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Analytical Techniques for Forensic Investigation of Oversized and Fragmented Exhibits in Mixed Explosive Detonations

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Analytical Techniques for Forensic Investigation of Oversized and Fragmented Exhibits in Mixed Explosive Detonations

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Abstract

Background/Objectives: Post-blast forensic investigations frequently involve oversized exhibits, such as vehicle fragments, concrete blocks, and metallic debris, which are often contaminated with mixed explosive formulations. These heterogeneous substrates present significant challenges for residue recovery and analysis when conventional methods, optimized for small, homogeneous samples are applied. This study aimed to develop and evaluate forensic strategies tailored to oversized and fragmented evidence from mixed explosive detonations. **Methods:** Oversized exhibits from ANFO–nitrate detonations were examined using sequential swabbing, solvent extraction, and spatial subsampling techniques. Organic residues were characterized using Thin Layer Chromatography (TLC) and Gas Chromatography–Mass Spectrometry (GC–MS), while inorganic ions were identified through chemical spot tests and Fourier Transform Infrared (FTIR) spectroscopy. The efficacy of syringe filtration was further assessed to improve the residue recovery efficiency. **Results:** GC–MS analysis of ether extracts confirmed the presence of high-boiling petroleum hydrocarbons, consistent with diesel fractions in ANFO. Inorganic analyses revealed the presence of ammonium and potassium nitrate, whereas chlorate, perchlorate, and metallic additives were absent. Spatial subsampling enhanced the detection sensitivity, and syringe filtration produced the highest recovery yield by minimizing background interference. **Conclusions:** The integrated workflow, which combines targeted swabbing, sequential solvent extraction, syringe filtration, and complementary analytical methods—proved highly effective for investigating oversized post-blast exhibits. This approach improves residue recovery, enhances analytical reliability, and strengthens evidentiary interpretation, providing a robust framework for the forensic investigation of complex detonation events in the future.

Keywords: forensic explosives; residue recovery; oversized exhibits; GC–MS; TLC; FTIR; ANFO; nitrate-based explosives; post-blast analysis

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1. Introduction

Forensic investigation of explosive incidents is critical for reconstructing events, identifying explosive formulations, and supporting judicial processes. Among the most widely encountered explosives are ammonium nitrate fuel oil (ANFO) and nitrate-based compounds, which are favored because of their accessibility, low cost, and high detonation efficiency [1,2]. Following detonation, residues from these materials disperse onto diverse substrates, including metallic fragments, soils, and concrete. Oversized and irregular debris present additional challenges owing to uneven residue distribution and contamination gradients [3].

Conventional extraction techniques, such as swabbing and solvent extraction, are widely used in forensic casework but are often insufficient for large-scale or heterogeneous exhibits, yielding low recovery rates and reduced sensitivity [4]. To overcome these limitations, novel approaches, including spatially resolved subsampling, syringe filtering, and gel-based sampling methods, have been proposed [5]. Instrumental methods remain central to residue characterization: gas

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chromatography–mass spectrometry (GC–MS) is the gold standard for organic explosive detection, whereas ion chromatography and Fourier transform infrared spectroscopy (FTIR) are widely applied for inorganic residues [6,7]. However, the success of these methods depends heavily on the effectiveness of sampling and extraction workflows.

This study aimed to evaluate integrated forensic strategies—combining sequential swabbing, solvent extraction, syringe filtering, and subsampling—applied to oversized debris from ANFO–nitrate detonations. By coupling these workflows with GC–MS, TLC, and FTIR, this study demonstrates a robust protocol for improving explosive residue recovery and strengthening forensic interpretation in complex post-blast scenarios.

2. Materials and Methods

2.1. Evidence Collection from the Crime Scene

The exhibits were collected from the crime scene where the accused person illegally stored explosive substances in a warehouse of his open property, and the same was exploded due to triggering. The blast damaged the main gate, compound wall of the warehouse and the blasting effect and it’s fragments damaged glasses and Roofs of the nearby houses. Exhibits of small scale to oversized were collected from the crime scene, such as metallic spades, metallic pots with a height of 12 in., defamed metallic pieces with a length of 29 in., metallic nails, Polythene, Nylon sheets, metallic wires, broken plastic sheets, plastic pipes, and multiple small-size fragments, as shown in Figure 1-6. Soil from the point of explosion and control soil were also collected for examination.

