Improvised PETN



[Agent Orange * (C) 1993 cTs]

Warning

There may be certain federal, state or local laws which prohibit the possesion or manufacture of certain substances mentioned herein. Severe penalities may be prescribed for violation of such laws. Be warned! The procedures to be followed in this manual and the resulting end-product are extremly dangerous. Whenever dealing with high explosives, special precautions should be followed in accordance with industry standards for experimentation and production of high explosives. Failure to strictly such industry standards may result in harm to life or limb.

Therefore, the author disclaim any liability from any damages or injuries of any type that a reader or user of information contained within this manual may encounter from the use of said information. Use this manual and any endproduct or by-product at your own risk.

Preface

The information in this manual serves as an introduction to the organic explosive PETN, pentaerythritol tetranitrate. Included are important physicaland chemical properties with which the reader should be familiar beforeproceeding to the section that describes in detail one of several provenmethods for the manufacture of high-grade PETN. Finally it should bementioned that the chemicals listed in this text are hazardous if used improperly or handled carelessly. The reader is urged to adopt a "think beforeyou do" attitude, pay attention to details, and use safe laboratory equipment and procedures.

PETN - Its Discovery and Properties

The high explosive pentaerythritol tetranitrate was first synthesized in 1894 at the Rheinish-Westfälische Sprengstoff Company in Germany by a chemist researching the nitration process of pentaerythritol in a high concentration fnitric acid. The resulting explosive was collected, tested, and found to have a very fast rate of detonation, high density, and good chemical stability, making it an ideal substance for military use. Unfortunately, at that time there was no means available to mass-produce the raw ingredient pentaerythritol. PETN thus remained a laboratory interest until World War II, when it was used in ammunition, bombs, and other fragmention devices. During this time, Germany produced as much as 1,440 tons of PETN per month, with the USA and the USSR following suit.

Still manufactured on a large scale, PETN remains one of the most powerful conventional explosives ever developed, rivaled only by HMX and RDX. The military, as might be expected, is the largest user of PETN, with annual purchasing well over 2,000 tons. Nonmilitary use is primarily limited to demolition work and as a booster material for secondary explosives such as ANFO, dynamite, etc.

Physical and Chemical Properties

PETN - $C_5H_8O_{12}N_4$ - is a white crystalline substance that feels powdery to the touch. In its pure form, PETN melts at 141.3 degrees centigrade.

The boiling point is:	The molecular structure is:
160 °C at 2mm Hg 180 °C at 50mm Hg 200 °C at 760mm Hg (standard atmosphere)	$\begin{array}{c} 0 \\ 0 \\ 0 \\ 0 \\ 0 \\ 0 \\ 0 \end{array} N - 0 - CH_2 \\ H_2 C - 0 - N \\ 0 \\ 0 \\ 0 \\ 0 \\ 0 \end{array} $

The calculated nitrogen content is as follows:

Element	# Atoms x Atomic Wt.	
Carbon	5 x 12.011	= 60.055
Hydrogen	8 x 1.00794	= 8.064
Oxygen	12 x 15.9994	= 191.993
Nitrogen	4 x 14.0067	= 56.027
	Total	= 316.139

<u>56.027</u> 316.139 (100) = 17.722% Nitrogen

Correspond with the military specification MIL-P-387 for PETN:

Melting Point	140.0 °C
Nitrogen Content	17.5% Minimum
Substances Insoluble in Acetone	.10% Maximum
Acid or Alkali Content	.003% Maximum
Vacuum Stability at 120 °C	5ml of gas / 20 hours
Overall Purity	98.7%

If we examine the ultraviolet absorption spectrum of a single PETN crystal, we find a narrow band in the 280æm area. Because ordinary sunlight emits UV in the same range, PETN should not be exposed to the sun for a long period of time as damage in the form of cracks in the crystals will appear and render the PETN useless.

PETN Manufacture

PETN can be made in a number of different ways, but the overall process is always the same and consists of the following steps:

- 1. *Nitration.* Adding pentaerythritol to a given amount of nitric acid for a specific time at a specific temperature to form crude PETN.
- 2. Washing. The crude PETN is rinsed with distilled water until free of acid, filtered, and throughly dried.
- 3. *Recrystallization.* The dry PETN is dissolved in heated acetone, purified with a neutralizing agent, and then poured into cold water where the purified PETN is precipitated.
- 4. *Washing.* The pure PETN is again rinsed with distilled water to remove remaining traces of acid and acetone, filtered, and dried to a 10 percent water content. The PETN is now ready for use.

