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NMSU EXPLOSIVE CHEMICAL MANAGEMENT PROCEDURES

These procedures are a guide to help prevent explosions and protect health and the environment. If you identify old explosives or old organic peroxide forming chemicals, immediately contact Environmental Health and Safety (EH&S) at 646-3327.

1. INTRODUCTION: All explosive chemicals should be identified and carefully managed. There are two main classes of explosive chemicals:

1.1. STANDARD EXPLOSIVE CHEMICALS: These consist of explosive compounds manufactured for detonation such as dynamite, gunpowder, blasting caps, and fireworks. These compounds are rare on the NMSU campus and relatively stable under normal conditions. If these compounds are not being used for a specific, authorized activity they should be immediately called in for pick up by EH&S at 646-3327.

1.2. POTENTIALLY EXPLOSIVE CHEMICALS (PECs): These consist of lab chemicals not intentionally made for detonation but which may explode and/or cause fires if not properly managed. A number of PECs are common on the NMSU campus and should be handled with extra precaution. The main focus of this SOP is to help identify PECs and describe the precautions that should be taken to manage them from purchase through disposal.

2. ORGANIC PEROXIDE FORMING CHEMICALS: This is the principle group of PECs that lead to problems in university labs. They are carbon-based chemicals capable of forming potentially explosive peroxide "O-O" bonds. Below is a breakdown of the four main subcategories of organic peroxide forming chemicals; common ones are specifically identified in the appendices.

2.1. Chemicals that form explosive levels of peroxides without a concentration step, e.g., evaporation, distillation, etc., are listed in **Appendix 1**. These chemicals can be a particular hazard since peroxides can form even without opening the containers. Therefore, only small amounts should be ordered and used as soon as possible. After opening, they should not be kept for over **three months.** When possible, store these chemicals under a nitrogen blanket.

2.2. Chemicals that form explosive levels of peroxides upon concentration are listed in **Appendix 2**. These chemicals typically accumulate hazardous levels of peroxides when evaporated, distilled, contaminated, or have their peroxide inhibiting compounds compromised. After opening, they should not be kept for over **12 months.**

2.3. Chemicals that may autopolymerize as a result of peroxide accumulation are listed in **Appendix 3**. These chemicals can under go hazardous polymerization reactions that are initiated by peroxides that have accumulated in solution. They are typically stored with polymerization inhibitors to prevent these dangerous reactions. Inhibitors do become compromised over time however, and thus after opening, these chemicals should not be kept for over **12 months.** Uninhibited chemicals should not be stored over **24 hours**. Uninhibited chemicals should be inhibited with the appropriate compounds before the 24-hour mark is exceeded. Do not store inhibited chemicals in this category under an inert atmosphere because some of the inhibitors require a small amount of oxygen to work.

2.4. Chemicals that cannot be placed into the above categories but still have the potential for forming hazardous levels of organic peroxides are listed in **Appendix 4**. After opening, they should not be kept for over **12 months.**

2.5. The above lists are not all-inclusive. A wide range of organic chemicals can be oxidized by reaction with molecular oxygen to form explosive peroxides. The chemicals listed above are considered PECs in their pure form. However, when they are mixed with other compounds, their hazards can change. When mixed with other compatible chemicals (especially water) their explosive hazard is sometimes decreased through dilution, but not always. Therefore, laboratory workers should consult more experienced laboratory personnel, container labels, Materials Safety Data Sheets (MSDSs), books such as “Hawley’s Chemical Dictionary,” or a list of **Common Peroxide Moieties (See Appendix 5)** if the hazards of a chemical are not well known or the chemical is suspected to form organic peroxides. It must be kept in mind that there are no “complete lists” of organic peroxide forming chemicals. Overall, all peroxide forming chemicals should be identified and carefully managed to best protect the health and safety of all laboratory co-workers.