Figure 1. Metal piece having length of 29 in. with other fragments.

Figure 2. Multiple size fragments.

Figure 3. Multiple size metal and plastic fragments.

Figure 4. Metallic spade.

Figure 5. Pot having height 12 in. and inner diameter 9 in.

Figure 6. Defamed oversized metallic piece.

2.2. Exhibits Preparation

The Laboratory received oversized exhibits in four parcels and two envelopes for soil samples. Diethyl Ether was obtained from Finar, Acetone (AR) was obtained from Advent Chembio Pvt. Ltd, Sodium Hydroxide, and Pyridine was obtained from SRL. Demineralized (DM) water procured from Labogen Fine Chem Industry, Ludhiana was used for Water and Alkali extraction. Whatman-42 filter paper was used for filtration. Allpure Nylon Syringe filter (pore size 0.22 μm) was procured from Membrane Solutions and used for the filtration of Ether and Acetone extracts. [10,11]

2.3. Extracts Collection and Analysis

All the small exhibits and their fragments were properly extracted using Ether, Acetone, Water, Sodium Hydroxide and Pyridine to identify both In-Organic and Organic Explosive traces. The extraction was collected from the oversized exhibits using the swabbing method, as shown in Figure 7. A Surgical syringe of 10 ml capacity and Kapas Absorbent cotton wool I.P. manufactured by Aster Corporation were used for the collection of extracts by the swabbing method. Complete swabbing was performed for the collection of Ether, Acetone, Water, Sodium Hydroxide and Pyridine extracts sequentially, as per the procedure [8,9].

Figure 7. Extraction collection by swabbing method.

A schematic diagram of the examination of all extracts is shown in Figure 8. In the Post-Blast phase, the collected exhibits will be heterogeneous, and the identification of the explosive ingredients will be challenging because of their uneven deposition. Therefore, the filter papers, syringe filters, cotton used for swabs, funnels and beakers used for the extraction of Ether extract from a particular exhibit should also be used for the extraction of Acetone, water, Sodium Hydroxide and Pyridine extracts. The extracts collected from the small and oversized samples were filtered using a syringe filter to remove solid impurities. The filtrate was collected in a 100 ml beaker and concentrated upto 2-5 ml by evaporation at room temperature. The Ether filtrates were analyzed using GC-MS for Diesel oil (hydrocarbons) to identify Ammonium Nitrate Fuel Oil (ANFO). Acetone filtrates were analyzed using TLC and GC-MS to identify Organic High Explosives. Water and Sodium Hydroxide Extracts were analyzed by chemical examination, FTIR, and IC for In-Organic Explosive ingredients. Elemental Sulphur will be identified through Pyridine extract.[8–11]