We will now proceed to the actual method for making high-grade PETN on a laboratory scale (larger amounts can be made by increasing the size of the equipment) that will yield about 500ml per batch. The amount of chemicals used can vary if one wishes to produce more or less PETN per batch. However, the proportions and the temperatures are exact and must not be changed under any circumstances. We will begin with a list of equipment and chemicals needed. Then each of the steps mentioned above will be detailed and fully discussed.

Hardware Required

- 1. Stirring hotplate, with at least a 10" x 10" surface area. This type of hotplate will heat solutions and stir them by spinning a glass-coated bar magnet that is dropped into the solution being treated. The temp. and mixing rate are both adjustable by turning two dials on the face of the machine.
- 2. Glass coated magnetic stir bar 2.75" long
- 3. One 4,000ml Pyrex beaker of standard configuration, with graduation
- 4. One 1,000ml Pyrex beaker, tall form with graduation
- 5. One thermometer graduated in degrees centigrade
- 6. One thermometer clip to hold thermometer to the beaker
- 7. A few glass stirring rods
- 8. Pack of fine-grade filter paper (grade: 230, 33cm diameter)
- 9. Glass funnel
- 10. Plastic bucket: one or two
- 11. Wood or plastic cookspoon (used to scoop PETN crystals)
- 12. Several pans for drying (disposable pizza pans work well)
- 13. One graduated cylinder used for measurement. A 100 or 200ml cylinder will do.
- 14. Several paper cups
- 15. Full face shield to protect eyes and face while working with acid.
- 16. Plastic or rubber gloves

Chemicals Required

The amount of chemicals listed is just enough to make one 500ml lot of PETN. The reader will have to decide how much to purchase if he decides to vary the amount of PETN produced.

- 1. 600ml nitric acid with a purity of 98 percent. (Don't use 70 percent or 90 percent only 98 percent.)
- 2. 250ml pentaerythritol, reagent grade
- 3. 1,500ml acetone, reagent grade
- 4. One teaspoon sodium carbonate powder, reagent grade
- 5. 800ml distilled water
- 6. 30 to 40 gallons of tap water for rinsing. Should tap water be hard or contain chlorine, distilled water must be used.
- 7. One vial of red litmus paper, used to test pH level alkaline range 7 to 14
- 8. One vial of blue litmus paper, used to test pH level in acid range 0 to 7
- 9. Small bag of crushed ice
- 10. Optional: Full range 0 to 14 pH indicator strips. These are helpful in determining the pH of a solution, but litmus will suffice.

All of the hardware and chemicals listed are readily available from major scientific supply companies. Consult the yellow pages and don't be afraid to shop around for the best prices.

Step One: Nitration

- (a) Take the 100ml tall form beaker and fill it with 600ml of nitric acid. Be sure to wear your face shield when working with acid.
- (b) Place the tall form beaker with the acid inside of the larger 4000ml beaker and center it.
- (c) Fill the larger beaker with crushed ice up to the same level as the acid in the tall form beaker. Now pour tap water over the ice until the level reaches the 700ml mark on the tall form beaker. Do not let any ice or water fall into the acid.
- (d) Carefully place the stir bar into the beaker of acid.
- (e) Using the thermometer clip, attach the thermometer to the rim of the tall form beaker and adjust it so that the bulb (the part with the mercury) falls just slightly below the surface of the acid. Be sure the numbers can be clearly read.
- (f) With two hands, carefully lift the whole assembly and place it on the stirring hotplate and move it around so that the stir bar is centered inside the tall form beaker. All controls on the hotplate should be in the off position.
- (g) Using the graduated cylinder, measure out 250ml of pentaerythritol and pour this into the paper cups (you will most likely need two paper cups).

During the manufacture process, acetone is used to purify crude PETN and to safely remove crystals that may have condensed on various pieces of laboratory equipment. Because acetone readily dissolves PETN, a table of solubility is given below as a reference.

