ETN Synthesis & Non-Energetic Plastic Binders

Below are the notes to extremely successful ETN synthesis

Erythritol Tetranitrate- Lab Notes:

4/3/06
Please note that I started off copying a method found already tested.
Reactants used:

100ml (178g) H_2SO_4 (93%) (I have found that SOME H_2SO_4 is higher % than others. I would digress and say that brands such as Rooto are 93 while others may be in the 95-6 range. Remember this is surplus acid from various sources.)
61g NH_4NO_3 (VERY finely powdered)
20g C_4-H_{10}-O_4 (Erythritol)
2000ml H_2O ESU (Estimated Substance Used)
200g $NaHCO_3$ ESU (Estimated Substance Used)

Equation:
H_2SO_4 + NH_4NO_3 --> HNO_3 + NH_4HSO_4? (Not Sure)
$C_4H_{10}O_4$ + 4HNO_3 --> $C_4H_6N_4O_{12}$ + 4H_2O

g (44.44ml) H_2SO_4 + 60g NH_4NO_3 --> 40g (28.57ml) HNO_3 + 73.23g NH_4HSO_4? (I would need a bit more H_2SO_4 since it's not concentrated therefore 50ml is good.)

19.52g C_4-H_{10}-O_4 + 40g (28.57 ml) HNO_3 --> 48.32g $C_4H_6N_4O_{12}$ + 11.52g H_2O
-=*=-
04/10/06
---->This is the best one:
20gr Erythritol
60gr ammonium nitrate
110ml H_2SO_4 (93%)
Mix the solid nitrate and acid w/ magnetic stirring device until clear solution is formed; cover with Al foil. Refrigerate. Maintain @ 20C. When COLD use ice/water bath to maintain temp and add Erythritol about ½ gram at a time. Solution will thicken quickly. Continue to stir w/ hand stirring. Then refrigerate one hour. Yield will be approx 85% or about 40gr ETN ! Re-crystallize with etoh. Acetone is too soluble but both will suffice. Re-crystalization is a major issue. What has proooven itself is /14 acetone - 3/4 etoh. Methanol -=does=- appear to be a useful solvant. This has been too long debated. Experiments conducted on 5/16/06 proove that re-crystalization from methanol will work. The issue is one of the alcohol's purity level. a reverse re-crystalization has shown itself to work under standard lab conditions. That is a hot-plate / magnetic stirrer to bring the methanol up to boiling. The result was excellent; crystals were well formed and uniform. Too strong is acetone; dissolving Erythritol Tetranitrate to an extreme. When used in moderation it will help the re-crystalization but will add a film if acetone has some impurities. Which is most hardware store acetone when heat is applied. Pure ethanol appears to be best but it takes a great deal of solvant to totally disolve the raw material. The object is to dissolve the raw Erythritol Tetranitrate and then re-crystalize it without any acid in the crystal and with some stabilizer such as dimethelamine. Urea may appear to be a subsitute if all other anti-acid measures are met.Unlike PETN, Erythritol Tetranitrate is not as stable and needs the attention given MHN or other powdered nitric esters. Proformance wise it is on par with NG, PETN, or MHN; about 7000+mps but it will not match PETN for overall stability. It is close, if given proper re-crystalization and stabilization. Far easier to make properly than MHN and it's raw material much more readily available than PETN. A quick test of placing a few milligrams on a steel anvil and striking it with a hammer will demonstrate it's success and, of course, it's power. A bit the size of a half a match-head folded in some foil and hit with a hammer will detonate with all the volume of a small caliber pistol. ETN is noted for it's sensitivty to percussion stimuli. While this does indicate a degree of success in the synthesis, there are other indicators. The crystal size and shape being almost as important as the MP. Under a microscope you will see a consistent shape to the crystal. There should be an abundance of very well formed crystals as well. Using the "reverse-recrystalization" method, the crystals should be the size of table salt: about

100 mesh. The colour should be just off white. A yellow discolouration is to be avoided. This would indicate that there exists some impurities within the ETN and while this presents no problem for the short term, it will evolve into a break down of NO_x after a time or upon exposure to storage temp above 20C for several months.

NOTE: I used differing weights of NH_4NO_3 and excess sulfuric acid; This had been altered from the original percentages of this synth. The same goes with the extra Erythritol, To improve yields should more nitric be produced, I lowered the orginal weights (from 25 to 20 gr of Erythritol.

