

Chemical Structure and Accidental Explosion Risk in the Research Laboratory

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Accidental laboratory explosions harm people, laboratory equipment, and infrastructure. Methods to anticipate undesired laboratory explosions therefore are important as new industrialization requires new chemical research, which in turn gives rise to new compounds with untested explosive properties. An explosion is either a deflagration (rapid subsonic autocombustion) or a detonation (instantaneous supersonic decomposition) with solid transforming into gas, high heat, and a shock wave. Previous articles, reviews, and books have treated the subject of explosive chemicals (1–14). Furthermore, the description of accidental explosions have a primary literature of their own and are conveyed often in the form of “safety letters” to magazine and journal editors and occasionally in the form of illustrative Web pages (15). Sometimes, in these media chemical structure is often not (well-) illustrated, and therefore this material is (i) not exceptionally instructional for beginning preparative chemistry students and (ii) not ideal in stimulating laboratory safety discussions.

This concise summary is by no means a substitute for a source such as *Bretherick's Handbook of Reactive Chemical Hazards* (6th edition), which encompasses the entire range of pertinent published literature to 1999 (16). *Bretherick's Handbook* is thus voluminous and perhaps cumbersome, repulsing people who really should be familiar with its important contents. Below, some common organic and inorganic explosion hazards involving the nitro, hydrazido, and azido

groups, the perchlorate ion, peroxides, acetylides, and fulminates are described succinctly. It is hoped that this article can assist those who may be unfamiliar with or daunted by the comprehensiveness of *Bretherick's Handbook*.

Chemical Explosions

An explosion can be defined as “a rapid transformation of a materials system, giving rise to a large emission of gases” leading to mechanical effects (5). In chemical laboratories, these events mainly involve sudden transformations that shatter glassware. Many of the compounds illustrated here are shock sensitive—they may detonate from “abrupt” handling such as scraping or chipping with a spatula. *Awareness of explosion potential of novel compounds may be anticipated from clear illustrations of species involved in previous hazards.* Fires (requiring oxidant, fuel, and initiator) that were nonexplosive are not covered (though of equal or greater importance in laboratory safety), while instances of explosive “burning” are included.

Nitro Group

While all nitrogen oxides may possess some level of explosion risk, nitro compounds, especially aromatic ones are notoriously explosive. Well-known materials such as TNT (17), nitroglycerine (2,4,6-trinitroglycerine), and “C4” (composed of mainly cyclotrimethylene trinitramine) possess nitro or nitroester groups (Figure 1). However, not all dangerous compounds are as *conspicuous* by name or structure as octanitrocubane (18), bearing both extreme nitration and bond strain.

Picric acid and picrates are also trinitroaromatic species (Figure 2)—note that some species contain two picrate moieties per molecule such as dipicryl sulfone and dipicrylamine. Examples of related arene derivatives with trivial names and the same or less extent of nitration are styphnic acid and dinitroresorcinol (Figure 2). Furthermore, activation of these compounds may be lowered in the presence of transition-metal impurities (19). An explosion occurring from the preparation and distillation of nitroindane underscores the notion that “any nitroaromatic is potentially explosive” (reviewer's comment) (20). Following from nitroglycerine, other nitroaliphatic compounds may also pose explosion hazards, especially nitro esters such as erythritol tetranitrate, nitromannite, nitroglycol, and dinitroglycol (diethylene glycol dinitrate), illustrated in Figure 3.

Nitrogen oxide salts are also worth mentioning here. Ammonium nitrate, the most widely industrially produced

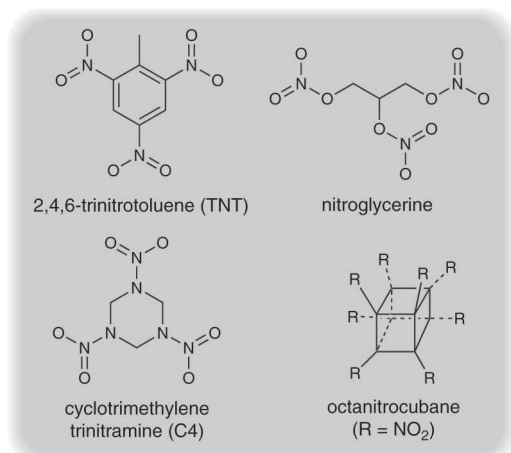


Figure 1. Three common explosives and octanitrocubane, a new potential high explosive (formal charges on the nitro groups are omitted here and elsewhere).

explosive, serves as a good example of a compound in which oxidizer (NO_3^-) and fuel (NH_4^+) are situated together as an ionic pair (Figure 4). Nitrite species may also pose hazards (21). Mishaps with *metal* nitrates have also been cited (22, 23).