Amount of PETN dissolved 100	Acetone Soluble in Water						
grams solution	55%	70%	80%	90%	92%		
(grams)	Temperature of Solution in °C						
5	1 gr @41	41.5	22				
10	4 gr @62	54.5	38.5	15	10		
15		62	48	24.5	20.5		
20			54	34.5	29		
25			59	41.5	34		
30			63	46.5	40.5		
35				51.5	45		
40				55	50		
45				58.5	54		
50				61.5	57.5		
55					60.5		
60					62,5		

The above table indicates a low solubility of PETN in water and this helps to explain the low toxicity of PETN on the human body. Handling or inhaling the dust in small amounts causes almost no effect. From a toxic point of view, PETN, unlike nitroglycerine, TNT, or dynamite, is safe to work with.

Explosive Properties

Pure PETN, when heated above its melting point, explodes at 205-225 °C.

Initiation	NO ₂	NO	N ₂ O	Ν	CO ₂	CO	Н	O ₂
Detonation	-	5.3	-	22.8	37.0	26.7	6.8	1.4
Impact	-	24.3	5.3	9.4	19.1	35.4	6.5	-
Thermal 210 °C	12.0	47.6	9.5	1.6	6.3	21.0	2.0	-

A Table of Gaseous Products Released During Decomposition of PETN

Heat of Explosion: 1,530 Kcal/Kg. Volume of Gases: 768 L/Kg. Temperature: 4,230 °C

The rate of detonation of a single crystal of PETN, the density of which is 1.77g/cubic centimeter, is 8,500 meters/second. In order to get a cartridge filled with PETN powder to detonate at the same rate, two things must be done:

- 1. Pure, dry PETN must be mixed by weight with 10 percent water. This will drive the air out and allow the detonation wave to travel rapidly from one crystal to the next. The water also makes the PETN safer to work with, as dry PETN explodes when subjected to a slight impact.
- 2. The PETN must be loaded into the cartridge under high pressure so that the density of the powder approaches the crystal density. The ideal loading density of a cartridge containing PETN should be in the range of 1.72 to 1.73g/cubic centimeter. At this density, the 8,500 meter/second detonation rate can be achieved and the maximum explosive power of PETN obtained. It should be mentioned, however, that even if a cartridge is only packed by hand, a very powerful explosive device will still result. The loading density of a cylindrical cartridge can be determined by using the following equations:

Pressure = Force

Volume = Area x Length

Loading Density = <u>Mass (amount of PETN)</u> Volume of Cartridge

We are now ready to continue. Be sure the work area has good ventilation, and wear your face shield to avoid acid burns in case any beakers are accidently knocked over.

- (h) Turn on the stirrer and adjust until a smooth rapid motion occurs in the acid. The heater will be in the "off" position during the whole nitration process. If you are not using a stir bar, the stirring must be continuously done by hand with a glass rod.
- (i) Look at the thermometer. When the temperature falls to 10°C, very slowly sprinkle in some of the pentaerythritol by squeezing the rim of the cup to form a spout and shake lightly. As soon as the temperature starts to rise, stop adding. Look to see where the temperature stops. If it is below 20°C, then continue adding the pentaerythritol and stopping whenever the thermometer reads 20°C. Allow the whole mixture to cool back to 10°C and then start sprinkling in the pentaerythritol until 20°C is again obtained. Continue adding in this manner until all 250ml of pentaerythritol has been added. When the temperature again falls to 10°C, remove the thermometer and the stir bar. Take the small beaker out of the larger one and set it aside for forty-five minutes. The stir switch can be turned off and the ice-water mixture thrown out.

Important: Never let the temperature of the mixture rise above 25°C during this operation.