Procedure:
H_2SO_4 was chilled inside of a flask, in the freezer. After taking it out, the procedure followed immediately. The chilled H_2SO_4 was measured out to be -10 degrees C. The flask was placed magnetic stir platform. NH_4NO_3 was added in small portions, not letting the temperature jump higher than 15 degrees C. After the full addition of the NH_4NO_3 I pulled out thermometer and noticed white fumes, signifying that I had high % nitric, the addition of C_4-H_{10}-O_4 (aka E) began. The E was added slowly again not letting the temp jump over 15 degrees C to reduce loss of possible nitric loss. The mixture was white with some NH_4NO_3 left over in it, but it dissolved after the addition of E. The flask was measured at 5 degrees C right before E addition. The mix was left to nitrate for 55 minutes with stirring ever 5 minutes or so. The mix was thick and creamy. This was dumped into a 400ml flask with water. The mix immediately became cloudy, signs of a decent yield. This mixed solution was then poured SLOWLY into a beeker of cold ice water. I was sure to wash the material with a solution of baking soda repeatedly as I do not attempt to neutilize at the time of nitration but always re-crystalize and at that time use other neutrilzation methods & diphenylamine for stablization. I added some more $NaHCO_3$ to test for complete neutralization (none occurred.) This was again dumped into water for removal of any water-soluble stuffs. The ETN was then filtered through a large paper towel. Drying was done in the free air on top of dry paper towels. There were some problems though because the pot that held my filter was not big enough so I had to do 3 filtrations, one of them worked very well with at least 15g of product. The second one failed and my "neutralized mixture" ate right through the 3 paper towels I was using for filtering. It had an estimated 15g on it. The third was a refiltration of the second and I got around 5-10g on it. The two filtered products are drying. My 1st attempt w/ KNO_3 was not nearly as good as the NH_4NO_3. I made sure of not only the weight but ground it further ina coffee mill. This produced a ultra fine powder that mixed very well.However it is to be noted that even w/ a magnetic stir plate after a bit the mix was too heavy to mix without a manual method.

Estimated (before filtration Yield):35+g of ETN
Estimated (after filtration) Yield: 25g of ETN
Actual Yield: 50 grams of ETN
at least 90% Efficiency ?

Note some stuff isn't filled in, this is because it's still drying I will edit that in later. Here are some things I learned (2nd nitration):
Methanol is actually one of the best re-crystalization solvants available. Clean glassware is a must with ETN as it's sensitivity to contaminants is profound. Length of time within nitration is important. The minimum appears to be two hours; optmum is six hours. temp is not too critical but the best is always colder, of clourse. there appears to be little chance of a run-a-way even at 30+ C but the HNO_3 starts to affected in a solid nitrate-nitration at higher temps.

Also be aware that ETN is profoundly detected as an explosive from almost every explosive detector ever made. In the words of an expert in that area; "it has a profound odor". It's dust or touch will make every detector alarm scream and one's hands should be washed very well indeed.

Plastics

One of the most successful materials for an ETN plastic is a compostion of VersaGel C-Series (GEL-WAX from Yaley thinned w/ naptha). VersaGel C-Series MSDS PEN13392 which is essentially a Hydrocarbon polymer!
There is a difference between the real product (Styrene Butadiene rubber) used in commercial production of PBX
and the wax. But in some countries I believe that it had been used for military purposes. The actual pariffin material is a polimer synthetic but I have no proof of it's compostion as Yaley won't talk about it's product except to say it's a patented wax product that it has exclusive rights to. Wax has been used with PBX for years
and has been a very successful phlemitiser for PETN, RDX, and a host of Los Alamos mil explos.
More to follow on "Gel-Wax":
This is gelled pariffin oil also known as VersaGel C-Series or C-Series mineral oil gel. A very unique material as it offers one of the best binders for explosives on the market that is not specifically designed as an energetic binder. With certain materials it will produce an energetic component where a refined petroleum is part of the embodiment of the explosive. It has been used with many if not most of the Los Alamos PBXs and is followed closely by the costly Silicon Oil as a binder.

Styrene Butadiene: Plastic Bonded Explosive - binder

Listed as CAS 9003-55-8 (started in late 2005)
Basically Styrene Butadiene Rubber is the bonding agent for both Semtex (the original & later) and C4. In addition the companies that mfg the polymers that use it also use the precursors that are used in mfg of various energetic materials.
Styrene Butadiene is incorporated in DuPont's Deta Sheet but has been used the most in the mfg of plastic energetic materials. Styrene Butadiene is a difficult product to obtain OTC. It is considered a Strategic Material by Jane's and although found in many common products is generally not sold to the public in unadulterated form. Styrene Butadiene combined with a powdered energetic material such as PETN, RDX in various percentages yields the closest thing to commercial PBX (Semtex, C4, etc). It binds VERY well and produces just what would be expected from a commercial PBX.

