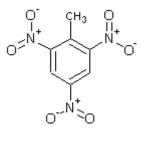
CHAPTER 11: THE PREPARATION OF NITRO BENZENES

TNT, F-TNB, TCTNB, PICRYL CHLORIDE, TNBCI

11-01. TNT. 2,4,6-trinitrotoluene; trinitrotoluene



TNT

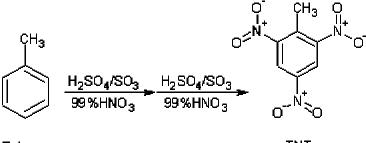
TNT is a white, slightly yellow, or yellow crystalline solid with a melting point of 80 Celsius. TNT is insoluble in water, but soluble in acetone, and benzene. It is widely used in explosives either by itself, or mixed with other explosives (Composition B). TNT is the highest manufactured high explosive in the world. It is widely used in grenades, bombs, missiles, demolition charges, and a variety of explosive compositions. It requires a blasting cap or detonator for initiation (usually initiated by RDX blasting caps or detonators). *Detonating velocity: 6930 meters per second. A secondary explosive.*

Procedure 11-01A: Preparation of TNT

Materials:	1. 50 grams 99% nitric acid
	2. 858 grams of 30% fuming sulfuric acid
	3. 34 grams of toluene
	4. 108 grams of 70% sulfuric acid

Summary: TNT can be prepared by reacting toluene with fuming sulfuric acid and 99% nitric acid. Afterwards, the reaction mixture is filtered to recover the TNT crystals. The TNT crystals are then washed with water, and dried. The dried TNT is then purified by mixing it with 70% sulfuric acid. After which, the acidic mixture is then filtered to collect the TNT. The TNT is then washed, and then vacuum dried, or air-dried. Commercial & Industrial Note: Part or parts of this laboratory process may be protected by international, and/or commercial/industrial processes. Before using this process to legally manufacture the mentioned explosive, with intent to sell, consult any protected commercial or industrial processes related to, similar to, or additional to, the process discussed in this procedure. This process may be used to legally prepare the mentioned explosive for laboratory, educational, or research purposes.

Hazards: Warning! 99% Nitric acid is a highly poisonous and corrosive liquid, which evolves highly poisonous fumes of nitrogen oxides. Wear gloves and use proper ventilation when handling 99% nitric acid and 30% fuming sulfuric acid. 30% Fuming sulfuric acid is a highly toxic and corrosive liquid, which evolves toxic and corrosive fumes of sulfur trioxide.



Toluene

Reaction Equation

Procedure: Place 50 milliliters of 99% nitric acid into a flask, and then carefully add 280 grams of 30% fuming sulfuric acid. Next, place the flask containing the acids into an ice/salt bath and cool to -5 Celsius. When the acid mixture reaches a temperature of about -5 Celsius, slowly add drop-wise, 34 grams of toluene over a period of 100 minutes while rapidly stirring the acid mixture and maintaining its temperature at -5 Celsius. After the addition of the toluene, continue stirring the reaction mixture and maintaining its temperature at -5 Celsius for 30 additional minutes. After 30 minutes, remove the ice/salt bath and allow the reaction mixture to warm to room temperature. Then place the reaction mixture into an ice bath and cool to 0 Celsius. When the temperature of the reaction mixture reaches 0 Celsius, add 572 grams of 30% fuming sulfuric acid over a period of 1 hour while stirring the reaction mixture and maintaining its temperature at 0 Celsius. After the addition of the 30% fuming sulfuric acid, add 100 grams of 99% nitric acid over a period of 1 hour while stirring the reaction mixture and maintaining its temperature at 0 Celsius. After the addition of the 99% nitric acid, remove the ice bath and allow the reaction mixture to warm to room temperature. Then heat the reaction mixture to 70 Celsius for 1 hour. After 1 hour, heat the reaction mixture to 80 Celsius for 30 minutes, and then heat to 90 Celsius for 30 minutes. After heating at 90 Celsius for 30 minutes, pour the hot reaction mixture into a clean flask and allow the reaction mixture to cool to room temperature. After which, place the flask into an ice bath and cool the reaction mixture to 0 Celsius for 1 hour. After 1 hour, filter-off the TNT precipitate, and then place the filtered liquid back into the flask and cool it to -10 Celsius by means of a ice/salt bath. Then keep at -10 Celsius for 1 hour. After 1 hour, filter-off the precipitated TNT using the same filter as before. Then wash all the TNT precipitate with 1000 milliliters of cold water, and then vacuum dry or air dry. Then place 108 grams of 70% sulfuric acid into a beaker, and then add the dry TNT product. Then stir the mixture to form a slurry. Continue to stir the slurry for 1 hour at room temperature, and then filter-off the TNT product. Afterwards, wash the TNT with 2000 milliliters of cold water, and then vacuum dry or air-dry the TNT product.

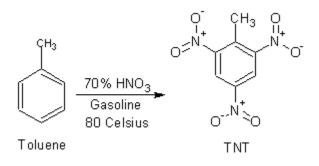
Notes:		

Procedure 11-01B: Preparation of TNT

Materials:	1. 920 grams toluene
	2. 2700 grams 99% nitric acid
	3. 3000 grams premium unleaded gasoline
	4. 1600 grams of 70% sulfuric acid

Summary: TNT can be made by treating toluene with 99% nitric acid in the presence of premium-unleaded gasoline. After the reaction, the TNT is then recovered by recrystallization. The crystallized TNT is collected by filtration, washed, dried, and then purified with 70% sulfuric acid. Commercial & Industrial Note: Part or parts of this laboratory process may be protected by international, and/or commercial/industrial processes. Before using this process to legally manufacture the mentioned explosive, with intent to sell, consult any protected commercial or industrial processes related to, similar to, or additional to, the process discussed in this procedure. This process may be used to legally prepare the mentioned explosive for laboratory, educational, or research purposes.

