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Diazodinitrophenol

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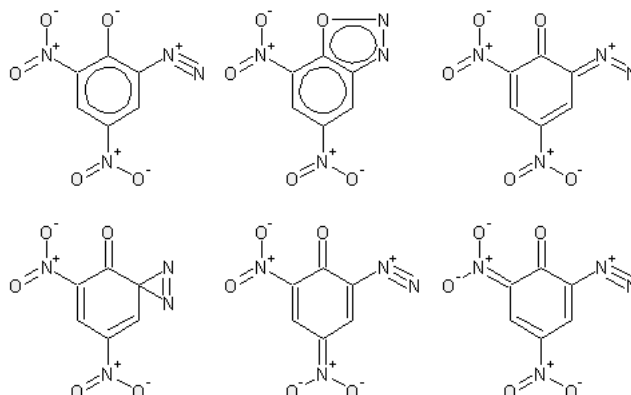
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Diazodinitrophenol, also called dinitrodiazophenol, Dinol, DDNP or dinitrobenzene dinitoxide, is a primary explosive. It is the first diazo compound obtained and the only diazonium salt used in pyrotechnics. It was obtained by Griess in 1858 and a slightly modified method used by him is still used today. DDNP is obtained by diazotization of picramic acid obtained from picric acid by reduction with sodium sulfide. DDNP does not decompose under the influence of concentrated acids, but it decomposes very easily under the influence of alkalis, even 10% sodium hydroxide solution. This solution can be used to clean dishes after working with diazodinitrophenol and destroy its residues. DDNP well in concentrated HCl, acetone, glacial acetic acid, nitrobenzene, aniline, pyridine, nitroglycerin and EGDN, moderately in methanol, ethanol, ethyl acetate, poorly in gasoline and chloroform, almost insoluble in water. DDNP is more stable than mercury fulminate, but less stable than lead azide. The stimulating power of diazodinitrophenol is similar to that of lead azide, for example, 0.115 g of DDNP (equivalent to 0.12 g of lead azide) is needed to ignite picric acid. For MWI, DDNP has exceptional parameters. It has a large volume of gases produced - 876 l/kg. It is not ironed "dead" even under

sky-high pressures of 9,000 atmospheres. The result of the Trauzla test using 1 g of the substance is 25 cm³ (for comparison, lead azide 7 cm³, mercury fulminate 8 cm³). Compressed detonation speed of 6000 m/s at a density of 1.5 g/cm³. For DDNP, the crystal size - sensitivity relationship is opposite to that for most MWIs, i.e. the larger the crystals, the lower the sensitivity.

Despite the high crystal density of 1.63 g/cm³ Using standard diazotization conditions, the result is amorphous and essentially unusable for pyrotechnic purposes brown product with a bulk density of 0.27 g/cm³. By using diazotization at an elevated temperature (approx. 35°C) and a very slow reaction, it is possible to obtain crystalline with a bulk density of 0.86 g/cm³.

Chemically, DDNP is a very interesting compound. Formally, it is an internal diazonium salt (6-diazonium 2,4-dinitrophenolate). However, due to its larger size, the equivalent structure with two aromatic rings prevails. DDNP has an intense color due to the participation of resonance structures containing pi quinoid rings. Some of the resonance structures of DDNP are shown below:



Synthesis of sodium salt of picramic acid

Reagents

- Picric acid $C_6H_3N_3O_7$
- Sodium Na sulfide $2S \cdot 9 H_2O$
- Sodium hydroxide NaOH
- Sodium chloride NaCl

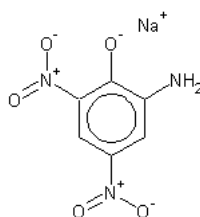
Where to get it?

Unfortunately, none of these reagents except salt is available in grocery stores, construction stores, etc., and even if they are, they are very contaminated. I also do not recommend using NaOH in the form of "Mole". The best sources are mail order or purchases from chemical stores. Picric acid must be prepared according to [recipe in this article](#), and if you buy a solution of picric acid, first evaporate it almost to dryness or take an appropriately calculated amount of the solution. If you don't have sodium sulfide, you can use hydrogen sulfide solution (more on this later)

Equipment

- Dropper
- Beaker
- Magnetic or mechanical stirrer (you can also use an aquarium pump as a stirrer - you blow air into the solution) Large vessel for a cooling bath
- Thermometer
- Ew. equipment for obtaining hydrogen sulfide

The first stage of the synthesis is to obtain the sodium salt of picramic acid. It is sodium 2-amino-4,6-dinitrophenolate:



Sodium picramate is obtained by reducing picric acid with sodium sulfide. First, prepare a solution of sodium picrate in water. For this purpose, he dissolves picric acid and 7.5 g (approximately 5.4 ml) of a 35% sodium hydroxide solution in 450 ml of distilled water. Alternatively, we can first dissolve 2.6 g of sodium hydroxide in 450 ml and then add 7.5 g of picric acid. Now, using a dropping funnel, we add a solution of 30 g of sodium sulfide (in hydrated form) in 75 ml of water, which should not contain any solids, if it does contain them, we filter the solution through a cloth, filter, coffee filter or cotton wool. Now we start the actual reaction: add a solution of 150 g of sodium sulfide to water dropwise from the addition funnel (possibly from a beaker in portions, but you need to be sensitive) and, in parallel, add 95.5 g of powdered picric acid in portions. We add the reagents simultaneously so that the temperature inside the beaker is approximately 55°C. It should not exceed 65°C. Stir vigorously while adding the reagents! It is best to construct some kind of mixer. The reaction is faster if the beaker in which the reaction is carried out is placed in a bowl of cold water. We regulate the temperature by adding warm or cold water to the bowl. When the mixture overheats, add the crushed ice to the beaker. After adding everything, remove the beaker from the bath and stir for another 10 minutes. We leave the beaker with its contents for a day. The next day, we filter off the precipitate at the bottom of the beaker, wash it with 75 ml of table salt solution and then with 20 ml of cold water. The product should be light yellow in color. Receives 80 g of wet sodium salt of picramic acid. It is best to store it in this form to avoid dusting.