US-Mil contractor: DOW-Riechhold Specialty Div.
Styrene Butadiene Latex 9003-55-8 (no CAS # prior to 2004)

SBR   Styrene Butadiene Rubber   DOW-Riechhold Specialty Latex LLC offer 36 different Styrene Butadiene products.
Other major suppliers have been Vector and Dexco. Their Styrene Butadiene products are in common production.

Solution Styrene Butadiene Rubber (S-SBR)

Solution styrene butadiene rubber (S-SBR) is used in a wide variety of applications, including the production of tires, footwear, conveyor belts, hoses, flooring and adhesives.

Solid solution polymerized styrene butadiene rubber, produced by anionic batch polymerization is available in a wide variety of styrene and vinyl contents. S-SBR provides excellent balance between wet grip, rolling resistance and dry handling in silica and carbon black compounds for high-performance tires. They are also used for the manufacture of high quality technical rubber goods. Styrene Butadiene SBR (SBR) is a polymeride consisting of styrene and butadiene. It has good abrasion resistance and good aging stability.

{I don't want to give this away so there will be a run on this product but what the Hell...}
OTC source in the USA: Styrene Butadiene of a very high quality and % is used as floor covering adhesive by several companies that sell their adhesive in bulk. It is nearly impossible to find high percentage Styrene Butadiene except for one outlet. A common hardware store in the USA that begins with the letter "A" and has stores in most all large cities and some smaller ones lists this product as MSDS #601135 and is listed as

Multipurpose Floor Covering Adhesive. Internal store product #12875. It is available in one quart and larger cans. The % of Styrene Butadiene Rubber is over 90%, the rest is paraffin oil (mineral oil) & a trace amount of naptha. It's flash point is 93 C which is in the correct range for this product. Color is beige turning to dirty white when dry. Just as it should be. It binds VERY well with most any powdered material, making a clay-like substance that is off white if the powder is white.

-=IF=- you have found another quality source please let it be known.

These companies will be able to supply a variety of materials.

Rubber adhesives and rubber sealants are highly flexible, natural or synthetic materials that are used to join components or fill gaps between seams or on surfaces. Natural rubber is based on polyisoprene. Synthetic rubbers and elastomers include silicone, polyurethane, polysulfide, styrene-butadiene rubber (SBR), butyl, acrylic or polyacrylate, isoprene, polyisobutylene, ethylene-vinyl acetate (EVA), vinyl and nitrile compounds. Pressure sensitive adhesives (PSAs) are often based on non-cross linked rubber adhesives in a latex emulsion or solvent-borne form. Rubber adhesives and rubber sealants are also available as aerosols, films, gels, liquids, slurries, solids, pastes, powders, and putties. They are compatible with substrates made from ceramics and glass, concrete and masonry, paper and paperboard, rubbers and elastomers, leather, textiles, metal, plastic, wood, porous surfaces, and composite materials. Rubber adhesives and rubber sealants are used in a variety of aerospace, automotive, electrical, electronic, marine, medical, and military applications. They are also used in abrasives, optics, photonics, and semiconductors.

Rubber adhesive and rubber sealants use several curing technologies. Air setting or film drying forms bonds by evaporating water or organic solvents. Anaerobic adhesives cure in the absence of air or oxygen. Thermoplastic or hot melt adhesives can be repeatedly softened by heat and then hardened or set by cooling. Thermoset adhesives are cross linked polymeric resins that are cured with heat or heat and pressure. Single component adhesive or sealant systems consist of a resin that hardens by reaction with surface moisture or the application of heat. Two or multi-component systems consist of two or more resins or a resin and hardener, crosslinker, activator or catalyst that, when combined, react and cure into a polymerized component or bond. Some abrasives use ultraviolet (UV) light, visible light, or electron beam (EB) irradiation to initiate curing. Reactive resins such as polyurethane reactives (PUR) are single component adhesives that react with moisture to cross link and polymerize.

Important specifications for rubber adhesives and rubber sealants include viscosity, full cure or set time, ultimate tensile strength, elongation, shear strength, and maximum use temperature. Viscosity, a measurement of a fluid’s resistance to flow, determines the amount of adhesive dispensed on a substrate. Full cure or set time depends upon the curing temperature and varies among thermosetting systems. Ultimate tensile strength (UTS) is the amount of applied stress required to cause failure in a control specimen under tensile load conditions. Elongation is the amount of permanent deformation after a controlled tensile test. Shear strength is the maximum shear load per unit cross-section that an adhesive joint can withstand before mechanical failure or breakage occurs. Maximum use temperature is the temperature range to which a material can be exposed without degradation of structural of other required end-use properties.