3. ORGANIC PEROXIDE FORMING CHEMICALS DISCLAIMER: There are significant uncertainties about the hazards and safe handling procedures for peroxide forming organic chemicals. Currently, no specific Federal Occupational Safety and Health Administration (OSHA) regulations apply for the identification and handling of organic peroxide forming chemicals. Also, there are no definitive data about the concentration at which organic peroxides pose a hazard and several common peroxide detection methods may not detect all types of unstable peroxides. The steps outlined below are simply recommended “best management procedures” followed at other universities to help prevent potential explosions. Overall, workers are responsible for determining what procedures are best suited for their individual situations. The resources identified in Section 2.5 should be consulted <u>before</u> carrying out potentially hazardous experiments in the laboratory.

4. BEST MGT. PROCEDURES FOR ORGANIC PEROXIDE FORMING CHEMICALS:

4.1. RECEIPT OF ORGANIC PEROXIDE FORMING CHEMICALS: Containers should be **dated** and **labeled “Potentially Explosive Peroxide”** immediately upon receipt of organic peroxide forming chemicals. The containers also should be dated when first opened and annotated with the required disposal date according to the peroxide lists above.

4.2. STORAGE OF ORGANIC PEROXIDE FORMING CHEMICALS: Keep all PECs away from ignition sources such as open flames, hot surfaces, sparks, heat, and direct sunlight. Store them in tightly closed original containers to protect them from evaporation or possible contamination. Protect them from shock, friction, and do not shake. Ensure they are stored away from incompatible materials such as oxidizers (for exact incompatibility information consult specific MSDSs). When possible, store most of them under inert gas. Consult the attached appendices for details.

4.3. USE OF ORGANIC PEROXIDE FORMING CHEMICALS: Extreme care should be taken when opening and pouring organic peroxide formers. **The cap and threads on the container should be wiped down with a damp towel after use.** This is to prevent the deposition of peroxides around the cap area that could detonate simply from the friction of turning the cap. **Most peroxide explosions occur during purification or distillation procedures.** Therefore, before distilling a PEC or mixture of PECs, follow the designated peroxide test procedures outlined below.

4.4. TESTING ORGANIC PEROXIDE FORMING CHEMICALS:

4.4.1. NEVER test containers of unknown age or origin. Old containers may contain hazardous levels of peroxides, or peroxides may have crystallized on the cap threads, both of which present serious hazards when opening the bottle for testing. EH&S should be immediately notified in this situation and the suspect container should not be handled by anyone in the laboratory.

4.4.2. The best method to test for peroxides involves semi-quantitative analysis using **peroxide detection dip strips**. These can be purchased from **Lab Safety (1-800-356-0783; Product No. 1A-1162)**. The peroxide strips are similar to the use of pH paper and a simple instruction sheet accompanies the strips. If a test strip indicates a peroxide concentration **above 10 ppm**, the chemical should not be used and should be immediately turned in for disposal.

4.4.3. Chemicals that reach their expiration dates can be regularly tested. If they reveal a peroxide concentration below 10 ppm they can still be used. Post-expiration date chemicals should be **tested every six months and test dates and results annotated on the bottle.** Once a 10 ppm or greater peroxide level is detected, chemicals should be sent for disposal.

4.4.4. All peroxide forming chemicals to be distilled must be tested for peroxide content AND SHOULD NOT BE DISTILLED if they contain <u>any</u> measurable peroxides. When distilling peroxide forming chemicals, volume reductions should not exceed 80%. The remaining 20% of the chemical should remain in the distillation column to prevent the apparatus from drying out. **Distilling peroxides to dryness guarantees explosions.** If possible, a nonvolatile organic compound such as mineral oil should be added to the distillation mixture. The oil will remain behind and dilute any potential peroxides. During distillation, the solution must only be stirred mechanically or with inert gas; air or other oxygen-containing mixtures can cause peroxide formation. PECs without peroxide inhibitors should not be distilled.