Hydrazido and Azido Derivatives

With respect to functionality, hydrazine and hydrazoic acid (Figure 5) feature the respective hydrazido and azido groups (24–26). “Organic azides are known to exhibit dangerous instability, especially those containing unsaturated car-

bon–carbon bonds and more than one azido group. Heat, shock, and traces of strong acids or metallic salts may trigger explosive decomposition” (25). 1,4-Diazido-2-butene and 4-azidobenzaldehyde, illustrated in Figure 5, are examples with multiple azido functionalities as well as unsaturation (27). “If there is an unusually high proportion of nitrogen and N–N bonds in the compound, instability should be suspected (hydrogen azide, 97.6% N, and hydrazine, 87.4%, are both explosively unstable; ammonia, 82.2%, is not)” (14). Nitrogen content is also connected to oxygen balance mentioned below. Precise and clearly balanced reactions of energetic materials can be found elsewhere in the literature (5, 6, 14).

NaN_3 serves as an explosive in automotive airbags and must not be used with halogenated organic solvents or cosolvent or it may give rise to organic azides, such as diazidomethane (from dichloromethane) (28, 29). Thus in the laboratory, deposition of (heavy) metal azides may either be deliberate or surreptitious, such as buildup of $\text{Pb}(\text{N}_3)_2$ in plumbing lines.

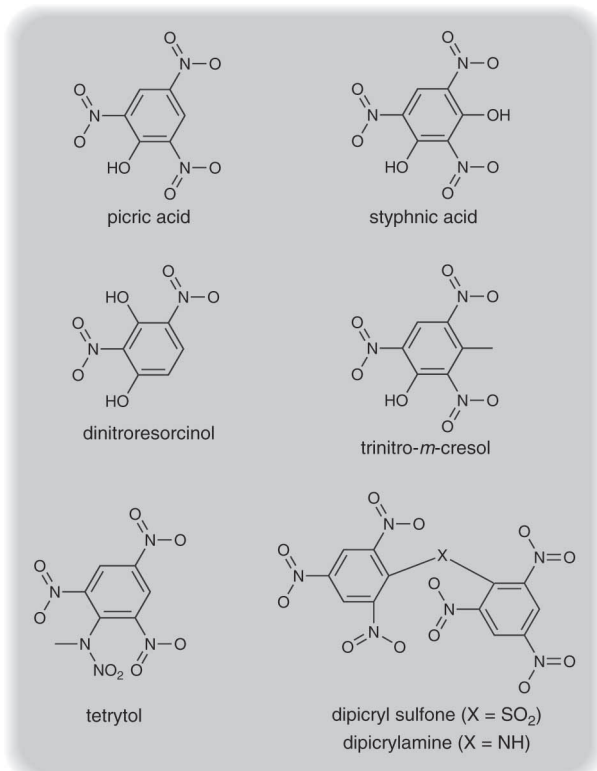


Figure 2. Select nitroaromatics, some with nondescriptive trivial names.

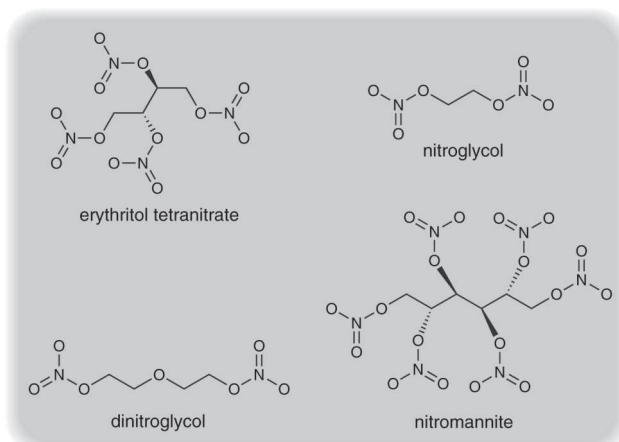


Figure 3. Examples of nitroester-aliphatic compounds.