If we do not have sodium sulfide, we can prepare it in this way: saturate the measured volume (which suits us) of concentrated NaOH solution (50%) with hydrogen sulfide from the apparatus described in the article about gases. Then we add the same amount of 50% NaOH solution to the obtained solution as we saturated before. We place the beaker with the solution in ice and sodium sulfide crystallizes at the bottom. We filter it and use it for further syntheses. The solutions needed for the synthesis can also be prepared directly. Prepare the first solution with 6.6 ml of 50% NaOH solution with hydrogen sulfide, adding another 6.6 ml of the same NaOH solution and diluting with 80.8 ml of water. The volume of the solution to be saturated is small, so we carry out the analysis in a test tube or take several times the amount of the substance and then take only part of the obtained solution. The second sodium sulfide solution needed is obtained by saturating 32.8 ml of a 50% NaOH solution with hydrogen sulfide, adding another 32.8 ml of a 50% NaOH solution and diluting it with 328.8 ml of water. All work with sodium sulfide and its solutions should be carried out under a fume hood.

The finished sodium salt of picramic acid looks like this:

Sodium picramate dust is a very strong dye. Avoid dust - otherwise everything around will turn yellow! The obtained salt does not need to be converted into the free acid - we use it directly for the synthesis of DDNP.

Synthesis of diazodinitrophenol

Reagents

- Sodium picramate (previously prepared) $C_6H_4N_3O_5Na$
- Sodium nitrite $NaNO_2$
- Hydrochloric acid HCl

Where to get it?

Sodium picramate should be prepared according to the previous recipe. The remaining reagents must be purchased in a chemical store or by mail order, preferably in non-technical grade. Do not confuse sodium with nitrate ($NaNO_3$) - using sodium nitrate will result in you not receiving DDNP.

Equipment

- Magnetic or mechanical stirrer (possibly an aquarium pump) Dropper
- (absolutely necessary!)
- A large dish
- Beaker 800 ml or 1 l
- Thermometer

Now that sodium picramate is ready, we can start preparing DDNP. Weigh 25 g of dry salt or an equivalent amount of wet salt. We can assume that wet salt has a water content of 10%. Place the beaker in a bowl of water so that it is stable. We mount the dropper in a stand or other "holding device", pour 150 ml of 5.5% hydrochloric acid into it (dilute it with 20 ml of concentrated (36%) hydrochloric acid and 130 ml of water). Turn on the mixer and wait until a completely homogeneous suspension is obtained. Then add sodium nitrite in 50 ml of water. Add some hot water to the bowl under the beaker so that the temperature of the liquid in the beaker is approximately 30°C. We start adding the hydrochloric acid solution dropwise - the solution should drip at a rate of 1 drop per 4 seconds. This is crucial to getting the right product. Temperature is equally important - it should be maintained at 25-45° degrees Celsius. We do this by adding cold or hot water to the bowl, but adding cold water is usually only necessary when we add too much hot water. The reaction is able to provide the appropriate amount of heat at this dropwise rate to warm the contents of the beaker to this temperature. Once we have added all the acid, we check the completion of the reaction - if it has ended, the sample taken from the beaker will turn the iodine-starch paper blue. If I don't have it, adding a drop of the mixture from the beaker to a solution containing approx. 1% potassium iodide and approx. 0.1% starch will fulfill the function. Excess nitrous acid escaping from the beaker can smell. After obtaining a positive test, we stir for another 5 minutes to complete the reaction, then turn off the mixer and set aside until the DDNP precipitate settles. Pour off the liquid, add distilled water to the beaker and distribute the DDNP again until a suspension is obtained. Set the liquid aside to decant again, pour off the cold water and pour portions of demineralized water and then filter off the DDNP. We obtain about 20 g of it. The DDNP obtained in this way has the form of small crystals with a bulk density of about 0.63 g/c, they flow perfectly, and are black or dark brown in color. When crushed, the crystals are red or yellow-brown in color. The rate of HCl addition and the temperature are very important to obtain a product of appropriate quality. Through faster diazotation, carried out at a temperature of approx. 5°C, we obtain amorphous product with a density of only 0.27 g/cm3 and much worse stimulating properties.

Security

DDNP, like any initiating explosive, is very dangerous - handle it with extreme caution. Hydrochloric acid and sodium hydroxide are corrosive, while sodium nitrite. Be careful not to acidify the sodium sulfide solution - this will cause the release of highly poisonous hydrogen sulfide.

Usage

DDNP can be used as the first link in a detonating primer. It works best in primers combined with PETN. DDNP should be pressed firmly into the primer to obtain better parameters.

Below are photos related to DDNP. When you hover over an image, its description will appear. Click to enlarge photos.

Special thanks to BLASTER for providing the photos and developing the synthesis. Special thanks to BLASTER for contributing the photos and providing synthetic procedure.

Article

Sources:

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