)2CH3. First a pseudonitrosole is formed (a nitro and nitroso on the same carbon), the nitroso can then be oxidized to form another nitro group. It is commonly known that gem-dinitro compounds are easily derived from oximes. As a side note, pseudonitrosoles [such as (CH3)2C(NO)(NO2)] tend to have a **green to blue** color when in alkaline solution, whereas nitro acids [such as CH3C(NO)(NO2)] tend to have a **red** color in alkaline solution.

5. a nitration on acetone cannot be performed with nitric acid. A nitro group is, in fact, initially added to the acetone, but since HNO3 is so acidic, the hydroxylamine is immediately formed from the nitro group (see point #2) and hydroxylamine, a good reducing agent, quickly gets oxidized. This is why the pH cannot be allowed to go too low when performing the nitration with NO2, otherwise NH2OH will form and NO2 will oxidize it. NaNO2 buffer might help. (the nitration of acetone can be used to produce salts of nitroic acid CH3C(=NOH)NO2 )

many of the reactions used are rather esoteric and not commonly known of. And I would not blame anyone for thinking these reactions are too bizarre to work. I do not know which particular reaction in the synthesis is in doubt.

1. Acetone does indeed react with nitrogen dioxide

Nitrogen dioxide on an alkene will form the nitroalkene. Of course the gas does not react (unless combusted) with an alkane such as heptane.

CH3C(O)CH3 has an equilibrium with CH2C(OH)CH3. The latter begins to dominate as the pH is lowered, that is basic textbook stuff.

2. Nitromethane reacts with a hot concentrated HCl solution, in what is known as a Meyer reaction.

CH3NO2 + H2O + HCl -> HCOOH + NH3(OH)Cl.

(Hexamine reacts with a concentrated HCl solution in a slightly different disproportionation reaction. Alternatively NH4Cl will react with (2) equivalents of CH2O, to form CH3NH3Cl, and HCOOH.)

3. NH2OH does indeed condense with a ketone to form an oxime =NOH. Ammonia, by contrast, only transiently forms an imine =NH so there is only a small condensation product in equilibrium. The industrial production of hydrazine sometimes uses acetone to form imines with the hydrazine product, to prevent further oxidation, improving yields.

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The trinitrate ester of glycerin might appear to be an ideal explosive, but in fact there are many others more powerful that simple oxygen balance. Ketoximes (R2C=NOH, where R is a hydrocarbon) react with NO2 (or N2O4) to form 2-nitro-2-nitroso alkane; To get 2,2-dinitro the nitroso must be oxidized the nitroso, which may be accomplished with dilute H2O2.

**Route 2:**

A different way to make 1,2,2-trinitro propane is mentioned in the literature. Russian researchers say that NitroEthylene and dinitroEthane react rapidly at 60degC with a tiny bit of sodium acetate present to form 1,2,2 trinitropropane. A mistake may have been made in the research paper or I may have misread, since it would seem this reaction would instead produce 1,3,3-trinitrobutane. Perhaps using dinitromethane instead of dinitroethane would form the desired 1,3,3-trinitropropane.

7.3g CH2=CHNO2 was dripped over 30min into 48g of CH3CH(NO2)2 in 35mL methanol. The temperature rose from 20 to 35C. Left overnight at 50C. Diluted with H2O, oil separated out. Watery solution treated with ether. Ether extract and oil combined again. Diluted with water again, then saturated with NaCl. (presumably to salt out the trinitropropane)

*"Addition of 1,1-dinitroethane to 1-nitro-1-alkenes"*

*Journal Russian Chemical Bulletin*

*S. S. Novikova, I. S. Korsakova1 and K. K. Babievskii*

*N. D. Zelinskii Institute of Organic Chemistry Academy of Sciences USSR, USSR*

**Route 3:**

A third way to prepare 1,2,3-trinitropropane may be a direct addition reaction between 2-nitro propylene with nitrogen dioxide.

Credit to "Rhodium" for the picture



Credit to "Rhodium" for the picture

### 2-Nitropropylene CH2=C(NO2)CH3

**Preparation**

*Step 1:*

75.1g Nitroethane, 0.3g calcium hydroxide and 80g 40% formaldehyde solution was dissolved in 75ml ethanol with stirring and was allowed to stand for 48h at room temperature. Distillation at 100-105°C/13 mmHg (85-86°C/6 mmHg, a 99°C/10 mmHg) gave 48g 2-nitropropanol (46%) and 14.3g of 2-nitro-2-methyl- 1,3-propanediol, the latter remaining as a crystalline residue in the distillation flask after distillation of the 2-nitropropanol.