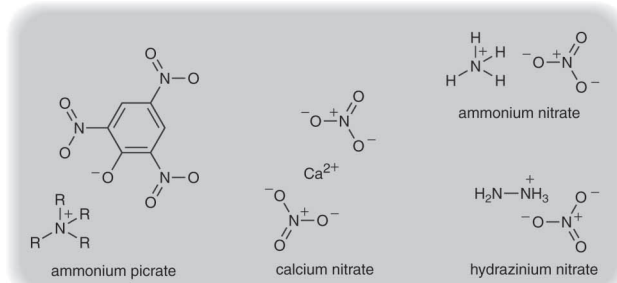


Figure 4. Some nitrate salts.

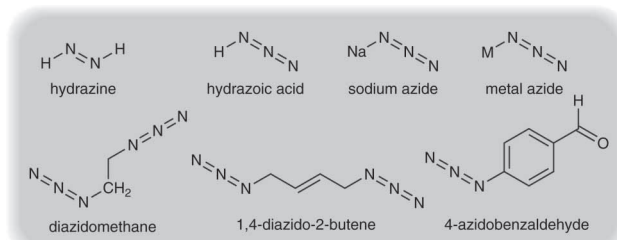


Figure 5. Hydrazido- and azido- and related derivatives. Dinuclear metal azide bonding modes also exist.

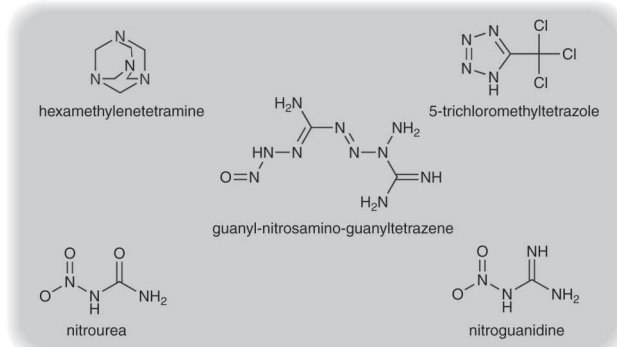


Figure 6. Derivatives containing reduced nitrogen.

Compounds with no particular functionality except for high nitrogen content can also be explosive. Simple binary compounds such as NCl_3 , NI_3 , and sulfur nitride (S_4N_4) are examples of these (30). Cyclic compounds such as 5-trichloromethyltetrazole (31–33) and hexamethylenetetramine (34) as well as chain compounds such as guanyl-nitrosamino-guanyl-tetrazene (Figure 6) possess high nitrogen and may directly or indirectly release N_2 . Danger from derivatives of these types that also bear nitro groups (e.g., nitrourea and nitroguanidine) is compounded (Figure 6).

Perchlorate Ion

Perchloric acid is a potential laboratory hazard (35, 36). The perchlorate anion, whether in an organic or inorganic system, is an extremely strong oxidant (Figure 7). For example, NH_4ClO_4 serves as a component in solid rocket fuel (37, 38). “Safety letters” reporting accidental explosions involving perchlorate salts are published regularly (6, 35–59). “Potential hazards involving perchlorates are multiplied with increasing temperature, dryness, and/or perchlorate content” (60). Some specific examples of “organic” systems include tropylium perchlorate (53, 57) and 2-dimethylaminomethylene-1,3-bis(dimethylimmonio)propane perchlorate (44). Here, as

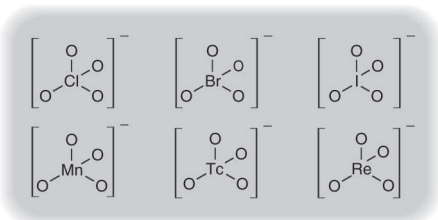


Figure 7. The perchlorate ion and its relatives.

