

CHAPTER 13: THE PREPERATION OF NITRATE ESTERS

different sizes of mesh sieves (after the roll milling process). 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.

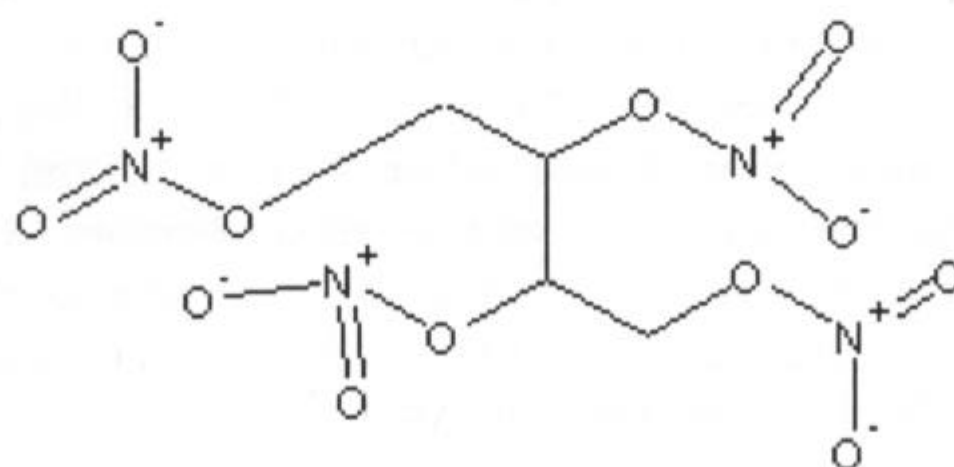
Nitrocellulose blasting powder

Into a suitable blender, add 52 grams of freshly prepared, filtered-off, wet nitrocellulose, and then add 6 grams of glycerin, followed by 1 gram of oleic acid (olive oil may be used). Then begin blending of the ingredients at a moderate speed. Shortly thereafter, gradually add in 152 grams of ammonium nitrate, while stirring the mixture moderately. After adding the ammonium nitrate, increase the mixing speed to fast, and then blend for 1 hour to form a uniform blended mixture. After which, stop the blending, and then place the doughy mixture onto a shallow tray, and then dry in an oven at 50 celsius until the water has been removed. Thereafter, remove the dry mixture, and then simply pack it into a waxed coated cardboard tube, and then fold over both ends, and seal the folded ends with molten wax. The cardboard tube in practice should be about 2 inches wide, by 6 inches long. A blasting cap or detonator is required 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.

Nitrocellulose blasting powder (suitable for making anti-personnel bursting munitions)

Into a crucible, add 160 grams of dry powdered nitrocellulose, 200 grams of potassium nitrate, and then 40 grams of powdered sulfur. Thereafter, vigorously blend the mixture for several hours to obtain a uniform mixture. After the blending period, press it into any desirable container under high pressure. The containers may be made of steel/iron, aluminum, lead, glass, tin, or copper. For best results, a thick walled steel tube, 1 inch wide by 3 inches long, sealed at both ends with steel end caps works (remember a blasting cap hole must be drilled into one of the end caps prior to use; the blasting cap hole, after insertion of the blasting cap, may be sealed with grease). This will produce a casualty radius of about 5 meters. Larger steel tubes may be used, but larger devices are not recommended for anti-personnel use, because large devices tend to blow upwards, decreasing the effectiveness of shrapnel. For demolitions use, simply pack the dried well-mixed explosive into a cardboard tube 2 inches wide by 6 inches long, and then seal both ends in the usual manner. 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.