*Step2:*

105 grams of 2-nitropropanol (1 mol) was placed in a 250ml two-necked RB flask equipped with a reflux condenser and an addition funnel, and 110g of acetic anhydride (1.078 mol) was added dropwise with good stirring. The solution was then refluxed for 30 minutes, cooled and poured into 200ml cold water, the organic phase separated and washed with 100ml water, and then vacuum distilled (bp ca 100°C/10 mmHg, yield 90%). The acetate is a clear water-white liquid with a faint slightly mustardlike odor, almost insoluble in H2O.

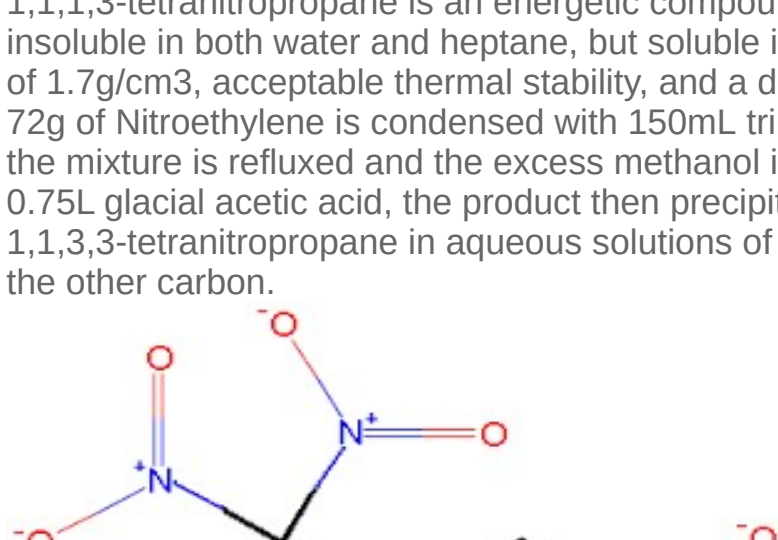
*Step3:*

Sodium carbonate (0.25 mol, 14.5g) is added to a solution of 2-nitropropyl acetate (0.5 mol, 73.5g) dissolved in 50ml benzene and refluxed in a RB flask for six hours. The flask is cooled and the product decanted from the solids. The residue (consisting of sodium acetate) is extracted with 50 ml benzene, dissolved in 100ml water, and the solution extracted with 3x25ml benzene. The pooled organic extracts are dried over anhydrous MgSO4, and the mixture fractionally distilled to afford 2-nitropropene (bp 57°C at 100 mmHg). It should be noted that 2-Nitro propylene has high toxicity, similar to phosgene, when vaporized in the air, so distillations of this compound are best avoided.

### 1,1,1,3-tetranitropropane

1.1.1.3-tetranitropropane is an energetic compound with slight plasticity and a melting point around 50 degC. The solid is insoluble in both water and heptane, but soluble in toluene, methanol, acetic acid, and especially acetone. It has a density of 1.7g/cm<sup>3</sup>, acceptable thermal stability, and a detonation velocity around 6.75 km/sec.

72g of Nitroethylene is condensed with 150mL trinitromethane, using 300mL methanol as a solvent at 0degC. Next the mixture is refluxed and the excess methanol is then distilled off with reduced pressure. The residue is dissolved in 0.75L glacial acetic acid, the product then precipitates after addition of water. 1,1,1,3-tetranitropropane turns into 1,1,3,3-tetranitropropane in aqueous solutions of alkali or ammonium hydroxide, one of the nitro groups rearranges onto the other carbon.



**Dinitropropanes that do not have a hydrogen atom on the same carbon as the dinitro group require a higher temperature for thermal decomposition than those that have such a hydrogen. P. S. DeCarli, D.S. Ross, Robert Shaw, E. L. Lee, H. D.Stromberg**

### mono-Nitro, di-Nitro, and tri-Nitro Ethane

#### 1,1-Dinitroethane

To a stirred solution of 2.66 g (66.5 mmoles) of sodium hydroxide in 15 ml of water at 20° was added 5.0 g (66.5 mmoles) of nitroethane. When all the nitroethane dissolved, the solution was cooled to 5°-7° in an ice-water bath.

