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basic explosive

Note that the word "Basic" in the above title implies that this is the alkaline form of the salt. This is a seldom known of novelty explosive salt, which is very easy to prepare.

Basic Stannous Nitrate, with the formula Sn3(OH)4(NO3)2, is a white crystalline substance which is slowly oxidized upon exposure to air, and partially decomposes in pH neutral water. It is a high explosive, detonating when strongly rubbed, hit with a hammer, or heated above 100degC. Thermal decomposition proceeds at 125degC, resulting in formation of SnO2, nitric oxide, and water.

The salt may result when elemental tin (Sn) foil (not common aluminum foil) is reacted with a solution of copper nitrate to form a precipitate that is a sensitive explosive when dried. When detonated with heat or friction, it gives off sparks. It can also be made by using cold, very dilute nitric acid to dissolve tin, then adding some sodium carbonate (baking soda might work instead), but not enough sodium carbonate to cause to cause the expected precipitation of stannous (tin II) carbonate.

Solutions of tin(II) sulfate can be formed by reaction of copper(II) sulfate with metallic tin. The tin(II) sulfate then reacts with sodium carbonate to form tin(II) carbonate. Alternatively, NaOH or baking soda can be used instead for this preparation. The SnCO3, or Sn(OH)2 if NaOH was used, precipitates out as a solid from the solution, the solid tin carbonate, SnCO3, reacts with dilute nitric acid to form solutions of Sn(NO3)2, which are reasonably stable. However, attempting to evaporate the solutions to dryness results in decomposition, which can in some instances be violent. The decomposition products are mainly SnO2, nitrous oxide, and hydroxylamine, with other oxides of nitrogen also produced.

At the Royal Powder Works at Spandau, Prussia, frequent ignition of the powder at a certain stage of the process led to an examination of the machinery, when it was found that where, at certain parts, bronze pieces which were soldered were in constant contact with the moist powder, the solder was much corroded and in part entirely destroyed, and that in the joints had collected a substance which, on being scraped out with a chisel, exploded with emission of sparks. It was suspected that the formation of this explosive material was in some way connected with the corrosion of the solder, and the subject was referred for investigation to Rudolph Weber, of the School of Technology in Berlin. The main results of his investigation appeared in Journal für Praktische Chemie are given below..

The explosive properties of the substance indicated a probable nitro-compound of one of the solder metals (tin and lead), and as the lead salts are more stable and better understood than those of tin, it was resolved to investigate the latter, in hope of obtaining a similar explosive compound. Experiments on the action of moist potassium nitrate on pure tin led to no result, as no explosive body was formed. Stannous nitrate, Sn(NO3)2, formed by the action of dilute nitric acid on tin, has long been known, but only in solution, as it is decomposed on evaporating. By adding freshly precipitated moist brown stannous oxide to cool nitric acid of specific gravity 1.20, as long as solution occurred, and then cooling the solution to -20°, Weber obtained an abundance of crystals of the composition Sn(NO3)2* (20)H2O. They resemble crystals of potassium chlorate. They cannot be kept, as they liquefy at ordinary temperatures. An insoluble basic salt was obtained by digesting an excess of moist stannous oxide in solution of stannous nitrate, or by adding to a solution of stannous nitrate gradually in small portions, with constant stirring, a quantity of sodium carbonate solution insufficient for complete precipitation.

Thus obtained, the basic salt is a snow-white crystalline powder, which is partially decomposed by water, and slowly oxidized by long exposure to the air, or by heating to 100°. By rapid heating to a higher temperature, as well as by percussion and friction, it explodes violently, giving off a shower of sparks. This compound is also formed when a fine spray of nitric acid (sp. gr. 1.20) is thrown upon a surface of tin or solder. It is also formed when tin or solder is exposed to the action of a solution of copper nitrate, and thus formed presents the properties already described.

In this, then, we have a probable cause of the explosions occurring in the powder works; but the explanation of the formation of the substance is wanting, as potassium nitrate was shown not to give an explosive substance with tin. A thin layer of a mixture of sulphur and potassium nitrate was placed between sheets of tin and copper foil, and allowed to stand, being kept constantly moist. After a time the copper was found to have become coated with sulphide, while the tin was largely converted into the explosive basic nitrate. The conditions are obviously the same as those found in the powder machinery, where bronze and tin solder are constantly in contact with moist gunpowder. The chemical action is probably this: the sulphur of the powder forms, with the copper of the bronze, copper sulphide; this is oxidized to sulphate, which reacts with the niter of the powder, forming potassium sulphate and copper nitrate; the latter, as shown above, then forms with the tin of the solder the explosive basic nitrate, which, being insoluble, gradually collects in the joints, and finally leads to an explosion.