Step Two: First Washing

- (a) After forty-five minutes have elapsed, the crude PETN will have settled to the bottom of the beaker, as a thick yellow-white sludge, with a layer of acid on top. Pick up the beaker and slowly pour off the top layer of acid, being careful not to disturb the PETN sludge. When finished, set the beaker aside and dispose of the waste acid.
- (b) Fill a plastic bucket with two gallons of water. Use distilled water if your tap water is hard or chemically treated.
- (c) Pour the beaker of PETN into the bucket and scoop out any sludge with a wood cookspoon. Using a glass rod, stir the contents of the bucket vigorously for ten seconds and then wait for the PETN to settle. If a foam has developed on the surface of the water, be sure to agitate the surface with your stirring rod. The foam, which is just air trapped in PETN, will settle to the bottom of the bucket.
- (d) After the contents have settled, take a strip of blue litmus paper and dip it into the bucket. You will notice that the litmus paper turns red, indicating the presence of any acid. This acid must be removed, so take the bucket and slowly pour off the layer of water. Be careful not to pour off any PETN by accident.
- (e) Take two gallons of fresh water and pour it into the bucket. As before, stir to break up the foam and then allow the contents so settle again. Using a new blue litmus paper, test for the presence of any acid. Should acid be present, continue the cycle of pouring off the water adding fresh water, stirring, settling, and testing with litmus paper.
- (f) After several wash cycles, you will find that the litmus paper no longer turns red but instead remains blue, indicating that the rinse water is now neutral. Wash the PETN one more time, let settle, and pour off the water.
- (g) Place a filter paper into the glass funnel. Scoop the PETN from the bottom of the bucket and put it into the filter. The filter paper and funnel serve to allow the water to drip away, thus allowing the PETN to dry faster. Allow the PETN to filter for at least thirty minutes.
- (h) After filtering, take the PETN and spread it out as a thin layer in the drying pans. The PETN must be allowed to dry completely. When dry, it will be a white crystalline powder. The drying might take several days.
- (i) Clean and dry the beakers, stir bar, and thermometer, as they will be used again during the next step.
- (j) After the PETN has dried, place it in any handy container other than the beakers and set it aside.

Step Three: Recrystallization

- (a) Take one teaspoon of sodium carbonate and place it in the tall form beaker. Add 800ml of distilled water and stir until completely dissolved. This, your neutralizing solution, will be used shortly.
- (b) Fill the 4,000ml beaker with 1,500ml of acetone. Put in the stir bar and thermometer and place on top of the hotplate. Turn the stir dial to a medium speed and turn on the heat. The temperature of the acetone will rise and must be held between 50°C and 60°C. Do not let the temperature stray out of this range during the next steps. Aim for a temperature of 55°C. Heated acetone is highly flammable use caution!
- (c) Take two heaping tablespoons of dry PETN, add it to the acetone, and let it dissolve completely. The temperature will now drop slightly, but if it is still above 50°C, take two more tablespoons of PETN, add it to the acetone, and let it dissolve. Keep up this method of adding two tablespoons of PETN and letting it dissolve until the temperature reaches 50°C, stop adding PETN and let the temperature rise to just under 60°C. Then begin adding PETN again two spoons at a time and allowing it to dissolve until all the PETN has dissolved
- (d) With all the PETN now dissolved in acetone and the temperature held at 55°C, we will begin neutralizing the solution.
- (e) Have both the blue and red litmus papers ready
- (f) Fill the graduated cylinder with the sodium carbonate solution that was prepared in the tall form beaker.
- (g) Keeping the contents of the large beaker in the 50ø to 60°C range, add to it 10ml of the sodium carbonate solution (SCS). Insert a blue litmus paper. If the paper turns red, add 10ml more of the SCS, and test again with the litmus paper. Keep up this process of adding 10ml of SCS at a time, until the litmus paper remains blue. When this happens, add a final 10ml of SCS. Test with both the red and blue litmus paper. If neither paper changes color, the solution is neutral. Turn off the hotplate.
- (h) Immediately fill a bucket with two gallons of cold water. Pour the contents of the large beaker into the bucket and stir. The PETN will now precipitate with its new fine crystalline structure.
- (i) Test the water in the bucket with blue and red litmus paper. If the instructions in this text were followed carefully, there will be no reactions. If they do react, then an error was made and salvaging the PETN will be beyond the scope of this text. You will have to try again.

Step Four: Second Washing.

- (a) This washing process is similar to Step Two, only we will be removing acetone instead of acid from the PETN. When the contents in the bucket (including the foam) have settled, pour off the water.
- (b) Add two gallons of fresh water, stir, and allow to settle. If you smell the water, you will notice a strong acetone odor. Continue the wash cycle until the acetone odor is no longer apparent and then rinse the PETN two more times to complete the washing.
- (c) After the PETN has settled, pour off the water. Filter the PETN and allow to dry to a 5 to 10 percent moisture content. The finished PETN should not feel wet when a sample is squeezed between your fingers. When the PETN has been dried, it will be ready for use. Store in a cool, dark place.

This concludes the manufacture of PETN.



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