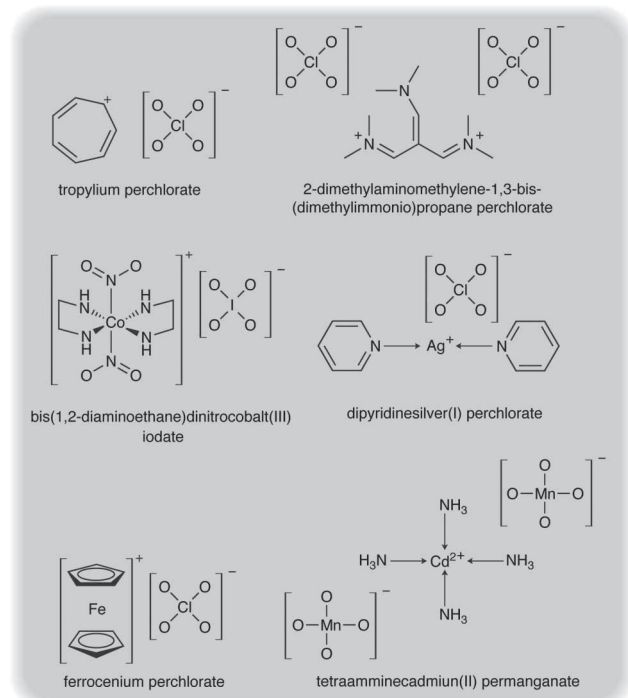


Figure 8. Perchlorate complexes.

with ammonium nitrate, a fuel–oxidizer pair is present. Use of lithium perchlorate may result in inadvertent formation of dangerous organic perchlorates (39).

The chlorine atom in ClO_4^- has a valence number of seven, as with its congeners in BrO_4^- and IO_4^- and atoms of group 7 in MnO_4^- , TcO_4^- , and ReO_4^- . Bretherick has listed three ammine metal oxosalts of notoriety: bis(1,2-diaminoethane)dinitrocobalt iodate, tetraamminecadmium permanganate, and dipyridinesilver perchlorate (Figure 8) (14). Again in these compounds there exists a fuel and strong oxidant source ionically bonded.

In preparative work, it should be considered whether other anions such as BF_4^- , BAR_4^- , PF_6^- , I_3^- , and CF_3SO_3^- ($\text{Ar} = \text{aryl}$) can be used in place of EO_4^- ($\text{E} = \text{Cl, Br, I, Mn, Tc, Re}$) especially when multiple counterions are required (61, 62). If perchlorate ion must be used, then it is prudent to *reduce the scale*, *avoid concentration* (desolvation, drying, crystallizing), and *avoid initiation* (shocking, scraping, friction, sparks).

Peroxides

As dioxygen, *especially* $\text{O}_2(\text{l})$, can play a major part in an explosive fire through its “oxidizing” ability (63), compounds with $\text{R}-\text{O}-\text{O}-\text{R}'$ (R and R' are illustrated in Figure 9) functionality or metal-based $\text{M}-\text{O}_2$ (64) are also effective oxidizers and notoriously shock sensitive. The simplest peroxide, hydrogen peroxide (65), can oxidize many organic compounds and can lead to shock sensitive compounds (66–69). Mishaps involving organic hydroperoxides, $\text{R}-\text{O}-\text{O}-\text{H}$, such as cumene hydroperoxide (70–72) or di-*tert*-butyl peroxide (73) have been reported. The peroxide dimer of acetone is also a grave explosion hazard (74) and the notorious triacetone triperoxide may have been used in the London 2005 public transit bombings (75).

Peracids such as peracetic acid and performic acid can lead to dangerous peroxide buildup (6, 25, 76). As with nitro, azido, and perchlorate compounds, *impurities* may play a role in *catalyzing* unexpected detonations or deflagrations