Hazards: Warning! Wear gloves and use proper ventilation when handling 99% nitric acid, which is a highly toxic and corrosive liquid evolving highly poisonous fumes of nitrogen oxides. Use great care. Extinguish all flames when handling premium unleaded gasoline, which is highly flammable and volatile.



Reaction Equation

Procedure: Place 920 grams of toluene, 2700 grams of 99% nitric acid, and 3000 grams of premium-unleaded gasoline into a flask fitted with stirrer and reflux condenser. Then heat the mixture to about 80 Celsius and reflux for about three hours with constant stirring (do no let the temperature rise above 85 Celsius). In some cases the reaction may take less then three hours, or may take longer. Monitor the nitric acid layer (bottom layer) because the reaction will cease when it disappears. After about three hours, or when the bottom nitric acid layer disappears, shut off the heat and allow the reaction mixture to cool. Upon cooling, some of the TNT begins to precipitate. Instead of filtering off this precipitated TNT, add 3000 milliliters of hot water and stir the whole mixture for about 2 hours. After which, filter off any precipitated TNT, and then decant the upper organic layer. Then place this organic layer into a shallow pan with a high surface area, and allow the upper organic layer to evaporate to recrystallize the bulk of the TNT. When about 80% (by volume) of the upper organic layer has evaporated (heat may be used to speed up the evaporation, but this is not needed due to the volatility of the gasoline), collect the TNT by filtration using the same filter as before, and then wash all the collected TNT product with 2000 milliliters of water. Then dry the TNT in oven at 50 Celsius, or vacuum dry or air-dry the product. Then place 1600 grams of 70% sulfuric acid into a beaker and then add the dry TNT product, and then stir the mixture to form a slurry. Continue to stir the slurry for 2 hours at room temperature and then filter-off the TNT product. Then, wash the TNT with 2000 milliliters of cold water, and then vacuum dry or air-dry the TNT product.

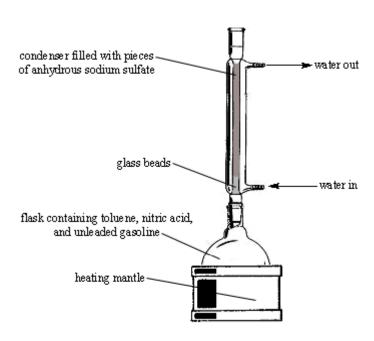


Figure 055. Apparatus for the preparation of TNT.

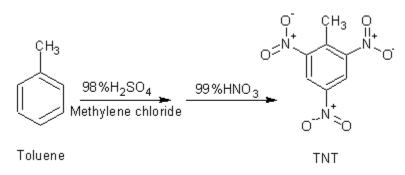
Notes:

Procedure 11-01C: Preparation of TNT

Materials:	1. 920 grams toluene
	2. 2700 grams 99% nitric acid
	3. 8000 grams methylene chloride
	4. 1600 grams of 70% sulfuric acid
	5. 8404 grams of 98% sulfuric acid

Summary: TNT can be made by treating toluene with 99% nitric acid in methylene chloride. After the reaction, the TNT is then recovered by recrystallization. The recrystallized TNT is collected by filtration, washed, dried, and then purified with 70% sulfuric acid.

Hazards: Warning! Wear gloves and use proper ventilation when handling 99% nitric acid, which is a highly toxic and corrosive liquid evolving highly poisonous fumes of nitrogen oxides. Use great care. Wear gloves when handling 98% sulfuric acid.



Reaction Equation

Procedure: Place 920 grams of toluene, 8404 grams of 98% sulfuric acid, and 8000 grams of methylene chloride into a flask fitted with a stirrer and reflux condenser, and then place the mixture into an ice bath while rapidly stirring the mixture. When the mixture reaches 0 Celsius, slowly add 2700 grams of 99% nitric acid over a period of 2 hours while stirring the mixture and maintaining its temperature at 0 Celsius. After the addition of the 99% nitric acid, remove the ice bath and allow the reaction mixture to warm to room temperature. After which, heat the reaction mixture to 70 Celsius for 2 hours while rapidly stirring the reaction mixture. After 2 hours, raise the temperature of the reaction mixture to 80 Celsius and hold this temperature for 2 hours while rapidly stirring the reaction mixture. Afterwards, remove the heat source and allow the reaction mixture to cool to room temperature. Then add 4000 milliliters of cold water and rapidly stir the reaction mixture for 20 minutes. After which, filter the entire reaction mixture to collect any precipitated TNT, and then decant the upper methylene chloride layer. Then place this methylene chloride layer into a distillation apparatus and distill-off the methylene chloride at 40 Celsius until dry solid remains. When dry solid remains, remove the heat source and allow the flask to cool to room temperature. Afterwards, collect the dry TNT product from the flask and then place onto the filter used earlier. Then wash all the collected TNT product with 1000 milliliters of water. After washing, vacuum dry or air-dry the TNT product. Then place 1600 grams of 70% sulfuric acid into a beaker, and then add the dry TNT product. Then stir the mixture to form a slurry. Continue to stir the slurry for 2 hours at room temperature, and then filter-off the TNT product. Finally, wash the TNT with 2000 milliliters of cold water, and then vacuum dry or air-dry the TNT product.