The sodium salt of nitroethane was prepared as above. To the stirred solution was added a solution of 20 g (288 mmoles) of sodium nitrite in 50 ml of water, followed by a solution of 4.4 g (13.4 mmole) of potassium ferricyanide in 25 ml of water. Finally, 16.0 g of solid sodium persulfate (67 mmoles) was added all at once. The reaction temperature, moderated by an ice-water cooling bath, increased to 50°. The orange mixture was stirred at 25° for 1 hour and then cooled to 10°. Urea, 20 g (0.33 mole), was added, followed by 10 ml of glacial acetic acid. The mixture was extracted with three 25 ml portions of ether and the combined extracts were washed with brine and dried. The crude product was distilled to give 4.2 g (52% yield) of 1,1-dinitroethane, b.p. 87°-89° (16 mm), identified by its nmr spectrum.

32.5 grams of ethyl bromide (0.3 moles) was poured into a stirred solution of 600ml dimethylformamide and 36 grams dry NaNO2 (0.52 mole) in a beaker standing in a water bath keeping the solution at room temperature as the reaction is slightly exothermic. Always keep the solution out of direct sunlight. The stirring was continued for six hours. After that, the reaction mixture was poured into a 2500 ml beaker or flask, containing 1500 ml ice-water and 100 ml of petroleum ether. The petroleum ether layer was poured off and saved, and the aqueous phase was extracted four more times with 100 ml of petroleum ether each, whereafter the organic extracts were pooled, and in turn was washed with 4x75ml of water. The remaining organic phase was dried over magnesium sulfate, filtered, and the petroleum ether was removed by distillation under reduced pressure on a water bath, which temperature was allowed to slowly rise to about 65°C. The residue, consisting of crude nitroethane was distilled under ordinary pressure (preferably with a small distillation column) to give 60% of product, boiling at 114-116°C.

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CH3CH2-NO2 + CH3CH2-ONO -> CH3CH(NO)NO2 + EtOH. KNO2 can also be used instead of NaNO2. If NaNO2 is used in DMF, 30g of urea can also be added as nitrite scavenger to minimize side reactions, as well as simultaneously increasing the solubility of the NaNO2 and thereby significantly speeding up the reaction.

If the ethyl bromide is substituted with ethyl iodide, the required reaction time is decreased to only 2.5 h instead of 6 h. In case ethyl iodide is employed, a slight change in the above procedure needs to be done. The pooled pet ether extracts should be washed with 2x75ml 10% sodium thiosulfate, followed by 2 additions of 75ml water, instead of four 75ml water as above, to remove trace leftover I2.

**Alternate Nitro Ethane Synthesis**

1.5 mole sodium nitrite (103.5g) is intimately mixed with 1 mole of sodium ethyl sulfate (158g) and 0.0625 moles of K2CO3 (8.6g). The mixture is then heated to 125-130°C, at which temperature the nitroethane distills over as formed. Heating is discontinued when the distillation flow slackens considerably, and the crude nitroethane is washed with an equal amount of water, dried over CaCl2. The nitroethane is then re-distilled, collecting the fraction between 114-116°C. Yield 46%

A third way to prepare nitroethane is from ethyl nitrite.

#### Conversion of Ethyl Nitrite into Nitroethane.

The solution of the nitrite in paraffin oil was placed in a flask provided with an upright spiral condenser, which in turn was connected with a flled with asbestos-wool (in order to expose a large surface to the gaseous nitrites), and heated in an asbestos box provided with a thermometer. The gaseous nitrite was carried into the tube by means of a slow stream of dry carbon dioxide. The nitro-compound was collected in a U-tube immersed in cold water, and the escaping nitrite in absolute alcohol or methyl alcohol cooled in ice-water. Fifteen or 20 c.c. of the nitrite solution were used at a time. A 25% solution is convenient, and a 50 per cent. solution loses nitrite very rapidly. As the gaseous nitrite remains only momentarily in contact with the heated asbestos, the yield of the nitro-compound is small, the greater portion of the nitrite escaping conversion. Within four or five hours the greater part of the dissolved nitrite passes through the heated tube. The oily nitro-compound was easily recognised by the formation of the sodium compound on the addition of alcoholic sodium hydroxide solution. The nitro-compound was also reduced to the corresponding amine, which was converted into the platinichloride.