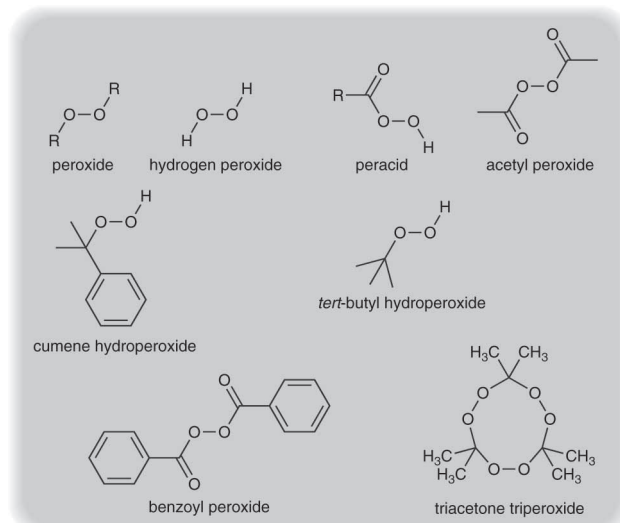


Figure 9. Peroxide and hydroperoxide functionalities commonly of a shock sensitive nature.

(71). Superoxides should also be mentioned; for example, potassium superoxide (KO_2) can form treacherously sensitive crusts on elemental potassium (77, 78).

Precipitation of peroxides through autoxidation from aged diethyl ether may occur (but *not just* ether, see Figure 10) (79). Peroxidation happens when O_2 formally inserts into a C–H bond to form the hydroperoxide, C–O–O–H (80). C–H bonds with susceptibility for peroxidation are provided in Figure 10 (14). The range encompasses ether and acetal groups, aldehydes, vinyl and allyl groups, in addition to bonds positioned at the benzylic position and at tertiary carbons. With peroxides, as well as other compounds, “oxygen balance” can gauge explosion efficiency: “If an explosive molecule contains just enough oxygen to convert all of its carbon to carbon dioxide, all of its hydrogen to water, and all of its metal to metal oxide with no excess, the molecule is said to have a zero oxygen balance” (81).

Fulminates and Acetylides

Fulminic acid is an unstable species ($\text{C}\equiv\text{N}-\text{OH}$) and metal fulminates, notably those of silver and mercury, are also very unstable (Figure 11). These complexes might form (i) when EtOH, nitric acid, and metal salts are present in solution or (ii) when nitromethane (NO_2-Me) is mixed with mercury, silver, or lead compounds (6).

Acetylene, while being an ubiquitous fuel for welding, is also a laboratory reagent (6). Acetylene is stored as an acetone solution to prevent exothermic acetylene polymerization (explosion). Also, dangerous acetylides may form if

copper is present (Figure 11). Structures of the hazardous common metal salts, mercury oxalate and mercury tartrate, have also been included in Figure 11.

Gases and Vapor Trails

An obvious and historically common source of explosions involves gaseous fuels such as (but not limited to) H_2 and CH_4 (13). Surreptitious leakage out of containment cylinders and large accumulation (high concentration) in enclosures is an obvious hazard. Gases such as H_2 , methane, and ammonia are *lighter than air* and may collect at the ceiling. In a related way, during solvent keg filling, vapors of spilled volatile solvents such as Et_2O or pentane might surreptitiously flow along the ground in a vapor trail and ignite in a location *remote from the source* (many meters away). These trails might flow down a corridor, or even stairs. *Vapors burn, not bulk liquids*, and possibly with explosive violence (80).

Physical Explosions

Some physical explosion hazards involve condensed-phase materials becoming gaseous, causing *over*-pressurization. Below are several practical points to consider.

- *Cylinders and bombs.* Compressed or liquefied gases, whether flammable or not, can form great pressure in the tank (Charles's law) when exposed to heat or fire leading to failure and giving much mechanical energy (82). If the contents are flammable, such as H_2 , a chemical explosion could then occur in tandem. Also the ubiquitous “bomb” reaction in organic chemistry in which pressurized gas is heated is innately hazardous and is never to be performed casually.
- *Cold traps.* In a vacuum line setup used to manipulate air- and moisture-sensitive materials, liquid nitrogen is frequently used in Dewar storage flasks to collect volatiles into “cold traps”. Firstly, if argon is used as the inert atmosphere, there is a risk it could condense into the cold trap. Secondly, imposing a static vacuum (“sealed” system) onto a vessel (e.g., Schlenk tube, ampoule) being cooled with $\text{N}_2(\text{l})$ is hazardous in that air can seep in between parts (plugs, seals, stopcocks) giving O_2 condensation inside the trap. This, as with many accidental explosion hazards, is surreptitious. If these sealed vessels are left to warm to room temperature, then a shattering physical explosion could occur. The hazard may be quelled by applying vacuum to the closed system, while keeping the condensate cold.
- *Implusions.* A hazard related to that of “cold traps” involves evacuating flawed (scratched, chipped, cracked) vessels (e.g., Schlenk tubes, ampoules) that could “fail”, via an *implosion*. One should inspect for defects and set marred glassware aside.
- *Water coolant.* In closed coolant systems, or wherever there is a large temperature difference between *hot elements* of the machine or instrument and the *coolant*, there is physical explosion potential. For instance, if water coolant is introduced after the device or instrument has long become overheated, a steam explosion could result. These kinds of events occur commonly with heaters and boilers. Alternately it can be dangerous to cool molten metals with water (83).