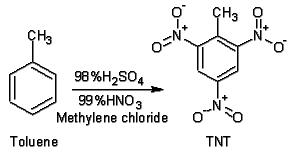
Notes:

Procedure 11-01D: Preparation of TNT

Materials:	1. 920 grams toluene
	2. 4320 grams of potassium nitrate or 3640 grams of sodium nitrate
	3. 44,000 grams methylene chloride
	4. 1600 grams of 70% sulfuric acid
	5. 16784 grams of 98% sulfuric acid

Summary: TNT can be made by reacting a 99% nitric acid/methylene chloride mixture with toluene in the presence of 98% sulfuric acid. The 99% nitric acid/methylene chloride mixture is prepared by extracting a mixture of potassium nitrate or sodium nitrate and sulfuric acid. The reaction mixture is then treated with water, and then filtered to collect any precipitated TNT. The upper methylene chloride layer is then decanted, and evaporated to yield dry solid of TNT. The TNT is then purified by mixing it with 70% sulfuric acid. The acidic mixture is then filtered to collect the TNT, which is then washed with water, and then dried.

Hazards: Wear gloves when handling 98% sulfuric acid and 70% nitric acid, which are both highly corrosive and toxic.



Reaction Equation

Procedure:

Step 1: Preparation of 99% nitric acid/methylene chloride solution

Place 8380 grams of 98% sulfuric acid into a beaker and then place the beaker in a ice bath and cool to 0 Celsius. When the sulfuric acid reaches a temperature of 0 Celsius, slowly add in portions, 4320 grams of potassium nitrate or 3640 grams of sodium nitrate over a period of 2 hours while stirring the 98% sulfuric acid and maintaining its temperature at 0 Celsius. After the addition of the potassium or sodium nitrate, slowly add over a period of one hour, 10,920 milliliters of cold water while continuously stirring the sulfuric acid mixture and maintaining its temperature at 0 Celsius (note: a precipitate may form before or after the addition of the water; if this is the case, never mind it). Afterwards, remove the ice bath and then extract the acid mixture with fourteen 3142-milliliter portions of methylene chloride. Then, combine all fourteen portions of methylene chloride (if not already done so), and then filter the mixture if any insoluble solids are visible.

Step 2: Preparation of TNT

Place the 99% nitric acid/methylene chloride mixture (prepared in step 1) into a beaker, and then add 8404 grams of 98% sulfuric acid. Then cool the mixture to 0 Celsius. When the mixture reaches 0 Celsius, slowly add 920 grams of toluene over a period of 2 hours while rapidly stirring the 98% sulfuric acid/99% nitric acid/methylene chloride mixture and maintaining its temperature at 0 Celsius. After the addition of the toluene, remove the ice bath and allow the reaction mixture to warm to room temperature. After which, heat the reaction mixture to 70 Celsius for 2 hours while rapidly stirring the reaction mixture. After 2 hours, raise the temperature of the reaction mixture to 80 Celsius and hold this temperature for 2 hours while rapidly stirring the reaction mixture. Afterwards, remove the heat source, and allow the reaction mixture to cool to room temperature. After which, add 8000 milliliters of cold water and rapidly stir the whole mixture for 30 minutes. Then, filter the reaction mixture to collect any precipitated TNT, and then decant the upper methylene chloride layer. Then pour this methylene chloride layer into a distillation apparatus and distill-off the methylene chloride at 40 Celsius until dry solid remains. When dry solid remains, remove the heat source and allow the flask to cool to room temperature. After which, collect the dry TNT product from the flask, and then place onto the filter used earlier. Then, wash all the collected TNT product with 2000 milliliters of water. After washing, vacuum dry or air-dry the TNT product. Then place 1600 grams of 70% sulfuric acid into a beaker, and then add the dry TNT product. Afterwards, stir the mixture to form a slurry. Continue to stir the slurry for 2 hours at room temperature, and then filter-off the TNT product. Then wash the TNT with 2000 milliliters of cold water, and then vacuum dry or air-dry the TNT product.

Notes:

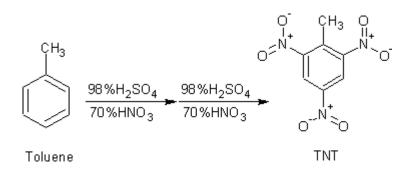
Procedure 11-01E: Preparation of TNT

Materials:	1. 920 grams toluene
	2. 3820 grams 70% nitric acid
	3. 8404 grams of 98% sulfuric acid
	4. 1600 grams of 70% sulfuric acid

Summary: TNT can be made by reacting toluene with a nitrating acid prepared by mixing 70% nitric acid with 98% sulfuric acid. The reaction mixture is then mixed with excess water, and the TNT precipitate is collected by filtration, washed, and then

dried. The dry crude TNT is then purified by treatment with 70% sulfuric acid to form a slurry. The slurry is then filtered to recover the TNT, which is then washed with cold water, and then dried to yield high purity TNT.

Hazards: Same as procedure 11-01D.