Rubber adhesives and rubber sealants are available with a variety of features. Products that are designed for electrical and electronics applications are often provide protection against electrostatic discharge (ESD), electromagnetic interference (EMI), or radio frequency interference (RFI). Thermal compounds that use a phase change are able to absorb additional heat from electronic devices or electrical components. Retaining adhesives position cylindrical mating parts such as bearings, pulleys, rotors, and gears. Transfer adhesives are coated onto single or double-sided liner. Thread locking products protect transverse and axial loads against vibration loosening. Removable adhesives can be repositioned without de-laminating a substrate or leaving a residue. Flexible adhesives and sealants form a layer that can bend or flex without cracking. Dampening products reduce sound, shock, or vibration. Chemically resistant products can withstand acids, alkalis, oils and fuels. Weatherproof or weather resistant materials are designed to withstand wind environmental variables. Non-toxic, low-odor, colorless, and transparent rubber adhesives and rubber sealants are also available.

source & Mfg.

BEMIS (Manufacturer) Shirley, MA
Can-Do National Tape (Manuf., Distrib. & Service) Nashville, TN
Henkel Corp. - Automotive (Manuf. & Service) Madison Heights, MI
Stik-II Products (Manuf. & Service) Easthampton, MA
32group / Raw & Processed Materials Division (Manufacturer) New York, NY
ADCO Products, Inc. (Manuf. & Service) Michigan Center, MI
American National Rubber Co. (Manuf. & Service) Farmington Hills, MI
Anchor Seal, Inc. (Manuf. & Service) Danvers, MA
APV Engineered Coatings (Manuf. & Service) Akron, OH
Basic Adhesives, Inc. (Manufacturer) Brooklyn, NY
Binding Source, LLC (The) (Distributor) Torrington, CT
Boss Products (Manufacturer)
Bostik Findley, Inc. (Manufacturer)Middleton, MA
Chamberlin Rubber Company, Inc. (Manuf., Distrib. & Service) Rochester, NY
Chase Corp. / Royston Laboratories Division (Manufacturer) Pittsburgh, PA
Chemsol, Inc. (Manufacturer) Farmington Hills, MI
Cytec Conap, Inc. (Manufacturer) Olean, NY
Cytec Engineered Materials (Manufacturer) Anaheim, CA
DAP Inc. (Manufacturer) Baltimore, MD
Daubert Chemical Co., Inc. (Manufacturer) Chicago, IL
Devcon (Manufacturer) Danvers, MA
Dyna-Tech Adhesives, Inc. (Manufacturer) Grafton, WV
EFTEC North America LLC (Manufacturer) Madison Heights, MI
Ellsworth Adhesive Systems (Distributor) Germantown, WI
Flamemaster Corp. Chem Seal Div. (Manufacturer) Sun Valley, CA
General Sealants, Inc. (Manuf. & Service) City of Industry, CA
Hanna Rubber Company (Service) Kansas City, MO
Henkel Surface Technologies (Manufacturer) Madison Heights, MI
Innovative Polymers, Inc. (Manufacturer) St. Johns, MI
Key Polymer Corporation (Manuf. & Service) Lawrence, MA
Normac Adhesive Products, Inc. (Manufacturer) Canada
Noveon, Inc. (Manuf. & Service) Cleveland, OH
NuSil Technology (Manufacturer) Carpinteria, CA
OCM, Inc. (Manufacturer) Vernon Hills, IL
On-Hand Adhesives, Inc. (Distrib. & Service) Mount Prospect, IL
PAK-LITE INC. (Manuf. & Service) Suwanee, GA
Patch Rubber Company (Manuf. & Service) Roanoke Rapids, NC
Permatex Inc. (Manufacturer) Hartford, CT
PHC Industries, Inc. (Distributor) Fort Wayne, IN
RELTEK, LLC (Manufacturer)Santa Rosa, CA
Rubber Mold Compounds & Plastics (Distributor) Clifton Park, NY
Rubber-Seal Products (Manufacturer) Dayton, OH
Scapa Tapes North America (Manufacturer) Windsor, CT
Silpak, Inc. (Manuf. & Service) Pomona, CA
Slocum Adhesives Corporation (Manufacturer) Lynchburg, VA
Specialty Adhesives, Inc. (Distrib. & Service) Pelham, AL
Specialty Products of Michigan, Inc. (Distributor) Novi, MI
Stag Enterprise, Inc. (Distributor) Ball Ground, GA
Surebond, Inc. (Manufacturer) Schaumburg, IL
Textile Rubber & Chemical Company (Manufacturer) Dalton, GA
TVM, Inc. (Manufacturer) Johnstown, PA
Tyco Adhesives (Manufacturer) Norwood, MA