The conversion commenced at 100°C, and was fairly rapid at 120-130°C, when oily drops began to collect in the empty U-tube. At the end of four to five hours about 1 c.c. of the liquid collected, which responded very readily to the alcoholic sodium hydroxide test. If the temperature is raised to about 135-150°C, acetaldehyde and acetic acid are produced along with the nitro-compound. At 150-180°C the acid is the main product of the decomposition. The formation of the aldehyde and the acid is readily explained by the fact, fact that at a higher temperature the nitrite dissociates into alcohol and nitrous acid, the latter decomposing into nitric acid, and the higher oxides of nitrogen which oxidise the alcohol to the aldehyde and the acid. The best yield of the nitro-compound is obtained at 120-130°C.

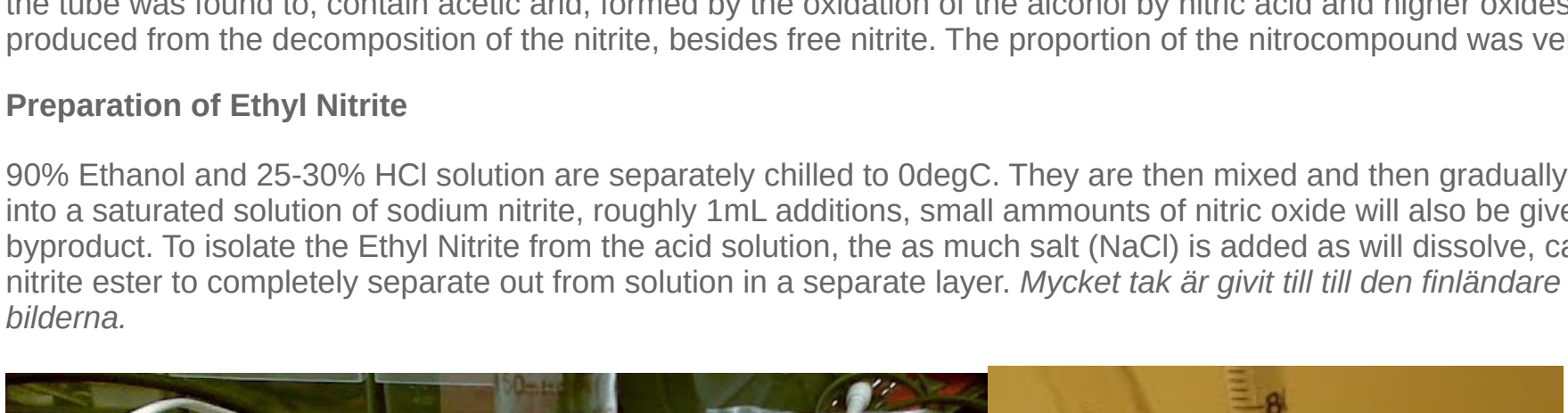
*Effect of Water and Alcohol on the Reaction*

The presence of moisture, which is always present in the rapidly dried nitrites, facilitates the conversion of the nitrite into the nitro-compound. In order to ascertain if the presence of larger quantities of moisture in the gaseous nitrites gives rise to a larger percentage of nitro-compounds, the nitrites were passed through a wash-bottle containing water before entering the heated tube. It was found that when saturated with moisture the nitrite dissociated more rapidly, yielding larger quantities of alcohols, aldehydes and acids without any appreciable increase in the yield of the nitro-compound. It appears that in the presence of traces of moisture, two reactions proceed simultaneously, the conversion of the nitrite into nitro-compound and the dissociation of the nitrite; and with the increase in the proportion of water the latter reaction proceeds with greater rapidity.

In order to determine if a better yield is obtained when an alcoholic solution of the nitrite is heated in a sealed tube, 10 cc of the solution were sealed in a glass tube and heated over flame. During the act of sealing, the part of the tube containing the solution should be immersed in a freezing mixture, otherwise explosions take place. The yield, however, was not very satisfactory. When heated at 100°C for five to six hours the solution, after distilling off the unchanged nitrite, and alcohol, gave about 1 cc of liquid, which positively tested for a nitro-paraffin, and which boiled at 98-104°C. Because of the mixture with alcohol, the actual boiling point was lowered. If, however, the solution was heated to 150°C for twelve to fifteen hours, the tube was found to, contain acetic acid, formed by the oxidation of the alcohol by nitric acid and higher oxides of nitrogen produced from the decomposition of the nitrite, besides free nitrite. The proportion of the nitrocompound was very small.