Reaction Equation

Procedure: Place 3820 grams of 70% nitric acid into a beaker, and then gradually add over a period of 2 hours, 8404 grams of 98% sulfuric acid. Then place the beaker into a cold-water bath and cool to 10 to 15 Celsius. When the acid mixture reaches a temperature of about 10 to 15 Celsius, pour 1910 milliliters of this acid mixture into a clean separate beaker, and then cool to 10 to 15 Celsius by means of a cold water bath. When the temperature of this 1910-milliliter portion of acid mixture reaches 10 to 15 Celsius, slowly add 920 grams of toluene over a period of 4 hours while rapidly stirring the acid mixture and maintaining its temperature at 10 to 15 Celsius. After the addition of the toluene, continue stirring the reaction mixture for an additional 2 hours while keeping the reaction temperature at 10 to 15 Celsius. After which, add the remaining acid mixture obtained at the start of the procedure, to the reaction mixture and then after the addition, raise the temperature of the reaction mixture to 70 Celsius, and hold this temperature while rapidly stirring the reaction mixture for 2 hours. After 2 hours, raise the temperature to 80 Celsius, and heat at 80 Celsius for 2 hours while rapidly stirring the reaction mixture. After heating the reaction mixture to 80 Celsius for 2 hours, remove the heat source, and allow the reaction mixture to cool to room temperature. Then add the entire reaction mixture to 5000 milliliters of cold water, and then filter-off the precipitated TNT product. After which, wash the TNT precipitate with 2000 milliliters of water, and then vacuum dry or air-dry the TNT precipitate. Then, place 1600 grams of 70% sulfuric acid into a beaker, and then add the dry TNT product. Afterwards, stir the mixture to form a slurry. Continue to stir the slurry for 2 hours at room temperature, and then filter-off the TNT product, wash with 2000 milliliters of cold water, and then vacuum dry or air-dry the TNT product.

Notes:

Standard Explosives "Composition A1" TNT explosive composition

Place 200 grams of TNT into a beaker, and then heat to 90 Celsius. When the TNT melts, maintain the molten TNT at 90 Celsius while stirring, and then add in order, the following ingredients: 1) 1.3 grams of polybutadiene homopolymer liquid resin, marketed under the tradename Poly bd Liquid Resin R-45 A, by the ARCO Chemical Company; then 2) 1.6 grams of a mixture of dehydroabietyl alcohols, sold under the trademark Abitol by the Hercules Chemical Company; then 3) 400 milligrams of epichlorohydrin, marketed by the Shell Chemical Company as "Epon 828"; then 4) 1.6 grams of "Adiprene" L-100. After the addition of the ingredients, continue heating at 90 Celsius with stirring for 2 hours. After 2 hours, pour the molten mixture into any desirable mold, container, bomb casing, or warhead casing under pressure (the container should be able to withstand a temporary temperature of 90 Celsius), and then allow it to cure for several days. For demolitions use, remove the explosive from its mold or container after several days, and then wrap in paper, plastic, or cardboard. Commercial & Industrial Note: For related, or similar information, see Application No. 664,798, March 8, 1976, by The United States Army, to William H. Voigt, Jr., Stanhope, NJ, Lawrence W. Pell, St. Simon's Island, GA, and Jean P. Picard, Morristown, NJ. Part or parts of this laboratory process may be protected by international, and/or commercial/industrial processes. Before using this process to legally manufacture the mentioned explosive, with intent to sell, consult any protected commercial or industrial processes related to, similar to, or additional to, the process discussed in this procedure. This process may be used to legally prepare the mentioned explosive for laboratory, educational, or research purposes.

TNT 20/80 blasting explosive "composition A2"

Mix 200 grams of Estane 5702 into 3000 milliliters of methyl ethyl ketone, and then allow the mixture to stand overnight at room temperature. Afterwards, manually stir the mixture to dissolve the Estane 5702. After the Estane 5702 has dissolved, add 800 grams of TNT. During the addition of the TNT, stir the mixture rapidly until all the TNT is dissolved. After the TNT is dissolved, prepare a solution by dissolving 5 grams of STR-2 powder (marketed by the St. Regis Paper Co., of New York) into 15,000 milliliters of cold water. Then, add the TNT mixture while rapidly stirring the cold water. After the addition, continue stirring the cold-water mixture for 1 hour, and then filter-off the composition A2 precipitate. After which, wash the filtered-off precipitate with four 2-liter portions of cold water, and then dry the precipitate in an oven at 70 Celsius. The dry product mixture thus obtained will be in the form of uniform, free-flowing granules. The explosive composition can then be used directly as granules, or pressed into any desirable mold, container, bomb casing, or warhead casing under high pressure. For demolitions use, after pressing the explosive into any desirable mold or container, remove the explosive from the mold or container and then wrap in plastic, paper, or cardboard. The mold or container should be 1-2 inches wide by 5-11 inches long. For making military dynamite, simply press the explosive into a cardboard tube as illustrated in figure 056. Requires a blasting cap or detonator for initiation. Commercial & Industrial note: For related, or similar information, see Application No. 104,499, December 17, 1979, by The United States Army, to H. William Voigt, Jr., Wharton, NJ, and Bernard R. Banker, Mine Hill, NJ. Part or parts of this laboratory process may be protected by international, and/or commercial/industrial processes. Before using this process to legally manufacture the mentioned explosive, with intent to sell, consult any protected commercial or industrial processes related to, similar to, or additional to, the process discussed in this procedure. This process may be used to legally prepare the mentioned explosive for laboratory, educational, or research purposes.