U.S. Adhesives (Manufacturer)Chicago, IL
W.J. Ruscoe Company (Manuf. & Service) Akron, OH
H.J. Calo Company: 1600 Stewart Ave., Westbury, NY 11590 • Phone: 1.866.300.CALO • Fax: 516.832.7127 • Email: sales@jhcalo.com

Table 1.2 Examples of PBX compositions, where HMX is cyclotetramethylenetetranitramine (Octogen), HNS is hexanitrostilbene, PETN is pentaerythritol tetranitrate, RDX is cyclotrimethylenetrinitramine (Hexogen) and TATB is 1,3,5-triamino-2,4,6-trinitrobenzene

Explosive	Binder and plasticizer
HMX	Acetyl-formyl-2,2-dinitropropanol (DNPAF) and polyurethane
HMX	Cariflex (thermoplastic elastomer)
HMX	Hydroxy-terminated polybutadiene (polyurethane)
HMX	Hydroxy-terminated polyester
HMX	Kraton (block copolymer of styrene and ethylene–butylene)
HMX	Nylon (polyamide)
HMX	Polyester resin–styrene
HMX	Polyethylene
HMX	Polyurethane
HMX	Poly(vinyl) alcohol
HMX	Poly(vinyl) butyral resin
HMX	Teflon (polytetrafluoroethylene)
HMX	Viton (fluoroelastomer)
HNS	Teflon (polytetrafluoroethylene)
PETN	Butyl rubber with acetyl tributylcitrate
PETN	Epoxy resin–diethylenetriamine
PETN	Kraton (block copolymer of styrene and ethylene–butylene)
PETN	Latex with bis-(2-ethylhexyl adipate)
PETN	Nylon (polyamide)
PETN	Polyester and styrene copolymer
PETN	Poly(ethyl acrylate) with dibutyl phthalate
PETN	Silicone rubber
PETN	Viton (fluoroelastomer)
PETN	Teflon (polytetrafluoroethylene)
RDX	Epoxy ether
RDX	Exon (polychlorotrifluoroethylene/vinylidine chloride)
RDX	Hydroxy-terminated polybutadiene (polyurethane)
RDX	Kel-F (polychlorotrifluoroethylene)
RDX	Nylon (polyamide)
RDX	Nylon and aluminium
RDX	Nitro-fluoroalkyl epoxides
RDX	Polyacrylate and para¶n
RDX	Polyamide resin
RDX	Polyisobutylene/Teflon (polytetrafluoroethylene)
RDX	Polyester
RDX	Polystyrene
RDX	Teflon (polytetrafluoroethylene)
TATB/HMX	Kraton (block copolymer of styrene and ethylene–butylene)

Examples of energetic polymers and energetic plasticizers under investigation are presented in Tables 1.3 and 1.4, respectively.

Chemical Information
Chemical Name: Styrene/Butadiene copolymer
CAS Registry Number: 009003-55-8
Synonyms: Styrene/Butadiene copolymers; Benzene, ethenyl-, polymer with 1,3-butadiene; Ethenylbenzene polymer with 1,3-butadiene (9CI); Styrene polymer with 1,3-butadiene
Information from other National Library of Medicine databases
Health Studies: ***No information available in HSDB at this time***
Toxicity Information:
Search TOXNET
Chemical Information: Search ChemIDplus

Products that contain this ingredient

Brand	Category	Form	Percent
Liquid Nails Adhesive, For Small Projects, Original Formula	Home inside	tube	1-5
Henry 816 Wood Flooring Adhesive	Home maintenance	paste	
Hartco Do It Yourself Floor Adhesive	Home maintenance	paste	10-15
Liquid Nails Adhesive, Heavy Duty Construction and Remodeling, Ext/Int.	Home maintenance	caulk tube	5-10
Liquid Nails for Projects and Construction	Home maintenance	paste	1-5
Henry 377 Carpet Pad Adhesive	Home maintenance	liquid	
Liquid Nails Paneling and Molding Adhesive (Interior)	Home maintenance	caulk tube	1-5

look here: http://www.zeon.co.jp/business_e/enterprise/latex/#b