Ammonium Permanganate

(NH4)2MnO4 explodes with a copious cloud of dark brownish-purple smoke. The substance is sensitive to friction, and very easily is set off by flame.

It is not entirely chemically stable, and slowly degrades. It can be stored for about 4 months.

The permanganate ion is much more reactive oxidizer than perchlorate, which is why this salt is so sensitive and chemically unstable.

Like ammonium perchlorate, ammonium permanganate is another ammonium salt that is not very soluble (most ammonium salts are very soluble). Unfortunately, potassium permanganate is not very soluble either, so this is not advantageous for making it.

Preparation 1 (the below has not been tested)

Sodium permanganate can be made by dissolving a manganese compound, such as MnCl2 or MnO2 (but do not use potassium permanganate for this procedure), in bleach. The reaction turns purple. Add extra manganese compound to be sure that all the bleach has reacted. After thorough mixing for 30 minutes, a test is to add a little piece of manganese dioxide, which then should not dissolve, indicating all the hypochlorite in the bleach has been reacted.

Separately, mix a small quantity of ammonium sulfate (or ammonium nitrate or ammonium chloride), but only add a small quantity, with some ammonia. Too much ammonium sulfate will make the reaction acidic, which is bad since that will make the permanganate oxidize the ammonia. If you have a pH strip, the ammonia mixture with the ammonium sulfate should be preferably neutral, or slightly basic. Be sure all the ammonium sulfate is dissolved in the ammonia, some extra water might be added to help dissolve it.

Add the second mixture to the first, then lower the temperature to 10degC until purple crystals appear. Use a spoon to scoop out the crystals, then filter out the crystals using coffee filter paper, washing with with a little water. The crystals are ammonium permanganate. Note that no potassium compound can be used in the above preparation, since the potassium permanganate would then solidify out.

If there is trouble crystallizing out the ammonium permanganate, some ammonia (the concentrated type) can be added to the solution, which should help because of the "common ion effect". The solution should not be cooled too long, otherwise sodium sulfate (salt) will begin to solidify out with the ammonium permanganate. Actually, ammonium chloride or ammonium nitrate are better than ammonium sulfate to use in this procedure, because the sodium sulfate is less soluble than sodium chloride.

Preparation 2 (has been tested)

20 grams of potassium permanganate is dissolved into 200ml of boiling water, to this mix is added 250g of ammonium nitrate and allowed to dissolve. The solution is poured off into another beaker to leave any undissolved or decomposed products in the bottom. Chill the solution to 10°C to precipitate the NH4MnO4, the solution can be poured off to retrieve the reddish-purple crystals. Place the product onto absorbent paper and leave to dry at room temperature. Excess ammonium nitrate must be used, otherwise it is difficult to preferentially precipitate out only the ammonium permanganate, since it is not much less soluble than potassium permanganate.

For preparing solutions of permanganic acid:



The reaction is slow. The lead sulfate solidifies out, then is removed. To the remaining solution, add baking soda to make more alkaline (otherwise HMnO4 is a reactive oxidizer), then add the ammonia. The sodium ions are not as problematic as potassium ions, since the sodium permanganate is much more soluble.

Aqueous solutions of permanganic acid partially decompose when heated to 40degC, and completely decompose if boiled. The solutions slowly degrade at room temperature, especially above 20degC. The decomposition products are hydrated MnO2 and oxygen gas. If the water is allowed to evaporate out, the oxygen that forms from decomposition contains traces of ozone, giving it a distinct odor. The solutions slowly oxidize elemental sulfur after a few days to dilute sulfuric acid.

Tetramine Copper Nitrate (TACN)



As you can see in the first link, moist crystals of tetraamine copper nitrate (TACN) in a glass tube under flame first seem to melt, then instantly decompose in an explosive exothermic reaction (not actually a detonation as the thin glass does not shatter)

TACN deflagrating. The salt easily ignites from the flame of a butane torch. TACN rapidly deflagrates with greenish-blue flare, but there does not seem to be the instant "poof" which is seen with the deflagration of most other primaries:

I think I read somewhere that tetraamine copper nitrate is actually fairly insensitive to mechanical impact, that reliable detonation of this salt requires another primary. TACN is supposedly also very hygroscopic, and if not free from moisture can be difficult or impossible to ignite. If preparing the salt for the purpose of deflagration/detonation, pure methanol should be used as the solvent, with dry ammonia gas bubbled in, so that the product will be free from traces of water.