13-03. ETN. *Erythritol tetranitrate*



ETN

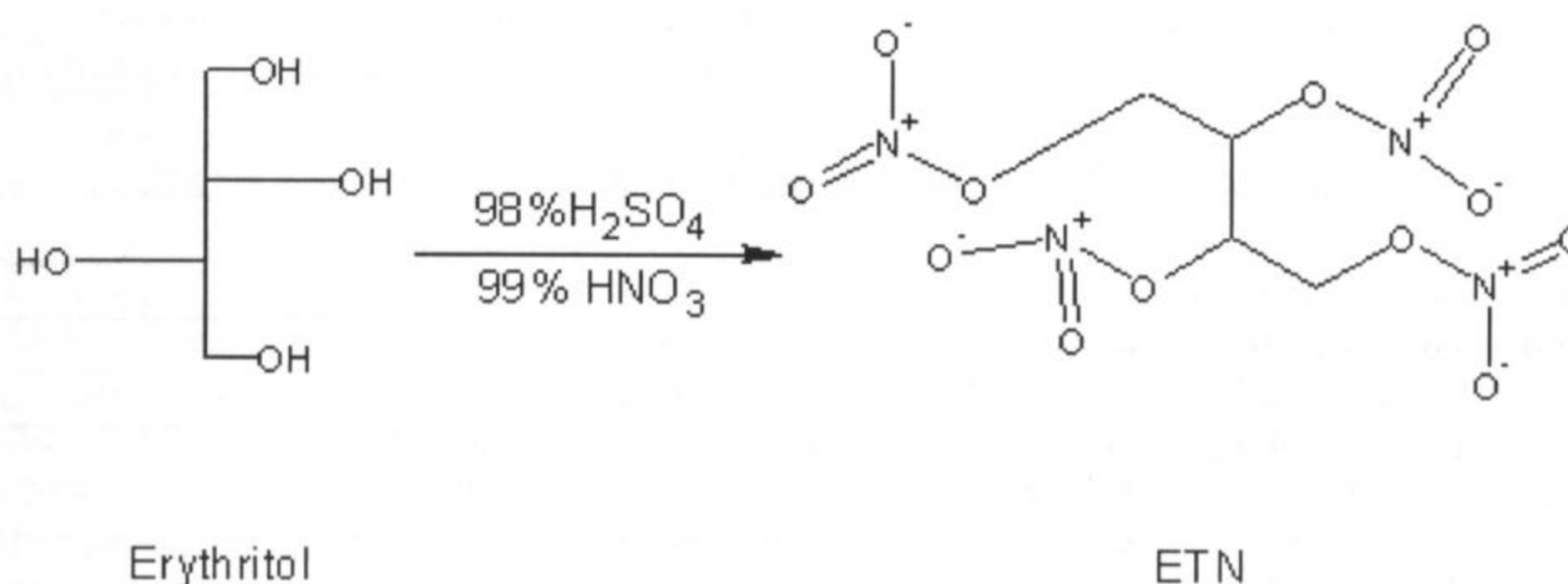
ETN forms colorless or white crystals, leaflets, or powder with a melting point of 61 Celsius. It is soluble in alcohol, ether, and glycerol, but insoluble in water. ETN explodes on strong percussion, but still requires a primary explosive for proper initiation when used in explosives compositions. ETN is used as a substitute for PETN in blasting cords, for use in explosives compositions, and nitroglycerine free dynamites when mixed with TNT, nitrocellulose, or ammonium nitrate.

Molecular weight: 302.108	Flammability: May flash when strongly ignited
Detonating velocity: Similar to PETN	Toxicity: Moderate
Sensitivity: Moderate	Classification: Secondary explosive
Stability: Good	Overall value (as secondary explosive): Moderate

Procedure 13-03A: Preparation of ETN

Materials:	1. 110 grams 98% sulfuric acid
	2. 20 grams of erythritol
	3. 140 grams of 90% nitric acid
	4. 200 milliliters of 10% sodium carbonate solution
	5. 1000 milliliters of 95% ethanol

Summary: ETN is prepared by dissolving erythritol into 98% sulfuric acid, followed by treatment with 90% nitric acid. The reaction mixture is stirred for a short time afterwards, and then allowed to stand. The whole reaction mixture is then mixed with cold water, which causes precipitation of the ETN. The ETN is then dissolved into warm 95% ethanol, and then after filtration (to remove impurities), the ethanol mixture is treated with an excess of cold water. The addition of the cold water causes the ETN to precipitate. The ETN is then washed, and then dried. Commercial & Industrial note: For related, or similar information, see Serial No. 184,180, November 20th, 1928, by E.I. Dupont de Nemours & Company, to Frank H. Bergeim, Woodbury, 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.

*Reaction Equation*

Hazards: Use caution when handling 90% nitric acid. 90% nitric acid is highly toxic, and emits highly poisonous fumes of nitrogen oxides; use caution. Wear gloves when handling 98% sulfuric acid.

Procedure: Add 110 grams of 98% sulfuric acid into a suitable flask, and then place the flask into an ice water bath. When the acid is cooled to about 10 Celsius, gradually add 20 grams of erythritol over a period sufficient enough to keep the acid at a temperature below 40 Celsius. Note: If the temperature rises above 40 Celsius, carbonization of the erythritol will begin. During the addition, rapidly stir the sulfuric acid. After the addition of the erythritol, place 140 grams of 90% nitric acid into a clean flask, and then cool this flask in a hot water bath at about 30 Celsius. After which, add the sulfuric acid/erythritol mixture over a period suitable to keep the nitric acid at a temperature below 60 Celsius. During the addition, rapidly stir the nitric acid. After the addition, rapidly stir the reaction mixture for 1 hour. After stirring for about 1 hour, stop stirring, and then allow the reaction mixture to stand for 30 minutes. Then, pour the entire reaction mixture into 1000 milliliters of ice water. Thereafter, filter-off the precipitated product, wash with several hundred milliliters of cold water, 200 milliliters of 10% sodium carbonate solution, and then several hundred milliliters of warm water. After washing, vacuum dry or air-dry the product. Then add and dissolve the product into 1000 milliliters of warm 95% ethanol. After which, filter-off any insoluble impurities, and then add 1000 milliliters of ice-cold water, and stir the mixture for ten minutes. After which, filter-off the predicated product, wash with several hundred milliliters of cold water, and then vacuum dry or air-dry the product. The result will be high purity ETN.

Notes:

13-04. PEN. Pentaerythritol trinitrate