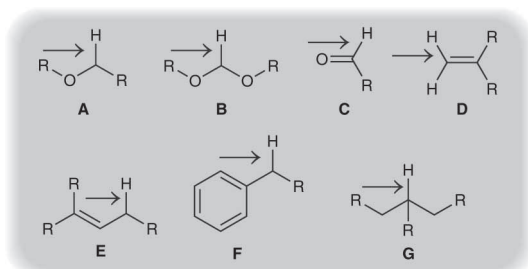


Figure 10. Ether (A), acetal (B), aldehyde (C), vinyl (D), allyl (E), benzylic (F), and tertiary (G) C–H bonds susceptible to hydroperoxidation (14).

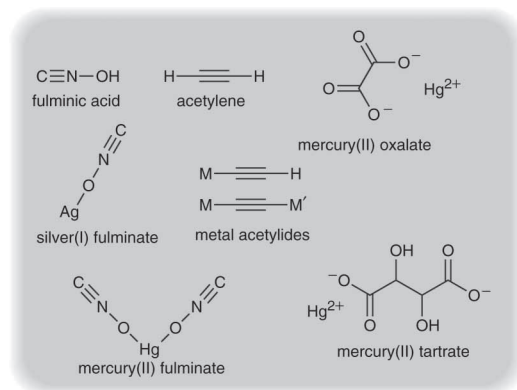


Figure 11. Hazardous metal complex functionalities.

- *Dry ice.* Dry ice, CO₂(s), is often stored in an ice chest or a cooler, but this enclosure must not be thoroughly sealed. Over time, the bulk solid loses volume to sublimation, giving lots of gas. Cases are known in which tightly sealed transportation containment has exploded (84).
- *Exothermic mixing.* The rapid addition of KOH or concentrated H₂SO₄ to water gives off heat or vapor and may create unsafe pressure differences if the system becomes tightly sealed. For runaway reactions see reference (85).

Summary and Conclusions

Several important explosion hazards have been briefly mentioned, including some nitro-containing substances, many of which are common, perchlorates, and peroxides as well as hazardous metal complexes such as the fulminates that are not generally illustrated. This discussion of explosion hazard with emphasis on clear illustrations of molecular structure may help guide chemical laboratory researchers, especially beginning graduate students. Those working with the hazardous materials above, or related materials, must proceed cautiously and may want to consider alternative and safer synthetic routes. There are also occurrences of physical explosions involving phase transformations or severe pressure increase that can cause equal devastation such as the shattering of glass vessels. Note that compounds are even more hazardous when multi-functionalities are present: multiple nitro, azo, and hydrazo groups; perchlorate anions; and multiple auto-hydroperoxidation sites.

If energetic materials must be used, this is time for protocol review and safety personnel consultation. *Do not take chances! There are standard protocols for both explosion safety and explosives sensitivity testing of new materials suspected of being explosive.* This is often the work for specially trained personnel with specialized equipment and techniques such as differential scanning calorimetry and shock sensitivity in an energetic materials laboratory. The most updated MSDS on a compound or related compound should be obtained. If one must proceed with full knowledge of explosive hazards, precautions might include (i) having a spotter, (ii) working on small scale, (iii) avoiding compound drying and heating (e.g., perchlorates, peroxides), (iv) using protective gear such as a blast shield, full-face shield, thick protective apron, fire-retardant lab coat, leather gloves, and bulletproof vest, and (v) having a fire extinguisher accessible.

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