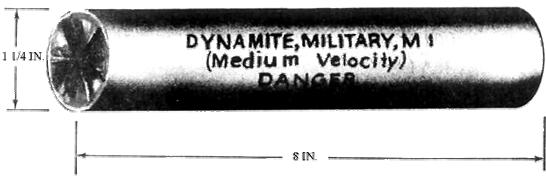


Figure 056. Illustration of US military dynamite. See FM 43-0001-38 for additional information.

TNT-wax castable explosive

Step 1: Preparation of wax composition

Place 170 grams of ozokerite wax into a 500-milliliter beaker and then heat to 90 Celsius to melt the wax. After the wax has melted, add 2 grams of dry lecithin. Afterwards, raise the temperature to 110 Celsius. Then, rapidly stir the molten mixture at 110 Celsius for 90 minutes. After 90 minutes, cool the mixture to 90 Celsius. Then prepare a nitrocellulose mixture by mixing 2 grams of methanol with 30 grams of nitrocellulose, and then add this mixture to the molten wax mixture. After the addition, continue stirring the molten mixture for 40 minutes at 90 Celsius. After which, raise the temperature to 110 Celsius and stir for 40 minutes. After 40 minutes, poor the molten mixture into a shallow pan and allow it to cool. After the wax mixture as solidified, break it up into tiny pieces.

Step 2: Preparation of Explosive Composition

Place 132 grams of TNT into a 300-milliliter beaker and then heat the beaker to 84 Celsius. Then place 36 grams of aluminum powder into a beaker and heat to 80 Celsius. When the TNT has melted, add 32 grams of the wax composition pieces (prepared in step 1) to the melted TNT, and stir rapidly until an emulsion forms. When the emulsion forms, add 36 grams of the heated aluminum powder, and then thoroughly mix the aluminum into the TNT/wax mixture. Afterwards, continue stirring the molten mixture at 84 Celsius for thirty minutes, and then pour the molten mixture into any desirable mold, container, bomb casing, or warhead casing and then cure for several days. For demolitions purposes, remove the mold or container and then wrap the explosive composition in paper, plastic, or cardboard. The mold or container should be 1 - 2 inches wide by 5 - 11 inches long. Requires a blasting cap or detonator for initiation. Commercial & Industrial note: For related, or similar information, see Application No. 925,960, November 3, 1986, by Stephen A. Aubert, Niceville, FL. Part or parts of this laboratory process may be protected by international, and/or commercial/industrial processes. Before using this process to legally manufacture the mentioned explosive, with intent to sell, consult any protected commercial or industrial processes related to, similar to, or

additional to, the process discussed in this procedure. This process may be used to legally prepare the mentioned explosive for laboratory, educational, or research purposes.

"Hexotonol" RDX/TNT explosive

Place 50 liters of water into a large container, and then heat the water to 90 Celsius while stirring. When the water reaches 90 Celsius, add 8 kilograms of RDX and then add 1.2 grams of oxazolin wax, and then stir the mixture rapidly for 1 hour. After 1 hour, add 1000 grams of wax (designated Wax Composition 1), and continue heating and stirring for 1 hour. After which, reduce the temperature to 80 Celsius and then add 1600 grams of TNT while stirring the mixture rapidly for 1 hour. Thereafter, reduce the temperature to 70 Celsius. Then place 6.4 kilograms of TNT into a beaker and heat to 90 Celsius to melt it. When the TNT has melted, add 3 kilograms of aluminum powder, and thoroughly mix for 1 hour. Afterwards, pour the molten TNT mixture into the RDX/TNT/wax mixture and then stir the entire mixture for 1 hour. After 1 hour, filter-off the explosive granules, and then place the granules on a tray and allow them to dry for several days. To use the explosive, heat the dry granules in a beaker at 90 Celsius until molten. When the granules have melted, pour the molten explosive into any desirable mold, container, bomb casing or warhead casing, and allow solidifying. For demolition purposes, remove the solidified explosive from the container, and then wrap in plastic, paper, or cardboard. Instead of melting the granules, the freshly prepared explosive granules can be pressed under high pressure into any desirable mold, container, bomb casing, or warhead casing. For demolitions use, the mold or container should be 1-2 inches wide by 5-11 inches long. Requires a blasting cap or detonator for initiation. Commercial & Industrial Note: Part or parts of this laboratory process may be protected by international, and/or commercial/industrial processes. Before using this process to legally manufacture the mentioned explosive, with intent to sell, consult any protected commercial or industrial processes related to, similar to, or additional to, the process discussed in this procedure. This process may be used to legally prepare the mentioned explosive for laboratory, educational, or research purposes.

"Octol" TNT/Ammonium nitrate explosive

Into a large beaker, place 1200 grams of freshly prepared TNT, and then add 800 grams of dry ammonium nitrate granules (the granules should be about 1 to 2 millimeters in diameter. Thereafter, thoroughly blend the mixture for 1 hour at room temperature. Afterwards, add 5 liters of hot water, and then carefully heat the mixture to 50 Celsius, and then continue stirring for 2 hours. After heating and stirring at 50 Celsius for 2 hours, add 20 grams of standard candle wax, i.e., paraffin wax, and then continue heating at 50 Celsius while stiring for 1 hour. After which time, remove the heat source, and allow the mixture to cool to room temperature. Then place the whole mixture onto a large shallow pan, and allow it to dry. When a dry solid mixture remains, take the dry solid and pulverize it into a coarse powder. Then thoroughly blend this mixture for 1 hour to form a uniform solid mixture. Thereafter, add 50 grams of lubricating oil (the oil may be any motor oil, cooking oil, or petroleum oil), and then manually blend the whole mixture for 2 hours at room temperature. To use the explosive, simply pack it into any desirable mold, bomb casing, or warhead casing under high pressure. For use in shape charges, simply pack it into the desired warhead under high pressure. Note: Octol is well suited for shape charges due to the excessive amount of gas produced by the proper detonation of ammonium nitrate. Requires a blasting cap or detonator for initiation.