"I can light it with difficulty. If I put a flame of a torch on it, then it first melts and then I get kind of 'poof' with a small cloud of black smoke. It does not really explode, it is more like nitrated cotton-wool, but with a less visible flame." (woelen)

"the problem (from an energetic aspect) is going to be hydration." (Axt)

"Yesterday I tried to make TACN by dissolving 2g pure copper and 5g ammonium nitrate in 10ml 10% ammonia solution. After 1 day the solution turned blue and bright blue crystals formed at the copper. I filtered the solution and ended up with 0.7g with crystals. I dried them well. Then I tried to detonate it. It got a very little "poof sound" and some smoke came. I also tried to use CuO insted of pure copper. I ended up with some very dark blue\purple crystals after I filtrated it. When I heated them up, nothing happend." (mikkello, who is from Norway)

"TACN is easily dehydrated in dry acetone, forming a stable dull-violet powder. Put the crystals in a beaker filled with dry acetone, crush them carefully, stirr and let it sit for a short time. The acetone will take up the water of crystalization. Pour off the acetone (which now contains water) and wash with another portion. Repeat until the color of the crystals no longer changes. Of course this works best with dry reagent grade acetone, but I tried it with the crappy commercial stuff (which contains a considerable amount of water) and even that worked fine. Then decant and press dry between filter paper. Let the crystals dry in warm air until all acetone has evaporated. Then do a final drying over CaCl2. During the acetone wash you will notice the chunky crystals change color from deep blue to a light violet and turn into a fine powder. This powder is stable in air and quite powerful. Its quite sensitive too - smack with a hammer and it goes boom. This is the only useful form of TACN. The hydrated form cannot be stored because it readily draws CO2 from air. It is also very insensitive and weak. I observed the same effect with TACP: The hydrated form is useles. It can hardly be ignited with a match and is very insensitive to impact. Remove the water of crystalization and you get a much more sensitive and powerful substance. Of course these materials must be stored in a well stoppered bottle to exclude moisture. From the hydrated form of TACN you will hardly get anything more impressive than a pop sound and some smoke." (edited compilation of quotes from Taoisearch)

TACN should actually be considered a primary explosive, although it seems less sensitive than many other primaries, but it can be difficult to reliably initiate. If free from water, it will deflagrate if subjected to the intense flame of a butane torch. Like other primaries, the deflagration likely will lead to detonation if the mass of TACN is more than a few grams and there is a small degree of confinement, such as in a little rolled up paper tube.

The main problem seems to be hydration. Although the crystals of TACN may appear dry, it is quite likely that molecules of water become incorporated into the solid crystal structure. CuSO4 is known to form such a solid hydrate CuSO4* (5)H2O. For reliable use as an explosive, TACN needs to be prepared without water, and be kept away from long exposures to air.

Anhydrous Cu(NO3)2 is apparently very reactive:

"at temperatures below -5", the reaction of diethyl ether with copper(II) nitrate yields exclusively gaseous ethyl nitrite and complexed acetaldehyde. At higher temperatures, the acetaldehyde is further oxidised to acetate, with liberation of gaseous NO and NO2"

Anhydrous copper(II) nitrate as an oxidising agent. L. C. Coard and R. E. Powell J. Chem. Soc. A, 1967, 296-297

Sulfur Nitride

The precursors for this compound are easily available, but the synthesis takes some time and effort. The S4N4 molecule is actually a highly skewed octagonal aromatic ring.

Tetrasulfur Tetranitride (S4N4)

DETONATION VELOCITY: 5.4 - 5.6 km/sec

SENSITIVITY: Very sensitive to friction great care would be needed to produce this compound. Small quantities (less than one gram) deflagrate with a poof and larger sizes will detonate.

TetraSulfur TetraNitride crystallizes in orange-red needles which melt at 178degC. Nitrogen sulfide is a dangerous compound to make. It is sensitive to friction and heat. About as powerful as lead picrate. This compound has slightly less brilliance than mercury fulminate and is more susceptible to friction. Storage stability is good for plain nitrogen sulfide. S4N4 also forms a complementary mix with Tetramine Copper Perchlorate. Mixtures of S4N4 and chlorates are less stable, and can spontaneously explode in storage, especially above 50 degrees C. One recommended filler with this primary explosive is nitrogen sulfide 78%, completely dry potassium chlorate 20%, and 2% baking soda, which acts as a stabilizer.