4.4.5. Modifications to the dip strip testing method are required to test **low volatile organic compounds**. For water-miscible chemicals, add three drops of water to one drop of a chemical to be tested. Wet the dip strip, wait until the color stabilizes, and then multiply the result by 4. For water-immiscible chemicals, add three drops of a volatile ether to one drop of a chemical to be tested. Wet the dip strip, wait until the color develops, and then multiply the result by 4.

4.4.6. Note: Lab Safety instructions indicate that their test strips will detect hydroperoxides and most higher peroxides, but some polyperoxides may be poorly detected. Thus use of test strips is not always 100% effective at identifying dangerous levels of peroxides.

4.4.7. Peroxide testing strips have a limited shelf life. Refrigeration is not recommended once the container has been opened because water condensing on the strips reduces their effectiveness. The strips should be stored in as dry an environment as possible.

5. OLD ORGANIC PEROXIDE FORMING CHEMICALS: When old peroxide chemicals are identified, leave them in place and call EH&S for disposal. Some of the chemicals could be too dangerous even to move. A quick look at the containers is helpful in revealing the dangerous situations (use of a flashlight as a light source can be helpful to peer through bottles). If **crystals, solid masses, cloudiness, string-like formations, layers, or discoloration** are observed, then there are likely very high levels of peroxides and every one in the lab should be warned not to touch the chemicals until EH&S has evaluated the situation. **Warning signs should be posted.** Never attempt to force open a rusted or stuck cap on a container of a PEC. Never scrape or scrub glassware or containers with oily or crusty residues that have been used with peroxide-forming compounds.

6. KNOWN ORGANIC PEROXIDE CHEMICALS: There are numerous chemicals produced that are pure organic peroxides. A number of these chemicals are easy to identify however because they contain the phrases "**peroxide**" or "**peroxy**" in their nomenclature. These chemicals are extremely explosive and should be handled as outlined in previous sections. Some are "**wetted**" and should not be allowed to dry out, e.g., **benzoyl peroxide** and **picric acid.** Others are temperature sensitive and often unstable at room temperature. Special **explosive proof refrigerators** with back up power supplies must be in place before purchasing such temperature sensitive chemicals. EH&S should also be contacted in advance of obtaining temperature sensitive chemicals. Unless specifically instructed by the chemical manufacturer however, do not refrigerate organic peroxide forming chemicals since some develop higher peroxide levels upon cooling. The best rule of thumb is to review the MSDS on each individual chemical on a case-by-case basis.

7. OTHER POTENTIALLY EXPLOSIVE CHEMICAL CATEGORIES: Below are additional categories of PECs that can lead to serious explosions or fires.

7.1. EXPLOSIVE AND POTENTIALLY EXPLOSIVE CHEMICAL FAMILIES: While much attention has been paid to peroxide forming chemicals, many non-peroxide chemicals are also explosive hazards. **Appendix 6** identifies a number of other common explosive/potentially explosive chemical families and specific examples targeted as "problem compounds." The bottom line is **not all chemicals have to be peroxides or potential peroxide formers to pose an explosion hazard.**

7.2. SPONTANEOUS COMBUSTIBLES: These chemicals ignite when exposed to **air or water and can lead to explosions**. Extreme care should be used in handling/storing these materials. Often these types of chemicals must be **stored under oil or inert gas**. Common campus examples are metallic **sodium** and **potassium,** both of which must be stored under oil.

7.3. OXIDIZERS: These chemicals can be considered PECs when combined with **organic materials**. Often just mixing can result in a fire or explosion. Examples of oxidizers include **salts containing nitrates, chlorates,** and most "**per**" prefixed chemicals such as **permanganates**, **persulfates, and peroxides**. (In fact, organic peroxide chemicals are dangerous because they are compounds with both oxidizer and organic components.) These compounds must be stored separately from flammable materials such as acetone, alcohol, or other volatile organic solvents.