Examples of TNT explosive munitions

Fragmenting grenades are common explosive munitions used in warfare. There are many types and sizes of fragmenting grenades, but they all function in a similar manner. Grenades are activated in series of steps beginning with the obvious, the pulling of the safety pin. When the safety pin is released, nothing happens unless the pressure on the safety lever is released (the pressure on the safety lever is maintained by the throwers hand). When this happens, a spring-loaded striker pin rotates over, throwing the safety lever clear, and then striking a primer located on the top of the grenade. The primer ignites a delay element composed of black powder. The delay element can take any where from 2 seconds to 10 seconds to burn down to the detonator, depending on the type of grenade fuse. The detonator is nothing more then a simple blasting cap similar to figures 044 and 045. The following munitions include a standard grenade fuse (left illustration), and a standard military grenade (right illustration). In the left illustration, the grenade fuse represents a standard grenade fuse, and is not used in the grenade illustrated on the right. The grenade fuse in the left illustration is composed of a safety lever, striker pin, primer, and detonator. As described above, when the grenade is thrown, the striker pin rotates over, and strikes a primer. The primer initiates the delay element, composed of a standard black powder fuse. The black powder fuse burns down to the priming mixture, composed of the usual ingredients. The primer mixture initiates the booster charge, composed of lead azide. The lead azide in turn detonates the base charge of RDX. The base charge of RDX detonates the man charge, composed of TNT. In the right illustration, a cutaway view of a standard military grenade is disclosed. When the main charge of TNT detonates, it shatters the cast iron layer, and the steel body, producing fragments of various sizes. These fragments are thrown in many directions with lethal force. Most fragmentation grenades have a casualty radius of about 5 meters. This means that any exposed personnel within the 5meter radius have a 90% chance of getting hit by fragmentation, and the killing percentage is about 60% within the 5 meters. Within 2 meters of the grenade, the killing percentage is about 90%. Beyond the 5 meter radius, the chance of any exposed

personnel getting hit by lethal fragments is less then 30% up to 10 meters, and less then 10% up to 30 meters. Due to the physics of flying fragments, as it corresponds with the amount of explosive used, the strength of the explosive, and the thickness and type of fragmentation used, the flying fragments will not reach a distance of more then 50 meters, 98% of the time.

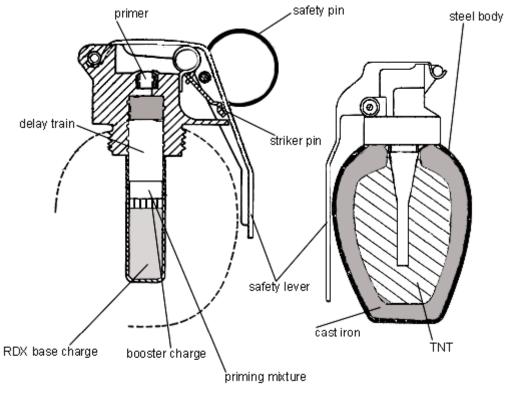
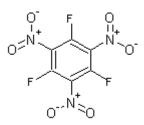


Figure 057. Left illustration: Grenade fuse. Right illustration: Standard military grenade.

11-02. F-TNB. 1,3,5-trifluoro-2,4,6-trinitrobenzene



F-TNB *A secondary explosive.*

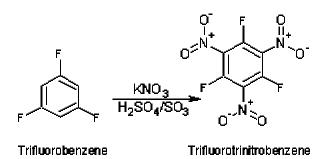
Procedure 11-02A: Preparation of F-TNB

Materials:	1. 2400 milliliters 30% fuming sulfuric acid
	2. 560 grams potassium nitrate
	3. 112 grams 1,3,5-trifluorobenzene
	4. 2400 milliliters methylene chloride
	5. 600 milliliters hexanes
	6. 20 grams crushed charcoal
	7. 40 grams anhydrous sodium sulfate

Summary: In some nitrations, potassium nitrate can be used as the nitrating agent instead of nitric acid. The potassium nitrate is mixed with 30% fuming sulfuric acid, which ultimately forms nitric acid in solution. This nitric acid then nitrates trifluorobenzene, producing F-TNB. Commercial & Industrial note: For related, or similar information, see Application No.

937,281, August 28, 1978, by The United States Navy, to William M. Koppes, Adelphi, MD, Horst G. Adolph, Silver Spring, MD), and Michael E. Sitzmann, Adelphi, MD. Part or parts of this laboratory process may be protected by international, and/or commercial/industrial processes. Before using this process to legally manufacture the mentioned explosive, with intent to sell, consult any protected commercial or industrial processes related to, similar to, or additional to, the process discussed in this procedure. This process may be used to legally prepare the mentioned explosive for laboratory, educational, or research purposes.

Hazards: Wear gloves and use proper ventilation when handling 30% fuming sulfuric acid. 30% fuming sulfuric acid is a highly corrosive, fuming liquid evolving poisonous fumes of sulfur trioxide. Handle with great care. Hexane is a highly flammable, volatile liquid. Do not smoke when handling hexane.