This preparation is fairly simple one, with a small assortment of easily obtained materials.

PRODUCTION

Place 100 grams of finely powdered sulfur (brimstone: garden supply store, pharmacy, industrial chemical supply) is placed in a tall narrow flask or narrow necked bottle equipped with a two hole stopper and placed in a frying pan filled with oil and heated until the sulfur melts (215 degrees C., 420 degrees F.). In this place a hose from the chlorine gas generator. This generator is a gallon jar with either liquid laundry bleach (5.25% Sodium hypochlorite aqueous solution) or 31% hydrochloric (Muriatic acid, swimming pool supply), to the bleach (total 1.2 gallons) is added in small portions sodium bisulfate ("Saniflush": bathroom cleaners, sodium acid sulfate, swimming pool additive). This generation with the bleach/ bisulfite generator should have the bleach split into three equal amounts and reacted with the bisulfite one at a time. The second and third 4 gallon refill should be done only after the greenish-yellow gas is no longer generated by new sodium bisulfate additions. The spent bleach is poured out of the gallonjug. The second or thirds fill are poured into the jug the process repeated until all three 2/3s gallon bleach solutions are reacted and the chlorine bubbled through the molten sulfur. To 255 grams hydrochloric acid is added 53 grams manganese dioxide (black manganese oxide: dry cell battery mfg., phosphating solutions, steel mfg.) in small portions. This is done in small additions until all the manganese dioxide is dissolved and the chlorine has stopped it's bubbling.

CAUTION: Chlorine gas is corrosive and very poisonous. Avoid breathing in fumes and use adequate ventilation.

Immediately after the addition and beginning chlorine generation place a one hole stopper to which some stainless steel or plastic (heat resistant) tubing has been inserted in the hole. The other end of this hose directs the chlorine gas generated through the two hole stopper into the bottom of the now molten sulfur. The other hole of the two hole stopper has a hose inserted just through the stopper. The end of this hose is placed into a flask or narrow necked bottle cooled by a salted ice bath. This sulfur will begin to absorb the chlorine generated. This reaction forming sulfur dichloride. A total of 42 grams of chlorine need to be absorbed by the sulfur. As this chlorine is dissolved sulfur dichloride will begin to form. Sulfur is very soluble in sulfur chlorides and will begin to be dissolved in the chloride already formed. This sulfur chloride will vaporize and collect in the bottle chilled by the salted ice bath. This is done until the temperature drops and begins to boil. Continue to pass the chlorine gas through the liquid. After all the chlorine has gone through the sulfur heat until the sulfur liquid no longer boils. Heat for another ten minutes and allow to cool. The last flask should have caught most of the sulfur dichloride liquid. Take the mixture off the heat and allow to cool. Dissolve 212 gram of this liquid in 1.7kg benzene (common industrial solvent).

CAUTION: Sulfur dichloride (Sulfur chloride) is a pungent oily liquid. All contact should be avoided, since it is highly corrosive. All steps of this process should be carried out with good ventilation. Benzene is a very dangerous liquid. Contact with the skin, breathing of the vapors are dangerous and should be avoided. Great care should be used when handling this known carcinogen and highly flammable. You may wish to use toluene or chloroform as the solvent instead.

Filter this solution through a paper coffee filter. This filtering should remove nearly all the sulfur. The remaining liquid should have no solids in it. Then ammonia gas generator is set up and ammonia gas is bubbled through the solution. The ammonia generator (ammonium nitrate/lye) is described in TACC section of the primary explosive section of this book. A dark brown powdery powder will collect in the bottom as the ammonia bubbles through the liquid. Keep bubbling the ammonia gas through the solution. Until this brown powder dissolves in the solution and a orange-yellow color is observed. Flocculent ammonia chloride crystals are seen in the liquid. Warm the benzene until it boils. Filter immediately through a filter with 200g fresh benzene. Add this benzene wash to the liquid just filtered (filtrate). Let this liquid evaporate until a mushy crystalline mass remains and filter. Let these crystals dry. These golden yellow to orange-red crystals are nitrogen sulfide.

CAUTION: This compound is friction, flame and shock sensitive. Handle with great caution.

Here is the chemical equation for the last step:



Kommentare