7.4. PERCHLORIC ACID: Is a PEC that requires very careful handling. Overall, perchloric acid is a strong oxidizer often used for the hot digestion of a variety of materials. **When used for digestions, it must be used in specifically designated hoods.** If at all possible, perchloric acid should be used cold. Anhydrous perchloric acid is unstable at room temperature and will decompose violently. Commercial perchloric acid is 72%. Do not store or use organic materials like solvents in a perchloric acid hood. If a vacuum is needed for perchloric acid work a water aspirator must be used instead of a mechanical pump since the latter contains hydrocarbon oils, which if contaminated with perchloric acid, can lead to explosive mixtures. Perchloric acid should be inspected for contamination. If the acid is discolored it must immediately be turned in for disposal. If a spill occurs, even if it is small, consult your supervisor immediately! Do not attempt to clean it up without supervision. When in doubt, call EH&S or 911.

7.5. CHEMICAL OVER-PRESSURIZATION RISKS: Some chemicals give off gaseous degradation by-products that can lead to over-pressurization of containers and possible explosions- especially when heated or when incompatible chemicals are mixed together. Containers that are bulging clearly reveal an over-pressurization hazard. Consult specific MSDSs and/or contact EH&S on a case-by-case basis.

8. DISPOSAL OF POTENTIALLY EXPLOSIVE CHEMICALS: Disposal is simple and straightforward. Complete an NMSU Waste/Material Tracking Form and call Environmental Health and Safety at 646-3327 for chemical pick-ups.

9. SPILLS: Spills of potentially explosive chemicals should be dealt with carefully. MSDSs should be immediately consulted to establish the precise nature of the hazard. Then the person responsible for the spill should determine whether they have the expertise and protective equipment necessary to clean up the spill. **If there is any doubt about safely cleaning up the spill contact EH&S at 646-3327 or dial 911.** The person cleaning up the spill must ensure the clean up materials are picked up by EH&S rather than thrown in the regular trash.

10. EMPTY CONTAINERS: Empty containers of peroxide forming chemicals can still pose a significant hazard. The containers should be **triple rinsed and the rinsate collected for disposal** as hazardous waste immediately after the last amount is removed. If after triple rinsing the container is deemed to be free of residue, the label may be removed and the container discarded into the regular trash. If the container cannot be satisfactorily rinsed, it must be turned in to EH&S as hazardous waste. Remember, **do not attempt to open or rinse an old container that contained a PEC.**

11. TRAINING: All personnel who work with potentially explosive chemicals need to be trained in the proper storage, use, and disposal of these dangerous chemicals. This SOP provides only basic training; personnel should discuss specific questions with their supervisor or EH&S staff.

APPENDIX 1

Chemicals that form explosive levels of peroxides without a concentration step, e.g., evaporation, distillation, etc., are listed below. These chemicals can be a particular hazard since peroxides can form even without opening the containers. Therefore, only small amounts should be ordered and used as soon as possible. After opening, they should not be kept for over three months. When possible, store these chemicals under a nitrogen blanket.

Butadiene (When Stored as a Liquid Monomer)
Chloroprene (When Stored as a Liquid Monomer)
Diisopropyl ether
Divinyl acetylene
Divinyl ether
Isopropyl ether
Potassium amide (Inorganic Peroxide Former)
Sodium amide (Inorganic Peroxide Former)
Sodamide (Inorganic Peroxide Former)
Tetrafluoroethylene (When Stored as a Liquid Monomer)
Vinylidene chloride

APPENDIX 2

Chemicals that form explosive levels of peroxides upon concentration are listed below. These chemicals typically accumulate hazardous levels of peroxides when evaporated, distilled, contaminated, or have their peroxide inhibiting compounds compromised. After opening, they should not be kept for over 12 months.