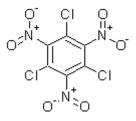


Reaction Equation

Procedure: Place 2400 milliliters of 30% fuming sulfuric acid into a 6-liter 3-neck flask equipped with a stirrer, condenser, and thermometer. Then cool to 0 Celsius using an ice bath. When the temperature of the 30% furning sulfuric acid reaches 0 Celsius, slowly add, in small portions, 560 grams of potassium nitrate while keeping the temperature of the 30% fuming sulfuric acid below 50 Celsius and stirring. After the addition of the potassium nitrate, remove the flask from the ice bath and then heat the flask to 50 Celsius. Afterwards, slowly add 112 grams of 1,3,5-trifluorobenzene while maintaining the reaction temperature at 50 Celsius. After the addition of the 1,3,5-trifluorobenzene, reflux the reaction mixture at 153 Celsius for 72 hours. After refluxing for 72 hours, allow the reaction mixture to cool to room temperature, and then extract the reaction mixture with six 400-milliliter portions of methylene chloride. After the extraction process, combine all 400-milliliter portions, and then place into a distillation apparatus. Then distill at 40 Celsius until the total volume of the remaining methylene chloride in the distillation flask equals 500 milliliters. When this volume is achieved, remove the heat source, and then allow the remaining methylene chloride in the distillation flask to cool to room temperature. After which, add 40 grams of anhydrous sodium sulfate (to absorb water), and then shake the mixture for about 20 minutes. Thereafter, filter-off the sodium sulfate, and then heat the filtered mixture to about 50 Celsius. Then add 300 milliliters of hexane, and continue heating the mixture at 50 Celsius for ten minutes. After which, remove the heat source, but before the mixture cools, add 20 grams of crushed charcoal (to absorb impurities), and stir the whole mixture for ten minutes. After stirring for ten minutes, filter-off the charcoal. Then place the mixture into a distillation apparatus, and distill at 40 Celsius until no more methylene chloride is collected in the receiver flask. Afterwards, filter the remaining liquid in the distillation flask hot, and then add 300 milliliters of hexane. Then stir the mixture for ten minutes. After stirring for ten minutes, place the liquid into a shallow pan, and allow it to evaporate to yield dry solid. After the evaporation is complete, recover the dry crystals of F-TNB.

Notes:

11-03. TCTNB. *Trichlorotrinitrobenzene; 1,3,5-trichloro-2,4,6-trinitrobenzene*



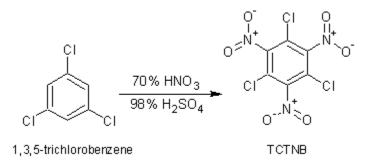
TCTNB *A secondary explosive*.

Procedure 11-03A: Preparation of TCTNB

Materials:	1. 68 grams 1,3,5-trichlorobenzene
	2. 100 grams 70% nitric acid
	3. 110 grams 98% sulfuric acid

Summary: TCTNB is prepared by reacting trichlorobenzene with a nitrating acid mixture composed of nitric and sulfuric acids. The resulting product is then filtered, washed with water, dried, and then recrystallized from benzene. Commercial & Industrial note: Part or parts of this laboratory process may be protected by international, and/or commercial/industrial processes. Before using this process to legally manufacture the mentioned explosive, with intent to sell, consult any protected commercial or industrial processes related to, similar to, or additional to, the process discussed in this procedure. This process may be used to legally prepare the mentioned explosive for laboratory, educational, or research purposes.

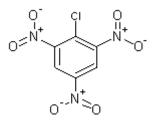
Hazards: Wear gloves when handling 70% nitric acid and 98% sulfuric acid. 98% sulfuric acid can cause severe skin burns, and 70% nitric can cause tissue damage due to its strong oxidizing nature. Use care.



Reaction Equation

Procedure: Prepare a nitrating acid mixture by adding 110 grams of 98% sulfuric acid into 100 grams of 70% nitric acid (always add the sulfuric to the nitric, and never the reverse). Then cool the acid mixture to 20 Celsius using a cold-water bath, and stir. Then slowly add 1,3,5-trichlorobenzene, in small portions, over a period of 2 hours while stirring the acid mixture and keeping the acid mixture at 20 Celsius. After the addition of the trichlorobenzene, heat the reaction mixture to 40 Celsius for 2 hours with strong stirring. After 2 hours, cool the reaction mixture to room temperature, and then pour the entire reaction mixture into 1000 milliliters of cold water. Afterwards, filter-off the precipitated product, and wash with 1000 milliliters of cold water several times. After washing the product with water, vacuum dry or air-dry. Next, recrystallize the product from 400 grams of benzene, and then wash with 1000 milliliters of cold water, and then vacuum dry or air-dry the product. During the recrystallization process, do not evaporate all the benzene. The last of the remaining benzene during the recrystallization process. The remaining volume of 15% will contain mostly dissolved by-products, and can be recycled for the next recrystallization of TCTNB if desired.