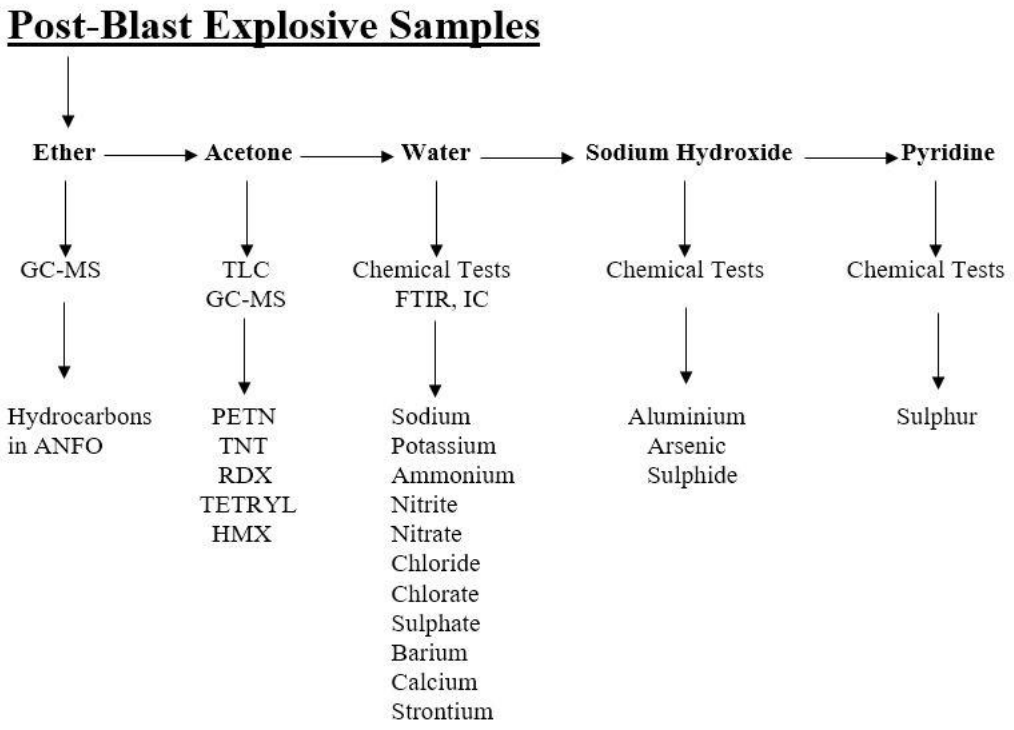

Figure 8. Schematic diagram for Post-Blast Explosive Analysis.

2.4. Thin Layer Chromatography

Silica gel 60G F254 Plates with a thickness of 200 micrometer and size of 20 x 20 cm were used for Thin Layer Chromatography (TLC) analysis. Chloroform, Acetone, Toluene and Cyclo hexane used for the test.[8–11]

Pre-coated TLC plates were activated by placing them in an air oven at 110°C for 30 minutes. One hundred milliliters of solvent [chloroform : acetone (1:1) and toluene : cyclohexane (7:3)] was taken in two different developing chambers (for 20 × 20 cm TLC plates), covered with a lid, and allowed to saturate for at least 30 min. The concentrated acetone extract of each sample was spotted on a pre-coated TLC plate along with reference standards of high explosives, leaving 2 cm from one edge at the bottom of the TLC plate and maintaining a minimum distance of 1.5 cm between two spots. The TLC plate was placed vertically in the developing chamber and allowed to develop until the solvent front rose to 10 cm from the spots by capillary action. After completion, the plate was removed and left at room temperature for the eluent to evaporate.[8–11]

The TLC plate was developed by spraying with 5% diphenylamine (DPA) in 95% ethanol, and the colour produced was noted. The plate was then placed under UV light (254 nm) to observe fluorescence and subsequently sprayed with concentrated sulphuric acid, and the resulting colours were recorded. The colours were compared with the Rf values for identification.[8–11]

2.5. Fourier Transform Infrared Spectroscopy (FTIR)

The Ether, Acetone and dried water extracts were examined using a Thermo Fisher Scientific Nicolet iS20 FTIR spectrometer instrument, which equipped with an IR source, an attenuated total reflectance (ATR) accessory, a DTGS detector and KBr beam splitter from Thermo Fisher Scientific. The instrument was operated at resolution of 4.000 between wavenumber 4000 cm-1 to 400 cm-1. The analysis was performed by scanning the background and sample using the Thermo Scientific OMNIC software. The sample was scanned 64 times to obtain a characteristic spectrum. The spectrum was searched using correlation search type in the libraries of the instrument to identify the sample.[8–11]

2.6. Challenges in the Analysis of Oversized Exhibits

Extraction from the exhibits of smaller sizes and their debris is possible by rinsing the exhibit with a minimum quantity of solvent in a beaker. In this method, the ingredients of the unexploded and exploded explosives can be easily collected from the exhibits by dissolution. However, in the case of oversized exhibits, the only possible way to collect the extract is by the swabbing method. Many factors, such as, the quality of the cotton, human error, spillage, and the collection of extract from the swab, will affect this method during the collection of the extracts. Care should be taken in the swabbing method to minimize these errors.

3. Observations

3.1. TLC and GC-MS Examinations

High-boiling fractions of petroleum hydrocarbons identified in the GC-MS analysis of the Ether extracts of the exhibit are shown in the Figure 1. The compound name was identified by the GC-MS library as Hexadecane at the RT 14.02 having SI value of 792 and RSI value is 929. The GC-MS spectrum has a peak area of 136911064.09 and peak height of 37343910.88, as shown in Figure 9 of the Total Ion Chromatogram (TIC).