Acetal
Acetaldehyde
Benzyl alcohol
Butadiyne
2-Butanol
Cellosolves
Chlorofluoroethylene
Cumene
Cyclohexene
Cyclohexanol
2-Cyclohexen-1-ol
Cyclooctene
Cyclopentene
Decahydronaphthalene
Decalin
Diacetylene
Dicyclopentadiene
Diethyl ether
Diethylene glycol
Diglyme (Dimethyl ether)
Dioxanes
Ethyl ether
Ethylene glycol dimethyl ether
Ethylene glycol ether acetates
Furan
Glyme
4-Heptanol
2-Hexanol
Isopropyl alcohol
Isopropylbenzene
Methyl acetylene
3-Methyl-1-butanol
Methyl cyclopentane
Methyl isobutyl ketone
4-Methyl-2-Pentanol
4-Methyl-2-Pentanone
2-Pentanol
4-Penten-1-ol
1-Phenylethanol and 2-Phenylethanol
2-Propanol
Tetrahydrofuran
Tetrahydronaphthalene
Tetralin
Vinyl ethers

APPENDIX 3

Chemicals that may autopolymerize as a result of peroxide accumulation are listed below. These chemicals can under go hazardous polymerization reactions that are initiated by peroxides that have accumulated in solution. They are typically stored with polymerization inhibitors to prevent these dangerous reactions. Inhibitors do become compromised over time however, and thus after opening, these chemicals should not be kept for over 12 months. Uninhibited chemicals should not be stored over 24 hours. Uninhibited chemicals should be inhibited with the appropriate compounds before the 24-hour mark is exceeded. Do not store inhibited chemicals in this category under an inert atmosphere because some of the inhibitors require a small amount of oxygen to work.

Acrylic acid
Acrylonitrile
Butadiene
Chlorobutadiene
Chloroprene
Chlorotrifluoroethylene
Dibenzocyclopentadiene
9,10-Dihydroanthracene
Indene
Methyl methacrylate
Styrene
Tetrafluoroethylene
Vinyl acetate
Vinyl acetylene
Vinyl chloride
Vinyl pyridine

APPENDIX 4

Chemicals that cannot be placed into the categories listed in Appendixes 1-3 but still have the potential for forming hazardous levels of organic peroxides are listed below. After opening, they should not be kept for over 12 months.

Acrolein
Allyl ether
Allyl ethyl ether
Allyl phenyl ether
p-(n-Amyloxy) benzoyl chloride
n-Amyl ether
Benzyl n-butyl ether
Benzyl ether
Benzyl ethyl ether
Benzyl methyl ether
Benzyl-1-napthyl ether
Bis(2-n-butoxyethyl) phthalate
1,2-Bis(2-chloroethoxyl)ethane
Bis(4-chlorobutyl) ether
Bis(2-chloroethyl) ether
Bis(chloromethyl) ether
Bis(2-ethoxyethyl) adipate
Bis(2-ethoxyethyl) ether
Bis(2-ethoxyethyl) phthalate
Bis(2-(methoxyethoxy)-ethyl) ether
Bis(2-methoxyethyl) carbonate
Bis(2-methoxyethyl) ether
Bis(2-methoxyethyl) phthalate
Bis(2-methoxymethyl) adipate
Bis(2-phenoxyethyl) ether
2-Bromomethyl ethyl ether
B-Bromophenetole
o-Bromophenetole
p-Bromophenetole
3-Bromopropyl phenyl ether
Buten-3-yne
tert-Butyl ethyl ether
tert-Butyl methyl ether
n-Butyl phenyl ether
n-Butyl vinyl ether
Chloroacetaldehyde diethylacetal
1-(2-Chloroethoxy)-2-phenoxyethane
Chloroethylene
Chloromethyl methyl ether
B-Chlorophenetole
o-Chlorophenetole
p-Chlorophenetole
Cyclopropyl methyl ether
Diallyl ether
p-Dibenzyloxybenzene
1,2-Dibenzyloxyethane
p-Di-n-butoxybenzene
1,2-Dichloroethyl ethyl ether
2,4-Dichlorophenetole
m,o,p-Diethoxybenzene
1,2-Diethoxyethane
Diethoxymethane
2,2-Diethoxypropane
Diethyl acetal
Diethyl ethoxymethylenemalonate
Diethyl fumarate
Diethylketene
1,1-Dimethoxyethane
Dimethoxymethane
3,3-Dimethoxypropene
Dimethylketene
2,4-Dinitrophenetole
1,3-Dioxepane
Di-n-propoxymethane
Di(1-propynyl) ether
Di(2-propynyl) ether
p-Ethoxyacetophenone
1-(2-Ethoxyethoxy)ethyl acetate
2-Ethoxyethyl acetate
(2-Ethoxyethyl)-o-benzoyl benzoate
1-Ethoxynaphthalene
o,p-Ethoxyphenyl isocyanate
3-Ethoxypropionitrile
1-Ethoxy-2-propyne