#### Preparation of Ethyl Nitrite

90% Ethanol and 25-30% HCl solution are separately filled with 0degC. They are then mixed and then gradually pipetted to into a saturated solution of sodium nitrite, roughly 1mL additions, small amounts of nitric oxide will also be given off as a byproduct. To isolate the Ethyl Nitrite from the acid solution, the as much salt (NaCl) is added as will dissolve, causing the nitrite ester to completely separate out from solution in a separate layer. *Mycket tak är givet till den finländare "HME" för bilderna.*



nitrit, sulfursä acid och isopropanol, reaktionen kanna också bli igång med HCl och etanol

nitritum

### 1,1,1-trinitro ethane

An idea for making 1,1,1-trinitro ethane.

NO2 is known to react with an oxime (R2C=NOH) to make R2C(NO)(NO2). Possibly NO2 would react with ethyl nitroformate CH3C(NOH)NO2 to make CH3C(NO)(NO2)2, which then might react with dilute H2O2 to give the final product of CH3C(NO2)3. This would be very powerful, but I am unsure how stable it would be. It might require a booster charge to detonate or it might explode if thrown to the ground.

Also I found something on 1911 Encyclopaedia Britannica:

"The silver salt (silver nitroformate), obtained by shaking an ethereal solution of nitroform with freshly prepared, moderately moist silver oxide, reacts with methyl iodide to form trinitroethane, a crystalline solid which melts at 56° C. Concentrated caustic potash decomposes the latter compound, forming the potassium salt of dinitroethane,

CH3 C(NO2)2K"

quoting another post.

"US patent 5043488: slow addition of nitric acid to acetone produces an unknown explosive"; probably ethylnitrolic acid (1-Nitro-1-oximinioethane). CH3C(NOH)NO2

"was prepared by mixing acetone with nitric acid (of 24% concentration) and a little fuming HNO3 and allowing the mixture to stand for 8 days at room temperature. An ether extraction gave on evaporation some acetylmethylnitrolic acid Beil 3,621 and R. Behrend & H. Tryller, Ann263,221- 3j (1894)

"...> right after the strong oxidation left behind was a clear, thick yellow oil of a pungent odor. According to Jahresbericht über Fortschritte der Chemie (1902), Behrend and Tryller, p. 1075-77 this oil contained one-third to half acetylmethylnitrolic acid (CH3.CO.C(NOH).NO2) (cryst., mp. 62 deg., very decomposable, and under other products, including pyruvic and oxalic acids). They say in the same instance methylethyl ketone affords CH3COOH, HCOOH, and considerable amounts of ethylnitrolic acid and dinitroethane.

There is a slight chance that using methyl ethyl ketone, which is easily obtained as a paint solvent, might also make, in very low yield, some CH3CH2C(NOH)NO2, which could then be used to make 1,1,1,3-trinitropropane.

### Trinitropentane

**Picric acid can be reduced with borohydride (using ether and alcohol solvents) to form 1,3,5-trinitro pentane, which should be as thermally stable as dinitroethane, unlike many other poly-nitro alkanes. M. Adam and T. Severin, Chem. Ber. Volume 96, p448. (1963)**

### Hexanitroethane

Hexanitroethane is a colorless crystalline solid which melts at 147 degC, with significant decomposition. It is not as hazardous to handle as tetranitromethane because of its low volatility. Hexanitroethane has a vapor pressure of 0.8 millibar at 30C. However, the toxicity between the two compounds is otherwise similar for a given concentration in air.

Hexanitroethane can give more powerful mixtures with nitroaromatics than tetranitromethane because of its higher density. Hexanitroethane is not thermally stable. At 70 degC, the half-life is about 400 hours. The decomposition rate is 10-20 times faster if dissolved in a hydrocarbon solvent. The measured density for Hexanitroethane is 1.85g/cm<sup>3</sup>.

For more information on hexanitroethane, see "Fulminate and Nitrolate".

### Comments