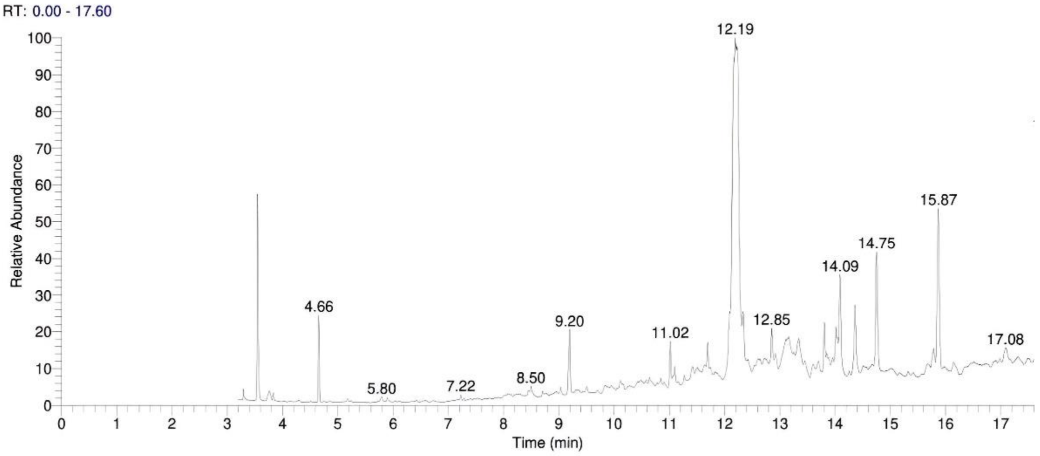

Figure 9. TIC of the exhibit having hydrocarbons.

The mass spectra of the forensic case sample obtained by analysis using the GC-MS method, along with the reference library of mass spectra, are shown in Figure 10. The x axis presents abundance and y axis presents m/z values.

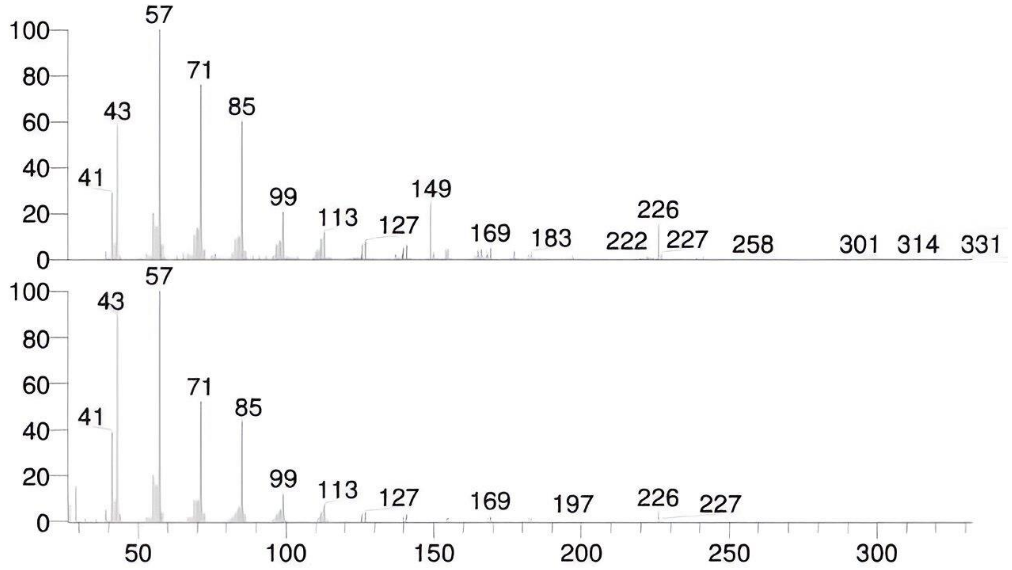

Figure 10. Mass spectrum of the Extract (Top) and it's NIST library (Bottom).

No High Explosives were identified through Chemical Examination, TLC and GC-MS of the Acetone Extract.

3.2. Chemical Examinations

The Low Explosives identified in the Water, Alkali and Pyridine extracts of the smaller-sized and oversized exhibits are listed Table 1. Except for the control soil, all the exhibits yielded positive results for low explosives.

Table 1. Observations of the Chemical Examinations.

Sl.No	Chemical Test	Target Ion/Analyte	Observation
1	Silver Nitrate	Chloride	Present
2	Griess Test	Nitrite	Present
3	Griess reagent + Zn dust	Nitrate	Present
4	Aniline sulphate	Chlorate	Absent
5	Methylene blue indicator	Perchlorate	Absent
6	Barium chloride	Sulphate	Present
7	Zinc Uranyl Acetate	Sodium	Absent
8	Sodium Cobaltinitrate	Potassium	Present
9	Nessler's reagent	Ammonium	Present
10	Magneson-I	Magnesium	Absent
11	Sodium Rhodizonate	Barium, Calcium, Strontium	Absent
12	Sodium Nitroprusside	Sulphide (NaOH Extract)	Absent
13	Alizarine-S	Metallic Aluminium (NaOH Extract)	Absent
14	Gutzeit's Test	Arsenic (NaOH Extract)	Absent

15	Pyridine + NaOH	Elemental Sulphur	Absent

3.3. FTIR Analysis

High Explosives were not identified in the FTIR analysis of the Acetone Extract. However, Potassium Nitrate and Ammonium Nitrate (Low Explosives) were identified in the Water Extract. The FTIR spectra are shown in Figure 11 and 12.

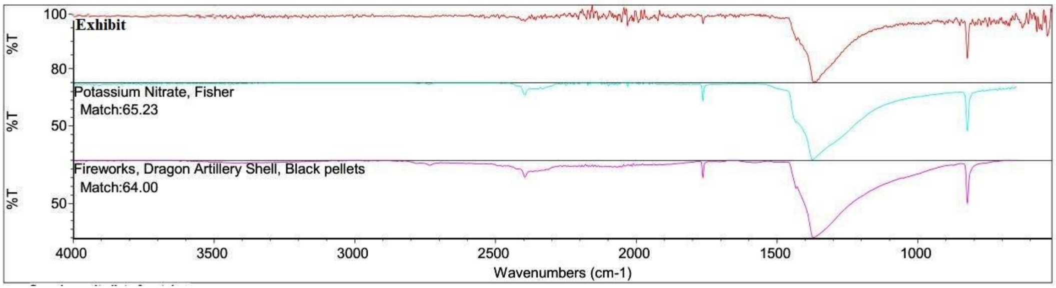

Figure 11. FTIR spectrum of Potassium Nitrate with Library search.

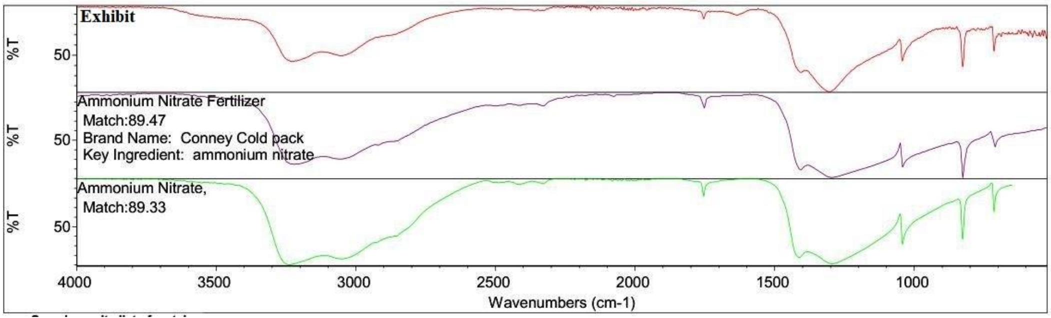

Figure 12. FTIR spectrum of Ammonium Nitrate with Library search.

The presence of high-boiling fractions of petroleum hydrocarbons and Ammonium Nitrate confirms the presence of Ammonium Nitrate Fuel Oil (ANFO) in the Oversized Exhibits.

4. Results and Discussions

The forensic investigation of oversized and fragmented exhibits from the mixed ANFO–nitrate detonation provided consistent evidence of explosive residues across several analytical platforms.

4.1. Organic Residue Detection (GC–MS and TLC)

GC–MS analysis of ether extracts confirmed the presence of high-boiling petroleum hydrocarbons, with hexadecane identified at a retention time of 14.02 min. This finding is characteristic of diesel oil fractions, supporting the presence of fuel oil in the ANFO formulations. The spectral match with the NIST library provided high similarity indices, indicating robust identification [1]. These results align with those of prior studies that demonstrated the reliability of GC–MS in detecting post-blast ANFO residues despite environmental degradation and substrate heterogeneity. Notably, no high explosives were detected in the Acetone extracts by TLC or GC–MS, suggesting that the device primarily relied on ANFO rather than high-explosive admixtures.

4.2. Inorganic Residue Detection (Chemical Tests and FTIR)

Chemical spot tests revealed positive results for nitrite, nitrate, ammonium, chloride, potassium, and sulphate ions across multiple exhibits, while chlorate, perchlorate, and metallic salts such as aluminium and magnesium were absent. These findings corroborate the presence of nitrate-based low explosives, consistent with ANFO residues. FTIR analysis further confirmed the ammonium nitrate and potassium nitrate signatures in the water extracts [7], reinforcing the outcome of the chemical examinations. Such multi-method corroboration strengthens the evidentiary value by reducing false positives and analytical uncertainties, as emphasized in previous forensic residue studies.

4.3. Workflow Efficacy in Oversized Exhibits

The study demonstrated that sequential swabbing followed by solvent extraction and syringe filtration maximized residue recovery efficiency. The syringe filter method, in particular, improved extract clarity and reduced interference in GC–MS runs [4]. These findings agree with recent advances highlighting that adapted workflows—such as spatially resolved subsampling and filtration—enhance residue detection from heterogeneous matrices.

4.4. Forensic Interpretation

The combined detection of hydrocarbons and nitrate ions confirms the presence of

ANFO in the oversized exhibits, thereby directly linking the recovered fragments to the explosive formulation used for detonation. Importantly, the absence of high explosives suggests the deliberate use of bulk ANFO rather than a composite device [3]. These results are consistent with the reported global trends in improvised explosive devices, where ANFO remains the dominant formulation owing to its accessibility and effectiveness.

5. Conclusions

This study demonstrates the effectiveness of an integrated forensic protocol for investigating oversized and fragmented exhibits from mixed ANFO–nitrate detonations in a field setting. By combining sequential swabbing, solvent extraction, syringe filtration, and complementary analytical techniques (GC–MS, TLC, chemical spot tests, and FTIR), the reliable detection of both organic (fuel oil hydrocarbons) and inorganic (nitrate-based) residues was achieved.

The results highlight three key conclusions:

1. The residue recovery efficiency was maximized by adapting the workflows to oversized exhibits, with syringe filtration proving particularly valuable.
2. Analytical corroboration across GC–MS, TLC, chemical tests, and FTIR reinforced evidentiary strength and reduced uncertainty.
3. Forensic reconstruction confirmed the exclusive use of ANFO, providing insights into the nature of the explosive device and its deployment strategy.

These findings emphasize that forensic protocols must be tailored to the scale and heterogeneity of post-blast exhibits to ensure accurate residue recovery and it’s interpretation. The methodological advances described herein contribute to strengthening forensic investigations of large-scale detonation events and support judicial processes by providing scientifically validated evidences.

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Conflicts of Interest: The author declares no conflicts of interest

Abbreviations

The following abbreviations are used in this manuscript:

TLC	Thin Layer Chromatography
GC-MS	Gas Chromatography Mass Spectrometry
FTIR	Fourier Transform Infrared Spectroscopy
IC	Ion Chromatography
in.	inches
TIC	Total Ion Chromatogram
NIST	National Institute of Standards and Technology
AR	Analytical Reagent
DM	De-Mineralized
ATR	Attenuated Total Reflectance
ANFO	Ammonium Nitrate Fuel Oil
PETN	Penta Erythritol Tetra Nitrate
TNT	Tri Nitro Toluene
RDX	Research Department Explosive/ Royal Demolition Explosive
NG	Nitro Glycerin
TETRYL	Trinitrophenylmethylnitramine
HMX	High Melting eXplosive - Octogen
DPA	Di Phenyl Amine
UV	Ultra Violet
DTGS	Deuterated Tri Glycine Sulfate
IR	Infra Red
NaOH	Sodium Hydroxide

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