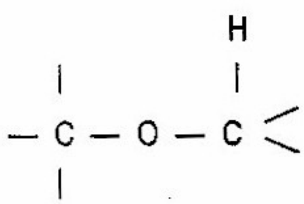
APPENDIX 4 Continued

2-Ethylacrylaldehyde oxime
2-Ethylbutanol
Ethyl-b-ethoxypropionate
2-Ethylhexanal
Ethyl vinyl ether
1,2-Epoxy-3-isopropoxypropane
1,2-Epoxy-3-phenoxypropane
4,5-Hexadien-2-yn-1-ol
2,5-Hexadiyn-1-ol
n-Hexyl ether
o,p-Iodophenetole
Isoamyl benzyl ether
Isoamyl ether
Isobutyl vinyl ether
Isophorone
B-Isopropoxypropionitrile
Isopropyl 2,4,5-trichlorophenoxyacetate
Limonene
1,5-p-Methadiene
3-Methoxy-1-butyl acetate
Methoxy-1,3,5,7-cyclooctatetraene
2-Methoxyethanol
3-Methoxyethyl acetate
2-Methoxyethyl vinyl ether
B-Methoxypropionitrile
Methyl p-(n-amyloxy)benzoate
n-Methylphenetole
2-Methyltetrahydrofuran
m-Nitrophenetole
1-Octene
Oxybis(2-ethyl acetate)
Oxybis(2-ethyl benzoate)
B,B-Oxydipropionitrile
1-Pentene
Phenoxyacetyl chloride
a-Phenoxypropionyl chloride
Phenyl-o-propyl ether
p-Phenylphenetone
n-Propyl ether
n-Propyl isopropyl ether
Sodium 8,11,14-eicosatetraenoate
Sodium ethoxyacetylide
1,1,2,3-Tetrachloro-1,3-butadiene
Tetrahydropyran
Triethylene glycol diacetate
Triethylene glycol dipropionate
1,3,3-Trimethoxypropene
4-Vinyl cyclohexene
Vinylene carbonate

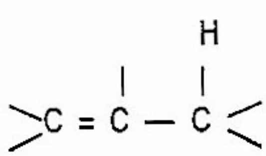
APPENDIX 5

Below are diagrams of moieties that can form organic peroxides. These moieties are ranked from highest (1) to lowest (14) risk of forming potentially dangerous peroxide concentrations.

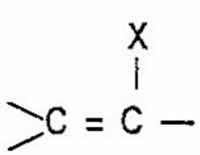
1. Ethers and acetals with α-hydrogen



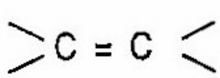
2. Alkenes with alyllic hydrogen



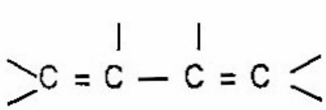
3. Chloroalkenes, fluoroalkenes



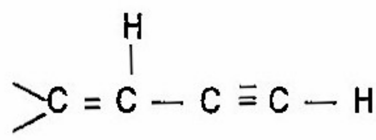
4. Vinyl halides, esters, ethers



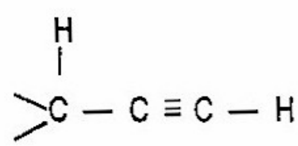
5. Dienes



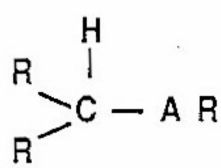
6. Vinylalkynes with α-hydrogen



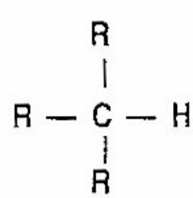
7. Alkylalkynes with α-hydrogen



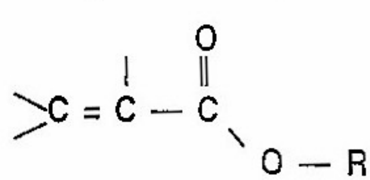
8. Alkylalkynes with tertiary α-hydrogen



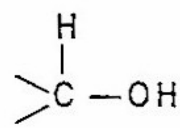
9. Alkanes and cycloalkanes with tertiary hydrogen



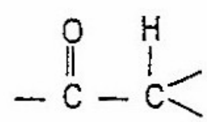
10. Acrylates, methacrylates



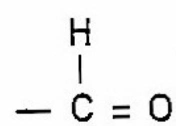
11. Secondary alcohols



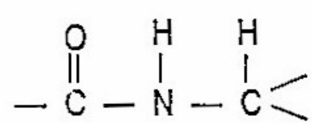
12. Ketones with α-hydrogen



13. Aldehydes



14. Ureas, amides, and lactams with α-hydrogen atom on a carbon attached to nitrogen



APPENDIX 6

While much attention has been paid to peroxide forming chemicals, many non-peroxide chemicals are also explosive hazards. The list below identifies a number of other common explosive/potentially explosive chemical families and specific examples targeted as "problem compounds." The bottom line is not all chemicals have to be peroxides or potential peroxide formers to pose an explosion hazard.

Azides:
Lead(II) azide
Silver(I) azide
Sodium Azide

Azo Compounds:
Diazomethane
Azobisisobutylnitrile

Metal Fulminates:
Lead(II) fulminate
Silver(I) fulminate

Metal Acetylides:
Lead acetylides
Silver acetylides

Nitrates:
Propyl nitrate
Pentaerythritol tetranitrate (PETN)

Nitro Compounds:
Cyclonite (RDX)
Dinitrophenol
Dinitrotoluene
Dinitrophenylamine
Nitrocellulose
Trinitrophenol (Picric Acid)
Trinitrotoluene (TNT)

Perchlorates:
Perchloric acid digests
Transition metal perchlorate salts

REFERENCES

1. "Standard for Storing and Using Peroxidizable Organic Chemicals"; Kelly, R.J. and Miller, G.; Lawrence Livermore National Laboratory Publication UCRL-AR-133218, Rev. 1, May 1999.

2. "Information on Peroxidizable Organic Chemicals"; Stanford University; www.stanford.edu/dept/EHS/lab/perox/perox.html.

3. "Peroxide Forming Compounds"; Cornell University; www.ehs.cornell.edu/ehs/newweb/chemhp/peroxides_perchloric.html.

4. "Peroxide Formation in Chemicals"; University of California Davis; www.ehs.ucdavis.edu/sftynet/sn-23.html.

5. "Guidelines for Explosive and Potentially Explosive Chemicals Safe Storage and Handling"; University of California Berkeley; www.ehs.berkeley.edu/pubs/guidelines/pecguidelines.html.

6. "Peroxide Hazards"; Boston University; www.bu.edu/ehs.

7. "Peroxides and Peroxide Forming Compounds"; Donald E. Clark, Chemical and Biological Safety Officer; Texas A&M University.