Notes:



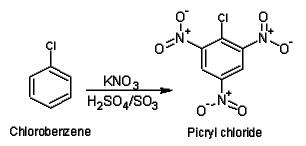
Picryl chloride *A secondary explosive*

Procedure 11-04A: Preparation of Picryl chloride

Materials:	1. 160 grams chlorobenzene
	2. 710 grams potassium nitrate
	3. 1600 milliliters 30% fuming sulfuric acid
	4. 200 milliliters acetone
	5. 600 milliliters methanol

Summary: Picryl chloride is prepared by treating chlorobenzene with potassium nitrate and fuming sulfuric acid. The picryl chloride is then precipitated, washed with water, and then dried. The picryl chloride is then purified by recrystallization from an acetone/methanol mixture. Commercial & Industrial Note: Part or parts of this laboratory process may be protected by international, and/or commercial/industrial processes. Before using this process to legally manufacture the mentioned explosive, with intent to sell, consult any protected commercial or industrial processes related to, similar to, or additional to, the process discussed in this procedure. This process may be used to legally prepare the mentioned explosive for laboratory, educational, or research purposes.

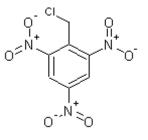
Hazards: Wear gloves and use proper ventilation when handling 30% fuming sulfuric acid, which is a highly toxic and corrosive liquid evolving toxic fumes of sulfur trioxide. Use great care. Wear gloves and use proper ventilation when handling chlorobezene. Chlorobenzene is a highly irritating substance. Avoid skin contact and eye contact. Use proper ventilation and do not smoke when handling acetone and methanol. Acetone and methanol are highly volatile and flammable liquids.



Reaction Equation

Procedure: Place 1600 milliliters of 30% fuming sulfuric acid into a 4-liter flask equipped with a stirrer, thermometer, and condenser. Then place the flask into a cold-water bath, and then slowly add 710 grams of potassium nitrate while stirring the fuming sulfuric acid and keeping its temperature below 60 Celsius. After the addition of the potassium nitrate, allow the solution to cool to about 30 Celsius (if necessary), and then slowly add 160 grams of chlorobenzene while stirring the reaction mixture, and keep its temperature below 50 Celsius. After the addition of the chlorobenzene, remove the cold-water bath, and then reflux the reaction mixture at 125 Celsius for 4 hours. After refluxing for 4 hours, remove the heat source, and allow the reaction mixture to cool to room temperature. After which, add the reaction mixture to 1000 grams of ice contained in a 3000 milliliter beaker. After the ice has melted, add 1000 milliliters of cold water, and stir the entire mixture for 20 minutes. Then filter-off the precipitated product, wash with two 500-milliliter portions of cold-water, and then vacuum dry or air-dry the product. After drying, dissolve the product in 200 milliliters of acetone, and after the entire product has dissolved, add 600 milliliters of methanol. Finally, recrystallize the product from this solvent mixture. After recrystallization, wash the product with two 200-milliliter portions of cold water, and then vacuum dry or air-dry the product.

11-05. TNBCI. Trinitrobenzylchloride



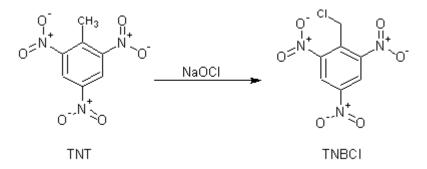
TNBCl *A secondary explosive*

Procedure 11-05A: Preparation of TNBCI

Materials:	1. 20 grams TNT
	2. 200 milliliters 5% sodium hypochlorite (bleach) (commercially available; clorox)
	3. 200 milliliters tetrahydrofuran (THF)
	4. 100 milliliters methanol
	5. 20 milliliters 35 – 38% hydrochloric acid

Summary: TNBCl is easily prepared by quickly adding a TNT solution to a 5% sodium hypochlorite solution, and then quickly adding dilute hydrochloric acid to stop the reaction and eliminate side reactions. The solution is then allowed to stand for 1 hour, and then filtered to collect the product. The product is washed, and then dried. Commercial & Industrial Note: Part or parts of this laboratory process may be protected by international, and/or commercial/industrial processes. Before using this process to legally manufacture the mentioned explosive, with intent to sell, consult any protected commercial or industrial processes related to, similar to, or additional to, the process discussed in this procedure. This process may be used to legally prepare the mentioned explosive for laboratory, educational, or research purposes.

Hazards: Wear gloves and use proper ventilation when handling 35 - 38% hydrochloric acid, which is a highly corrosive and toxic liquid evolving toxic and corrosive fumes.



Reaction Equation

Procedure: Place 200 milliliters of THF and 100 milliliters of methanol into a flask, and then add 20 grams of TNT while stirring the THF/methanol mixture. Then cool the solution to 0 Celsius by means of an ice bath. Then place 200 milliliters of a 5% sodium hypochlorite solution into a large flask, and then cool to 0 Celsius by means of an ice bath. Now, prepare a dilute hydrochloric acid solution by adding 20 milliliters of 35 - 38% hydrochloric acid into 2000 milliliters of water. When the 5% sodium hypochlorite solution reaches 0 Celsius, quickly add all of the THF/methanol/TNT mixture while stirring the 5% sodium hypochlorite solution, and maintain its temperature below 15 Celsius. As soon as the THF/methanol/TNT mixture has

been added to the 5% sodium hypochlorite solution, wait exactly 1 minute, and then stop the reaction by quickly adding the dilute hydrochloric acid solution while stirring the reaction mixture (waiting longer then 1 minute will cause a secondary side reaction). After the addition of the dilute hydrochloric acid solution, allow the reaction mixture to stand at 0 Celsius for about 2 hours while stirring. After1 hours, filter-off the product, wash with six 200-milliliter portions of cold water, and then vacuum dry or air-dry the product. The result will be 20 grams of trinitrobenzylchloride with a melting point of 85 Celsius.

Notes: