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NEW DELHI**

**WORKING PROCEDURE MANUAL: EXPLOSIVE  
2021**

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## **FOREWORD**

Forensic Sciences have a critical role to play in criminal justice delivery system. Successful prosecution of offenders hinges on the quality of test reports. Exhibits in crime cases need to be examined timely, precisely and accurately. In order to standardise and benchmark forensic analysis and test reports in respect of crime cases in Forensic Science Laboratories across the country, the Directorate of Forensic Science Services (DFSS), Ministry of Home Affairs has taken the initiative to prepare Working Procedure Manuals (WPMs) for various forensic disciplines.

Due to advancement of technology and its global access, crime is continuously evolving and adapting. New crime trends are emerging with people committing crimes in cyberspace, trafficking occurring in new psychoactive substances and drugs etc. Therefore, incorporation of new technologies in the WPMs to meet the ever increasing challenges for solving crime is the need of the hour, which is systematically and comprehensively reflected in these WPMs.

A long-felt need for such uniform WPMs has been fulfilled and I hope that these will be of immense use to forensic professionals of India. I congratulate the DFSS team for developing these manuals and urge them to keep on updating these at regular intervals.

(Punya Salia Srivastava)



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## **PREFACE**

The analytical procedures for examination of forensic physical clue materials in forensic science laboratories involve high degree of skill & expertise and play a significant role in a wide range of legal proceedings. The occurrence of error(s) in any of the forensic analytical activities is a serious matter for both laboratories and end users. For a laboratory, it can lead to re-testing of samples, if available, and loss of its credibility. The analytical techniques adopted by the scientist(s) for the forensic analysis may be one of the causes for this serious error.

The risk of committing error can be eliminated if the scientists undertake two or more independent validated techniques while conducting forensic analysis of crime case exhibits in the laboratory. Essentially, the procedures adopted must conform to the quality, sensitivity, repeatability and reproducibility of the examination so that the chances of error are absolutely avoided. It is, therefore, one of the essential requirements of good laboratory practices to introduce a Working Procedure Manual, which contains validated laboratory methods/techniques for forensic analysis of the exhibits. It is also necessary for all the Central/State Forensic Science Laboratories to follow these manuals in the country to maintain uniformity in test reports.

Keeping in view the advancement in science & technology and use of various protocols & procedures in the international arena of forensic science, the Directorate of Forensic Science Services (DFSS), Ministry of Home Affairs(MHA), has taken the initiative for preparing a systematic and comprehensive Working Procedure Manual for the discipline of 'Forensic Explosives' to bring uniformity and standardization in the examination methods. In this regard, DFSS/MHA formed Scientific Working Groups, comprising eminent forensic scientists from the CFSLs and FSLs for each forensic discipline to compile forensic analytical techniques in the form of Working Procedure Manuals. Several meetings were conducted with detailed deliberations among the scientists at National level and finally the manual has been prepared /updated in the present form.

I am sure that this Working Procedure Manual, which pertains to the discipline of 'Forensic Explosives' will help the forensic science laboratories to continue to follow standard and latest updated procedures in the examination of clue materials as well as to adopt quality control/ quality assurance in the forensic practices and also for obtaining accreditation from National Accreditation Board for Testing and Calibration of Laboratories (NABL).

I understand that there is always a scope of improvement and perfection can be achieved with collective efforts, therefore, stakeholders are welcome to offer their feedback and suggestion, if any, in this regard.

  
( Dr S K Jain)

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## **0.1 ABBREVIATIONS**

AAS–Atomic Absorption Spectrometry

ANFO -Ammonium Nitrate Fuel Oil

CE- Capillary Electrophoresis

DDNP-Diazodinitrophenol

DFSS-Directorate of Forensic Science Services

DNT-Dinitrotoluene

EDX-Energy dispersive X-Ray

EGDN-Ethyleneglycoldinitrate

FT-IR-Fourier Transform Infrared Spectroscopy

GC-Gas Chromatography

GC-IMS- Gas Chromatography-Ion Mobility Spectrometry

GC-MS-Gas Chromatography- Mass Spectrometry

HMTD-Hexamethylene tri peroxide diamine

HMX-Cyclotrimethylene tetra nitramine

HPLC-High Performance Liquid Chromatography

HPTLC-High Performance Thin layer Chromatography

IED-Improvised Explosive Device

IC- Ion Chromatography

ICP-Inductively Coupled Plasma

IR-Infrared spectroscopy

LC-MS-MS –Liquid Chromatography Tandem Mass Spectrometry

Lead Azide-Pb(N<sub>3</sub>)<sub>2</sub>

Lead trinitroresorcinate- Lead Styphnate

LOX- Liquid Oxygen Explosive

Mercury Fulminate-(Hg (ONC)<sub>2</sub>)

NC- Nitrocellulose

NG-Nitroglycerine

PETN- Pentaerythritol tetra nitrate

RDX- Cyclotrimethylenetrinitramine

Picric Acid -2,4,6-trinitro phenol

SEM-Scanning Electron Microscopy

TATP- Triacetone triperoxide

Tetrazene-1(5-tetrazylol)-4-guanyl tetrazene hydrate

Tetryl (CE)-2,4,6-trinitrophenyl methyl nitramine

TLC- thin Layer Chromatography

TNT-2,4,6-trinitro toluene

XRD-X –Ray Diffraction

XRF-X ray Fluorescence



## Chapter-1

### INTRODUCTION TO EXPLOSIVES

**1.1 Title:** Introduction to Explosive Materials

**1.2. Scope:** General information on about and classification of Explosive materials.

**1.3 Purpose:** To have a knowledge about the Nature, uses, characteristics, Classification composition, chemical nature, Physical and chemical properties of explosive materials.

**1.4 Nature of Explosives: -**

Explosives are reactive substances that undergo rapid burning (deflagration) or detonation resulting in the formation of large volumes of gases, liberation of heat and light along with sudden pressure effects (Shock wave and blast wave). These substances have a large amount of potential energy. The conversion of potential energy to kinetic energy with production of light, heat, sound and pressure simultaneously is called explosion.

**1.5 Uses of Explosives:-**

Explosives are used for both commercial/Industrial and military applications. Commercial use of explosives includes demolition for construction, fireworks and metal forming etc. Military use of Explosives is for security purposes. It includes rockets, missiles, bombs etc. Apart from that antisocial elements use explosives in the form of Improvised explosives devices to create havoc and instability.

**1.6 Categories of Explosives:-**

Generally, there are three categories of explosives i.e Chemical, Mechanical and Nuclear explosives. (Ref.1 page 10).

**Chemical Explosives** are compounds or mixtures of compounds that react to produce large volumes of rapidly expanding gases as well as energy in the form of heat light and shockwaves that exert sudden pressure on the surroundings.

**Mechanical Explosives** are those substances that tend to undergo physical change such as overloading of container with compressed air stream.

**Nuclear Explosives** are the most powerful explosives. They are reproduced by sustained nuclear reactions while releasing a tremendous amount of heat and energy.

For Forensic interest Chemical explosives are of Major concern as these are used in Improvised explosive devices for terrorist activities. This manual deal mainly with Chemical explosives.

### 1.7 Characteristics of Chemical Explosives:-

All explosive compounds can be considered to be composed of three components namely Fuel, Oxidizer and Sensitizer. Carbon, Hydrogen, Sulphur etc. provide the essential fuel for the oxygen in the oxidizer. Incorporation of a chemical or physical sensitizer enhances the ease with which the explosive can be made to react by means of an initiator. (Ref2,page17).

The characteristics of chemical explosives influence the type of explosives that are used for a specific application. Such characteristics include, but not limited to sensitivity, stability, rate of detonation and brisance.

### 1.8 Classification of Explosives:-

Chemical explosives are classified in many ways according to different criteria.

1. **According to rate of decomposition**- High explosives and Low explosives

2. **According to chemical structure:** - The most important class includes organic compounds, which contain the nitro (NO<sub>2</sub>) group. They are sub divided according to the atom to which the NO<sub>2</sub> group is attached.

Nitro compounds - C-NO<sub>2</sub> group,

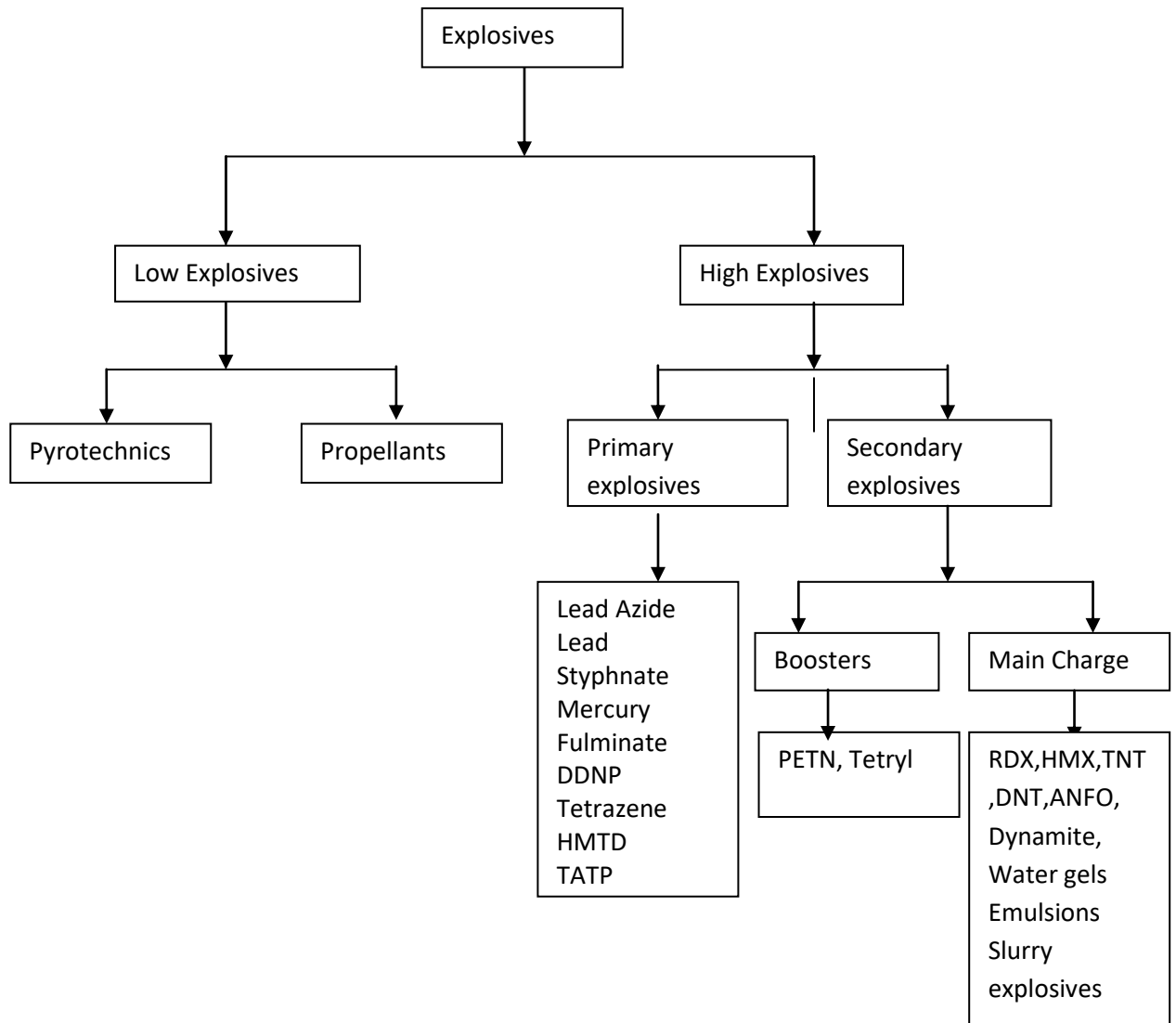
Nitrate esters - C-O-NO<sub>2</sub> group

Nitramines - C-N-NO<sub>2</sub> group.

3. **According to application:** - Industrial explosives and Military explosives. (Ref 3, page1).

Figure-1 is a classification of chemical explosives with representative examples. (Ref 3 page 2)

**CLASSIFICATION (BASED ON RATES OF DECOMPOSITION)**



*Figure-1 Classification of explosives*

## 1.9 Characteristics of High and Low Explosives:-

Table-1 – Characteristics of High and low Explosives

| Sr.No | Characteristics  | HIGH EXPLOSIVES   | LOW EXPLOSIVES                           |
|-------|--|---|--|
| 1.    | Method of initiation   | Primary explosives by ignition and Secondary explosives by detonation | By ignition                              |
| 2.    | Time of complete conversion of explosive to gaseous products | Micro seconds   | Milli seconds                            |
| 3.    | Velocity of consumption of explosives grains                 | 1.5 to 10 km s <sup>-1</sup>  | Few centimeters to meter s <sup>-1</sup> |
| 4.    | Velocity of flame front                                      | 1.5 to 10 km s <sup>-1</sup>  | 0.5 to 1.5 km s <sup>-1</sup>            |
| 5.    | Pressure of explosion  | 3,50 to 27,600 kpa  | Upto 350 kpa                             |
| 6.    | Exploded in gun  | Shatters gun  | Good propellant                          |
| 7.    | Demolition   | Excellent to poor   | Nil                                      |
| 8.    | Uses   | Demolition, blasting  | Propellant, blasting                     |
| 9.    | Velocity of Detonation (VOD)                                 | More than 1000 m s <sup>-1</sup>                                      | Less than 1000 m s <sup>-1</sup>         |

## 1.10 High Explosives

High explosives are detonating explosives. Detonation is the explosion effect that is caused by the transmission of high speed shock wave when the explosive compound or mixture of compounds decomposes and liberates energy. The chemical reaction propagates with such rapidity that the rate of reaction zone into the material exceeds velocity of sound and is greater than 1000m/s. High explosives are further sub-divided in to two groups according to their susceptibility to initiation i.e. Primary explosives and Secondary explosives. (Figure-1)

### 1.10.1 Primary HighExplosives:

Primary explosives are among the most powerful and most sensitive of all chemical explosives. These are the explosives which are used to start or initiate the explosion. These compounds are easily detonated by mechanical shock, friction and heat and readily ignited by direct contact with flame or electrical sparks. The main function of Primary explosives is to initiate a secondary explosive by shock wave. Because of their sensitivity they are used in small quantities. Initiators are mainly used in military detonators, industrial blasting caps and stab, and shock primers. (ref 3, page15 ). Examples of primary explosives are Mercury Fulminate, lead Azide, Lead Styphnate, Dizodinitrophenol (DDNP), Tetrazine etc.

#### 1.10.1.1 Mercury Fulminate :

Mercury fulminate ( $\text{Hg}(\text{ONC})_2$ ) is a heavy non hygroscopic crystalline solid. It is thermally unstable and very sensitive to impact, friction, spark, flame and shock when dry. It reacts with metals such as aluminium, magnesium, zinc, brass and bronze in moist atmosphere. Being a mercury derivative, it is a toxic compound. (Ref 3, page 15) and has been replaced by other primary explosives which are less toxic and more stable. Some properties of compound is given in Table-2

Table-2 properties of Mercury Fulminate

|                    |   |
|--------------------|---|
| Colour             | Colourless crystals                       |
| Solubility         | Practically insoluble in water            |
| Empirical formula  | $\text{C}_2\text{N}_2\text{O}_2\text{Hg}$ |
| Molecular weight   | 284.6                                     |
| Deflagration point | $165^{\circ}\text{C}$                     |
| Impact sensitivity | 0.1-0.2 kp                                |

### 1.10.1.2 Lead Azide: $Pb(N_3)_2$

Lead azide has a high temperature of ignition and less sensitive to shock and friction than Mercury fulminate. It has good stability to heat and storage. Due to its high temperature of ignition it is generally used in primary mixtures with Lead styphnate which is very easy to ignite. Its contact with copper must be avoided because it leads to the formation of Copper azide. Since it is a non-conductor it may be mixed with flaked graphite to form a conductive mixture for use in low energy electrical detonator. (ref 3, page15).Some properties of compound is given in Table-3

Table-3 properties of Lead Azide

|                    |                          |
|--------------------|--------------------------|
| Colour             | White buff powder        |
| Solubility         | insoluble in water       |
| Empirical formula  | $Pb(N_3)_2$              |
| Molecular weight   | 291.3                    |
| Deflagration point | 320 - 360 <sup>0</sup> C |
| Impact sensitivity | 0.25-0.4 kpm             |

### 1.10.1.3 Lead Styphnate (Lead trinitroresorcinate)

Lead styphnate is a toxic explosive which is thermally stable, non-corrosive and non-hygroscopic. It is sensitive to flame and electrostatic discharge. It is therefore used to sensitize lead azide and initiate burning in primer compositions. (ref 3, page 16 ). Chemical structure of compound is given in figure-2 and some properties of compound is given in Table-4.

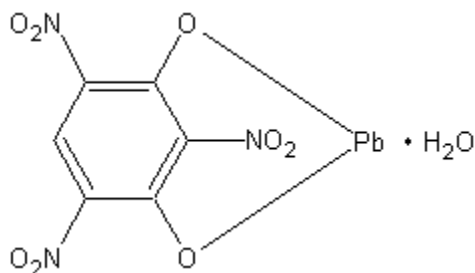


Figure-2- Chemical structure of compound

Table-4- Properties of Lead Styphnate

|                    |  |
|--------------------|--|
| Colour             | Reddish brown crystals                           |
| Solubility         | insoluble in water                               |
| Empirical formula  | C <sub>6</sub> HN <sub>3</sub> O <sub>8</sub> Pb |
| Molecular weight   | 468.3  |
| Deflagration point | 275 - 280 <sup>0</sup> C                         |
| Impact sensitivity | 0.25-0.5kpm                                      |

#### 1.10.1.4Tetrazene:

Tetrazene (1(5-tetrazylol)-4-guanyl tetrazene hydrate) is a colorless to yellowish solid which is used for sensitizing primary composition. It is slightly hygroscopic and explodes readily from flame, producing a large amount of smoke. It decomposes in boiling water. It ignites readily and is slightly more sensitive to impact than mercury fulminate. Its main use is for the sensitization of priming compositions. (ref 3, page 16).). Chemical structure of compound is given in figure-3 and some properties of compound is given in Table-5.

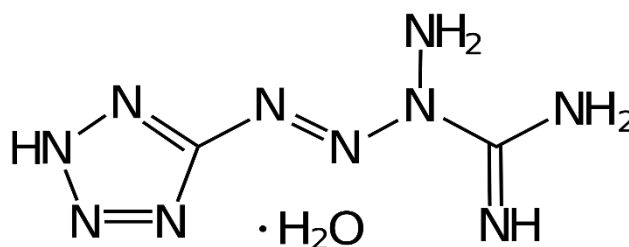


Figure-3- Chemical structure of compound

Table-5- Properties of Tetrazene

|                    |  |
|--------------------|--|
| Colour             | Pale yellow crystals   |
| Solubility         | insoluble in water   |
| Empirical formula  | C <sub>2</sub> H <sub>6</sub> N <sub>10</sub> . H <sub>2</sub> O |
| Molecular weight   | 188.2  |
| Deflagration point | 140 <sup>0</sup> C   |
| Impact sensitivity | 0.1kpm   |

### 1.10.1.5 Diazodinitrophenol(DDNP):-

DDNP is yellowish brown powder which is used for initiating high explosives in propellant primary devices. The sensitivity of DDNP to friction is about the same as Lead azide but less than Mercury fulminate. DDNP is used to form priming mixture where a high sensitivity to flame or heat is desired. Some properties of compound is given in Table-6.

Table-6 Properties of DDNP

|                   |                    |
|-------------------|--------------------|
| Colour            | yellow crystals    |
| Solubility        | insoluble in water |
| Empirical formula | $C_6H_2N_4O_5$     |
| Molecular weight  | 210.11             |

### 1.10.2 Secondary High Explosives

Secondary explosives are generally used as the main charge. These are relatively insensitive to heat, flame and shock. They usually require a primary explosive to initiate detonation. Secondary high explosives include boosters and main charge. The most commonly used booster is PETN. TNT, RDX, HMX, Dynamites, Binary explosives, emulsions, watergel etc. are used as main charge. The most important class of main charge is the organic compounds which contain the nitro group.

#### 1.10.2.1 Picric acid (2,4,6-trinitrophenol ):-

It was used in large caliber shells in world war I. In world war II a mixture of ammonium picrate and TNT was widely used in the press loading of armour piercing shells.(ref 2, pages 3-4).Chemical structure of compound is given in figure-4 and some properties of compound is given in Table-7.

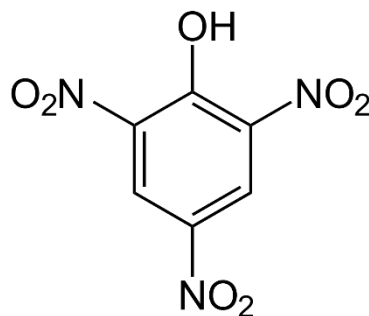


Figure-4- Chemical structure of Picric Acid



Table-7- Properties of Picric Acid

|                      |                                  |
|----------------------|----------------------------------|
| Colour               | yellow crystals                  |
| Solubility           | soluble in water, ether, alcohol |
| Empirical formula    | $C_6H_3N_3O_7$                   |
| Molecular weight     | 229.1                            |
| Deflagration point   | $300^{\circ}C$                   |
| Impact sensitivity   | 0.75kpm                          |
| Solidification point | $122.5^{\circ}c$                 |

#### 1.10.2.2 TNT(2,4,6-trinitrotoluene) :

TNT is one of the most commonly used High explosive in military weapons and in civilian mining. It is frequently used as main charge in artillery projects, mortar rounds and aerial bombs used military explosives. Its main features include low melting point, good chemical and thermal stability, low sensitivity to impact, friction and high temperature and its compatibility with other explosives. Chemical structure of compound is given in figure-5 and some properties of compound is given in Table-8

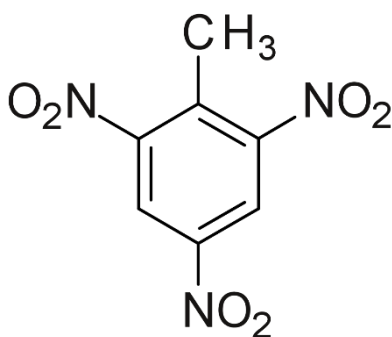


Figure-5- Chemical structure of TNT

Table-8- Properties of TNT

|                      |  |
|----------------------|--|
| Colour               | Pale yellow crystals and flakes                              |
| Solubility           | insoluble in water, soluble in benzene, toluene and acetone. |
| Empirical formula    | $C_7H_5N_3O_6$   |
| Molecular weight     | 227.1  |
| Deflagration point   | $300^{\circ}C$   |
| Impact sensitivity   | 1.5kpm   |
| Solidification point | $80.8^{\circ}c$  |

1.10.2.3 **Tetryl(2,4,6-trinitrophenylmethylnitramine ):**

It is used in military boosters. (ref 2, page 4).Chemical structure of compound is given in figure-6 and some properties of compound is given in Table-9.

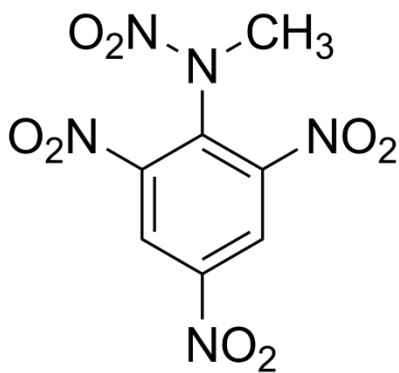


Figure-6- Chemical structure of Tetryl

Table-9- Properties of Tetryl

|                    |   |
|--------------------|---|
| Colour             | Pale yellow crystals                    |
| Solubility         | Insoluble in water, soluble in acetone. |
| Empirical formula  | $C_7H_5N_5O_8$                          |
| Molecular weight   | 287.1                                   |
| Deflagration point | 185 <sup>0</sup> C                      |
| Impact sensitivity | 0.3kpm                                  |

#### 1.10.2.4 PETN(Pentaerythritoltetranitrate):

PETN is one of the strongest known High explosive. It is used as a base charge in blasting caps and detonators, as the core explosives in detonating cord, in booster charges, in plastic explosives, and as an ingredient in other explosives. The chemical stability of PETN is very high and is considered to be more stable than all other nitrate esters. (ref 3, pages 9-10). Chemical structure of compound is given in figure-7 and some properties of compound is given in Table-10.

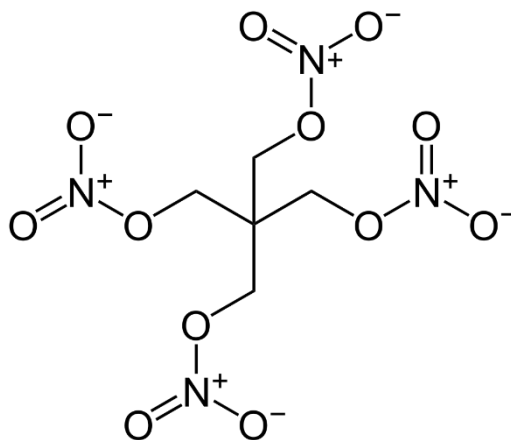


Figure-7- Chemical structure of PETN

Table-10- Properties of PETN

|                    |  |
|--------------------|--|
| Colour             | colourless crystals  |
| Solubility         | insoluble in water, soluble in acetone and methyl acetate    |
| Empirical formula  | C <sub>5</sub> H <sub>8</sub> N <sub>4</sub> O <sub>12</sub> |
| Molecular weight   | 316.1  |
| Deflagration point | 202 <sup>0</sup> C   |

#### 1.10.2.5 RDX (Cyclotrimethylenetrinitramine):

It is also known as cyclonite and hexogen. It is one of the most important military explosives used today. It has high chemical stability and an explosive power. RDX is considerably more susceptible to shock than TNT for detonation. RDX is used as a component in mixtures with other explosives such as TNT and as a plastic explosive also. A well-known plastic explosive, Semtex is based on RDX and PETN. Mixtures of RDX and wax are used for booster charges in many military ammunitions especially in artillery shells. (ref 3, pages 5-6). Chemical structure of compound is given in figure-8 and some properties of compound is given in Table-11.

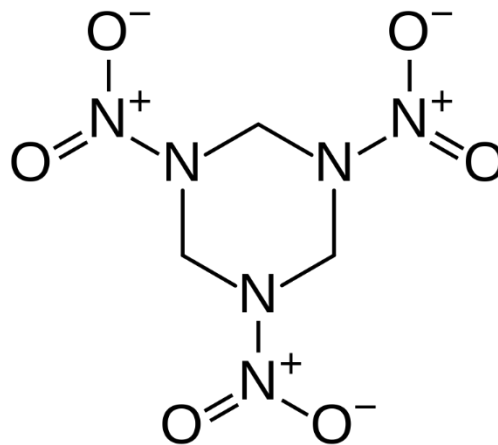


Figure-8- Chemical structure of RDX

Table-11- Properties of RDX

|                    |   |
|--------------------|---|
| Colour             | Colourless crystals                                       |
| Solubility         | insoluble in water, soluble in acetone and methyl acetate |
| Empirical formula  | $C_3H_6N_6O_6$  |
| Molecular weight   | 222.1   |
| Deflagration point | $204^{\circ}C$  |
| Impact sensitivity | 0.75kpm   |

#### 1.10.2.6 HMX(Cyclotetramethylenetetranitramine):

HMX is also known as octogen. It explodes violently at high temperatures. It is used as a component of rocket propellant. HMX exists as  $\alpha$  (orthorhombic),  $\beta$  (monoclinic),  $\gamma$  (monoclinic) and  $\delta$  (hexagonal) forms, of which ' $\beta$ ' form is the least sensitive to impact and the most stable (ref 3, page 7). Chemical structure of compound is given in figure-9 and some properties of compound is given in Table-12.

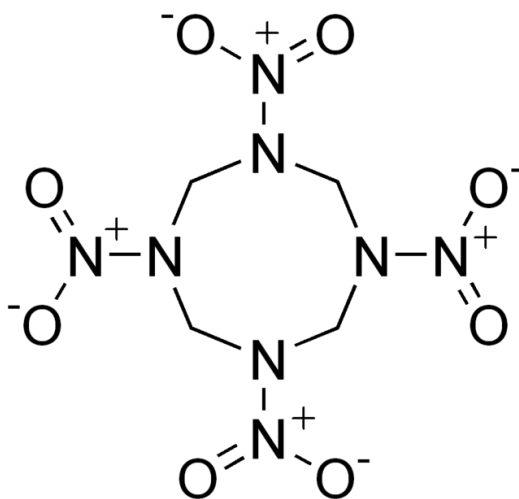


Figure-9- Chemical structure of HMX

Table-12- Properties of HMX

|                    |  |
|--------------------|--|
| Colour             | colourless crystals  |
| Solubility         | insoluble in water,<br>sparingly soluble in acetone and ether. |
| Empirical formula  | $C_4H_8N_8O_8$   |
| Molecular weight   | 296.2  |
| Deflagration point | $287^{\circ}C$   |
| Impact sensitivity | 0.75kpm  |

### 1.10.2.7 Nitroglycerine (NG):

Nitroglycerine is the most powerful explosive for commercial use. It is a key explosive ingredient used to produce dynamites and rocket propellant. It is very sensitive to shock, impact and friction and used only when desensitized with other liquids or absorbent solids or when mixed with nitrocellulose. (ref 3, page 8). Chemical structure of compound is given in figure-10 and some properties of compound is given in Table-13.

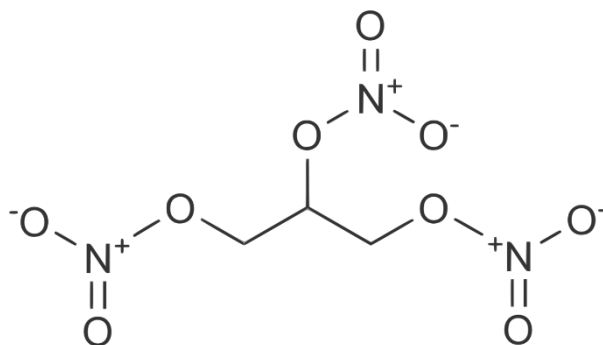


Figure-9- Chemical structure of NG

Table-13- Properties of NG

|                      |  |
|----------------------|--|
| Colour               | Brown crystals   |
| Solubility           | insoluble in water, spirangly soluble in carbon disulphide . |
| Empirical formula    | $C_3H_5N_3O_9$   |
| Molecular weight     | 227.1  |
| Solidification point | 13.2 <sup>0</sup> C  |
| Impact sensitivity   | 0.02kpm  |

**1.10.2.8 Dynamite:**

Over the years, formulas for dynamite have been developed to hit every type of rock blasting. Varieties have been developed for severe water conditions utilizing nitro cotton to gel the NG (the gelatins are one branch of the dynamite family) for cohesiveness to enable loading into up holes in mines, for safe usage in under- ground coal mines (the permissible having salts of various types to cool the explosive reaction as part of formula) and for economics by making formulas with very high ammonium nitrate percentages (and thus no cohesiveness or water resistance) where severe field conditions do not exists. Literally thousands of different formulas might be found, but for years all had the commonality of having nitroglycerine as a component. Some typical formulations have been shown in Table below (ref 2, pages 6-7).

Table-14 Dynamite formulations- generalized percent by weight

| Material         | Type* |      |      |      |      |
|------------------|-------|------|------|------|------|
|                  | 1     | 2    | 3    | 4    | 5    |
| Nitroglycerine   | 40.00 | 15.8 | 91.0 | 26.0 | 9.5  |
| Nitrocotton      | 0.1   | 0.1  | 6.0  | 0.4  | 0.1  |
| Ammonium nitrate | 30.0  | 63.1 | --   | 39.0 | 72.2 |
| Sodium nitrate   | 18.9  | 11.9 | --   | 27.5 | --   |

|                       |       |       |       |       |       |
|-----------------------|-------|-------|-------|-------|-------|
| Wood pulp or nut meal | 8.0   | 3.4   | 0.5   | 2.0   | 2.4   |
| Balsa                 | 2.0   | --    | --    | --    | --    |
| Starch or wheat flour | --    | 3.9   | 1.5   | 3.8   | 4.0   |
| Guar gum              | --    | 1.3   | --    | --    | 1.3   |
| Microballoons         | --    | --    | --    | 0.3   | --    |
| Sodium chloride       | --    | --    | --    | --    | 10.0  |
| Chalk                 | 1.0   | 0.5   | 1.0   | 1.0   | 0.5   |
| Total                 | 100.0 | 100.0 | 100.0 | 100.0 | 100.0 |

\* 1. NG dynamite (ditching dynamite), 2. 60% Extra dynamite,  
3. Blasting gelatin, 4. 60% Extra gelatin, 5. Permissible dynamite

#### 1.10.2.9 Liquid oxygen explosives (LOX):

Liquid oxygen explosives (LOX) is a simple formulated explosive. A cloth cartridge containing carbon black is soaked in a vat of liquid oxygen until the material in the cartridge is totally saturated. The cartridge is immediately lowered into the bore hole stemmed and shot. The drawback of LOX is that very limited number of holes can be loaded since the evaporation of the oxygen is rapid. (ref 2, pages 7-8).

#### 1.10.2.10 Ammonium nitrate fuel oil (ANFO)

ANFO is a high explosive component of many explosives which are used in mining. It is a mixture of ammonium nitrate and diesel fuel oil. These mixtures are made of porous pills of ammonium nitrate (94%) soaked in fuel oil. In some cases aluminium powder is added to increase the explosive strength. The components of ANFO are readily available and not regulated due to commercial use. ANFO is safe to handle but low in explosives strength and detonation velocity. It cannot be used in presence of water. (ref 3, page 20-21)



#### 1.10.2.11 Slurry explosives or water gels:

Slurry explosives or water gels are aqueous solutions of oxidizer and/or fuels. These explosives are made of aqueous solutions of ammonium nitrate and sodium or calcium nitrate, gelled by the addition of guar gum or cross linking agents. They are sensitized by nitro explosives or organic amine nitrates. Combustible materials such as Al, urea, sugar or glycol are mixed with these solutions.

Typical slurry explosives contain ammonium nitrate (30 – 70% O, Sodium nitrate ( 10-15%), calcium nitrate(15-20%), aliphatic amine nitrate upto 40%, Al ( 10-1L%), TNT or other explosives sensitizers (5-15%), gellants (1-2%), stabilizers (0.1- 2%), ethylene glycol (3-15%) and water (10-20%). (ref 2, page 9 & ref 3, page 21).

#### 1.10.2.12 Explosive emulsions:

Emulsions are sensitized by air bubbles introduced by means of hollow glass or plastic bubble. Because the emulsions are neither gelled nor cross linked, their storage time is limited. Their relative high detonation velocity compensates for low explosive strength of the constituents. The common ingredients of explosive emulsions are ammonium nitrate, sodium nitrate, fuel oil, wax and emulsifiers sensitized with plastic micro spheres and other micro-ingredient, (monomethyl amine nitrate, sodium perchlorate) (ref 2, page 9 & ref 3, pages 21-22).

#### 1.10.2.13 Mixture

A vast number of explosives consisting of mixtures of various explosives compounds were developed by combatants in world war II. Many of these combinations may include materials such as HMX, RDX, TNT, Al powder, wax, and plasticizers with or without other ingredients for special properties. A few worth mentioning are

Composition B- (60% RDX, 40% TNT plus wax)

Cyclotol - (60-75 % RDX, 25-40% TNT)

Torpex 2 - (42% RDX, 40% TNT, 18% Al)

Composition C4 - (91% RDX, 9% plasticisers)

Picratol - (TNT + ammonium picrate)

Tritonal - (TNT + aluminium powder)

Amatol - (TNT +AN)

MOX – (RDX + TNT + Al+Ba(NO<sub>3</sub>)<sub>2</sub> + Calcium stearate+ graphite+ wax)

Pentolite – ( PETN +TNT)

Semtex H – (RDX+PETN+Poly butadiene styrene + oil)

PTX 1- (RDX + TNT + Tetryl)

PTX 2 – (RDX+TNT+PETN) (Ref. 3 page 19)

C1 - RDX + Plasticizer

C2 - RDX+TNT +DNT + MNT +NC+DimethylFormamide

C3 - RDX+TNT+DNT+MNT + Tetryl+NC

### 1.10.3 Low explosives

Low explosives are compounds or mixtures that deflagrate. These explosives have propagation speed less than 1000m/s. Low explosives are mechanical mixtures of individual ingredients. They are used mainly used as propellants because they tend to exert a rapid pushing effect. Propellants are mixtures of one or more energetic materials, plasticizers, stabilizer and inorganic additives (ref 3, page 17).The main applications of propellants are in launching projectiles from guns, rockets and missile systems. **Black powder** (gun powder) and smokeless powder are the most commonly used low explosives.

#### 1.10.3.1 Black powder:-

Black powder is one of the oldest explosive. It is very mainly used today as an igniter for NC gun propellants and to some extent in safety blasting fuses, delay fuse, signal and distress rockets and in fire crackers. It is also used for mining when a lower power explosive is used.

The various compositions of black powder are:

Potassium nitrate + charcoal + sulphur in the ratio 75:15:10.

Sodium nitrate + charcoal + sulphur

Sulphurless powder : Potassium nitrate + charcoal

Ammonium powder : Potassium nitrate + Ammonium nitrate + charcoal

(ref 3, page 17)

#### 1.10.3.2 Smokeless powder:

Smokeless powder is an extremely flammable low explosive that burns rapidly and vigorously when ignited. It derives its energy from Nitrocellulose (NC) and Nitro glycerin ( NG). The various types of smokeless Powder are:-

- single based propellants.-NC+ Stabilizer e.g. Diphenyl amine (DPA).
- Double based propellants- NC + nitroglycerine and a stabilizer (DPA).
- Triple base propellants – Nitroguanidine+ NC + nitroglycerine and a stabilizer (DPA).

### 1.10.3.3 Pyrotechnic Compositions

These include military pyrotechnic and fire work compositions. Depending upon the use, various inorganic fuels and oxidisers are mixed in definite proportions to get required pyrotechnic effect. These are manufactured under valid licenses or under strict quality control in ordnance factories. The materials used in fire works include

- oxidizing agent such as chlorate of potassium, sodium nitrate etc,
- fuels – viz. aluminium powder ,sulphur ,charcoal ,phosphorous etc,
- colour producing agents –salts of different metals like sodium ,strontium ,and barium etc,
- smoke producing substances like pitch;hexachloroethane, white phosphorus
- binding agents like dextrin ,tapioca powder, pitch etc
- stabilizers such as linseed oil, boric acid etc. (ref 5, page 1)

#### References:

1. Narayanan T.V (1996). *Modern techniques of Bomb Detection and Disposal*. R.A.Security system, Delhi.
2. Alexander Beveridge. (1998).*Forensic Investigation of explosions*, Taylor and Francis Ltd, Bristol.
3. Yinon.J. and Zitrin. S. (1993). *Modern Methods and application of analysis of Explosives*, John Wiley and Sons, New York.
4. Yinon.J. and Zitrin.S. (1981).*The analysis of explosives* . Pergamon press, New York
5. Ghosh.K.M. (1988). *The Principles of fireworks*, Economic enterprise, Ahmedabad.
6. Chladek J. *The identification of organic peroxides. Advances in analysis an detection of explosives*. Proceedings of the 4<sup>th</sup> International symposium on Analysis and detection of Explosives September 7-10: 1992; J Yinon edition. Kluwer academic publishers, 1993;73-76

## Chapter-2

### IMPROVISED EXPLOSIVE DEVICES

**2.1 Title :** - Explosive Devices

**2.2 Scope :** - General Information about explosive devices including Improvised Explosive Devices ( IEDs)

#### 2.2.1 Improvised Explosive Devices(IEDs)

Improvised Explosive Devices are those which are not manufactured by any fixed standards. The materials of construction, composition of ingredients, Initiation method depends upon the availability of resources, knowledge and skill of user. These devices are mainly used by anti-social elements for insurgent activities.

##### **2.2.1.1 Common Explosives compositions of IEDs**

- 1) Potassium chlorate + Arsenic sulphide + projectiles (iron nails, glass pieces , stone chips etc.,) - Throw down device.
- 2) Potassium chlorate + sugar initiated by conc., Sulphuric acid - Acid bomb (ref1).
- 3) Crude RDX or TNT as main charge.

##### **2.2.1.2 Common Improvised Explosive Containers:-**

Tiffin carrier, transistor, cement pipe, iron pipe, telephone, Television, toys, cell-phone, computer, scooter, cycle, the articles which are easily available to the anti-social elements. ( ref1 ,2&3)

**2.2.1.3 Main charge :** - High explosives, Low explosives or combinations.

**2.2.1.4 Initiation device:** Time device, remote device , impact device, battery operated. Acid pen device etc.

**2.2.1.5 Projectile & Missiles:** Iron nails, glass pieces, stone ships, ball bearing, generally used in throw down improvised devices.

##### **References:**

1. Joseph Stofell, "*Explosives and Home Made Bombs*" 2<sup>nd</sup> edition 2<sup>nd</sup> printing 1977 Charles Thomas Publisher, Springfield Illinois USA.
2. Brodie T. "*Bombs and Bombings*" 3<sup>rd</sup> Printing Publisher Charles C Thomas Springfield Illinois USA.
3. Naranayanan T.V., "*Modern techniques of bomb detection and disposal*" First edition (1996). RA Security Systems ,New Delhi

## Chapter – 3

### SYSTEMATIC PROCEDURE FOR THE EXAMINATION OF EXPLOSIVE EVIDENCE

**3.1 Title:** -Systematic Procedure for the examination of explosive evidence.

**3.2 Scope:** Pre blast (Unexploded) and post blast (exploded) low and high explosives

**3.3 Purpose:** Detection and identification of explosives.

**3.4 Introduction:-**

The analysis of Explosive evidence is in one of two forms

- i) **Analysis of pre blast or unexploded material – bulk analysis**
- ii) **Analysis of post blast or exploded material – Trace analysis**

**3.5 Systematic Procedure:-**

The systematic procedure for both pre blast and post blast explosive evidence is divided in to Four major steps

1. Preliminary examination
2. Extraction of Explosive material
3. Detection of Inorganic explosives including pyrotechnic composition
4. Detection of organic explosives

**3.5.1- Preliminary examination**

1. All outside packaging and seal condition is noted.
2. Sample is examined for odour , colour, consistency and any visually observables ingredients. Ingredients with characteristic odour such as mononitrotoulene may often be detected at this stage. The colour and consistency may guide to find the general type of particular explosive . Some ingredients such as fragments / components of the device (pipe/container/wires/wrappers/fuses/ parts of timing devices/batteries etc)can be readily identified by their characteristic appearance. Visible contaminants can also be observed at this stage

### 3.5.2. Extraction of Explosive material:

The solvent extraction of the Post blast debris is a typical for extraction of explosive residues for further testing. Pre blast explosives are directly dissolved in to the solvent on the basis of solubility. Four extracts are required for identification of unknown explosive material/residues.

- a. Organic Solvent extract
- b. Water extract
- c. Alkali extract
- d. Pyridine extract

#### 3.5.2.1. Organic solvent extraction:

Acetone is universally accepted broad-spectrum solvent for majority of organic explosives such as RDX, HMX, TNT, PETN etc. Other organic solvents like ether, benzene and chloroform are also used, which are suitable for certain groups of organic explosives (nitro aromatic and some nitrate ester like EGDN or NG). In some instances, methanol or other solvents or their mixtures may be used instead of acetone. The extracts are concentrated and subjected to various analytical tests for detection and identification of organic explosives (ref 2, page 221-222).

#### 3.5.2.2. Water Extract:

The residue left after acetone extract is treated with hot water and filtered. The inorganic residue dissolves readily in hot water. The water extract is concentrated to a small volume. Chemical and other analytical tests are performed for the detection of water soluble ingredients ( anions and cations ) in explosives compositions such as low explosives, inorganic explosives and pyrotechnic compositions (ref 2, pages 222-223)

#### 3.5.2.3 Alkali Extract:

The residues left after water extraction often include metals, sulphides of arsenic and antimony, sulphur carbon etc. A small portion of the dried material is extracted with 2N NaOH and filtered. The filtrate is preserved for the detection of sulphide, arsenic, antimony, aluminum etc.(ref 5,page 309,229,231,252)

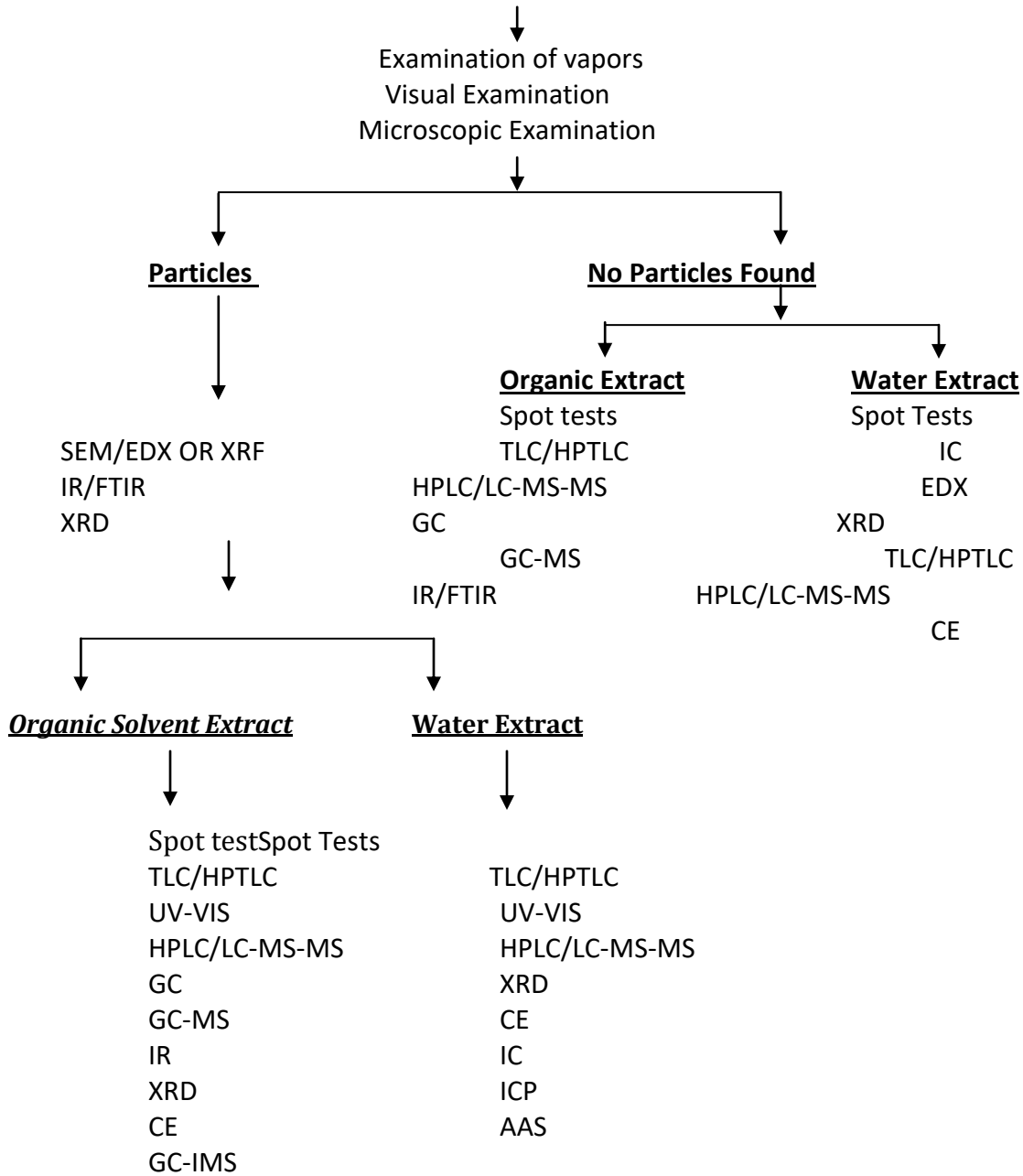
#### 3.5.2.4 Pyridine Extract:

The residue left after the above extraction is dried and extracted with pyridine and filtered. The extract is preserved for the detection of elemental Sulphur. (ref 4, page 373)

### 3.5.3 Scheme of analysis

The following analytical scheme in the flow diagram may be applied in general. The scheme is intended to serve as a general outline of the sequence of the examination. **The scheme is intended to serve as a general outline of the sequence of the examination. Not all of the tests listed in the scheme will be required for every examination and other unlisted tests may be applicable as required by the circumstances of individual cases (ref 1, page122)**

**POST BLAST EXHIBITS**



CE – Capillary Electrophoresis, EDX – Energy Dispersive X-ray, GC–Gas Chromatography, HPLC – High Performance Liquid Chromatography, IC – Ion Chromatography, IR – Infra-red Spectroscopy, GC MS – Gas Chromatography Mass Spectrometry, SEM – Scanning Electro Microscopy, TLC – Thin Layer Chromatography, XRF – X-ray Fluorescence, XRD – X-ray Diffraction, ICP – Inductively coupled plasma spectrometry, GC-IMS – Gas Chromatography – Ion Mobility spectrometry, AAS – Atomic absorption spectrometry, UV-VIS – Ultra Violet –visible spectro photometry, LC-MS-MS-Liquid Chromatography tandem mass spectrometry (ref 1, page 123)

## References

1. Alexander Beveridge.(1998). *Forensic Investigation of explosions*, Taylor and Francis Ltd, Bristol.
2. Yinon.J and Zitrin.S.(1981).*The analysis of explosives*, Pergamon press, New York.
3. Yinon.J and Zitrin.S.(1993). *Modern Methods and Application in Analysis of Explosives*, John Wiley and Sons, New York.
4. Fritz Fiegl, (1965). *Spot Tests in Inorganic Analysis*, Elsevier Publishing Company, Amsterdam
5. A.I Vogel,(1982) *Text Book of Macro and Semi micro Qualitative Inorganic Analysis*,5<sup>th</sup> edition, Longman London



## CHAPTER – 4

### IDENTIFICATION OF INORGANIC EXPLOSIVES /LOW EXPLOSIVES

**4.1 Title:** -Identification of inorganic explosives/Pyrotechnic compositions/Low explosives

**4.2 Scope:** Chemical tests/TLC/IC/EDXRF/ICP/CEanalysis for identification of inorganic explosives/ pyrotechnic compositions

**4.3 Purpose:** Detection and identification of anions and cations in pre blast and post blast inorganic explosives/ pyrotechnic compositions.

#### **4.4 Chemical tests for anions**

##### **4.4.1 Test for Chloride (Cl<sup>-</sup>)**

###### **Requirements:**

- i) 2M Nitric acid
- ii) Silver nitrate
- iii) Lead acetate.

###### **Preparation of reagents:**

Reagent A: - Nitric acid (2M). Pour 128 ml conc.nitric acid into 500ml water, dilute to 1lit.

Reagent B: - Silver nitrate (0.1M). Dissolve 16.99 g of AgNO<sub>3</sub> in water and dilute to 1lit.

Reagent C: - Lead acetate solution (0.25M).Dissolve 95 g lead acetate trihydrate in a mixture of 500 ml water and 10ml glacial acetic acid and dilute the solution with water to 1lit.

###### **Procedure:**

- I. Take 1ml of water extract in a clean test tube. Add 2-3 drops of reagent A to it. Then add 2-3 drops of reagent B. White precipitate indicate the presence of chloride ion, which is soluble in NH<sub>4</sub> OH and insoluble in HNO<sub>3</sub>.
- II. Take 1ml of extract in a clean test tube. Add 2-3 drops of reagent C to it. White precipitate indicates the presence of Cl<sup>-</sup>
- III. The about tests must be carried out along with the reference standard as well as blank. (ref 1, pages 325-326).

## Additional tests

### Requirements:

- i) Potassium dichromate
- ii) Conc. H<sub>2</sub>SO<sub>4</sub>
- iii) Diphenylcarbazide

### Preparation of reagents:

**Reagent A:** - 1% Diphenylcarbazide prepared in ethanol

**Procedure:**- Take 5 ml aqueous extract in a clean test tube, evaporate to dryness, add a small quantity of solid potassium dichromate and 1 ml conc. H<sub>2</sub>SO<sub>4</sub>. Heat the mixture gently. A red vapour of Chromyl chloride evolves which turns diphenylcarbazide spot on filter paper violet. (ref1 page 326).

### 4.4.2 Test for Sulphate: (SO<sub>4</sub><sup>2-</sup>).

#### Requirements:

- i) Dilute HCl
- ii) Barium Chloride
- iii) Sodium rhodizonate

#### Preparation of reagents:

Reagent A: - Dilute HCl.

Reagent B:- 0.25M Barium chloride (Dissolve 61.1 g barium chloride dihydrate in water and dilute to 1 lit.).

Reagent C: - 0.5 % aqueous solution of rhodizonate. Dissolve 0.5 g of sodium rhodizonate in 100ml of water. Reagent should be freshly prepared.

#### Procedure :-

I. Take 1ml of extract in a clean test tube. Add 2-3 drops of reagent A to it and then the test tube is kept in a water bath at 100<sup>0</sup>C for 5 minutes, then add 2-3 drops of reagent B to it. A white crystalline precipitate indicates the presence of SO<sub>4</sub><sup>2-</sup>

II. Place 1 drop of reagent B upon filter paper, followed by a drop of freshly prepared solution of reagent C. Treat the reddish-brown spot with a drop of acidified extract. The coloured spot disappears.

III. The above test must be carried out along with the reference standard as well as blank. (ref 1, pages 347-348 ).

### **Additional test**

#### **Requirements:**

- i) Barium chloride
- ii) Potassium permanganate
- iii) Hydrogen peroxide

#### **Preparation of reagents:**

Reagent A: 0.25M Barium chloride (Dissolve 61.1 g barium chloride dihydrate in water and dilute to 1 lit.)

Reagent B: 0.02 M Potassium permanganate solution in water. (Dissolve 3.16 gm Potassium permanganate in water and dilute to 1 liter)

Reagent c: 3% Hydrogen peroxide solution

#### **Procedure**

Take 3 drops of extract in a semi-micro test tube . Add 2 drops of reagent B and 1 drop of reagent A. a pink ppt appears. Add a few drops of reagent c and shake well. The coloured ppt becomes clearly visible. (Ref1 page 348).

### **4.4.3: Test for Nitrite ( $\text{NO}_2^-$ )**

#### **Requirement:-**

- i) Sulphanilic acid
- ii)  $\alpha$ -Naphthylamine
- iii) Acetic Acid
- iv) Indole
- v) Ethanol
- vi) Sulphuric acid

#### **Preparation of reagents:**

Reagent A:- 1% sulphanilic acid in 30% acetic acid. It is prepared by dissolving 1 g of sulphanilic acid in 100ml of warm 30% acetic acid.

Reagent B:-  $\alpha$ -naphthylamine solution. Take 0.3 g of  $\alpha$ -naphthylamine boil it with 70ml of distilled water, filter or decant and mix with 30ml of glacial acetic acid.

Reagent C:- Indole reagent. Add 0.015 g of indole in 100ml of 96% of alcohol.

Reagent D:- 8 M Sulphuric acid. To 500ml water add slowly under constant stirring, 445 ml conc. Sulphuric acid. Finally when the mixture is cool again dilute with water to 1lit.

**Procedure:-**

I. Place 1 drop of neutral or acid extract on a clean spot plate. Add 1 drop of Reagent A and then 1 drop of reagent B to it. Pink to red colour indicates the presence of  $\text{NO}_2^-$ . (Griess test).

II. Place 1 drops of the extract in a semi micro test tube. Add 10 drops of the reagent C and 2 drops of the reagent D to it. A purplish-red colour indicators the presence of  $\text{NO}_2^-$ .

III. The above tests must be carried out along with the reference standards as well as blank. (ref 1, pages 312-313 ).

**4.4.4 Test for Nitrate ion ( $\text{NO}_3^-$ )**

**Requirements:**

- |                     |                             |
|---------------------|-----------------------------|
| i) Sulphanilic acid | ii) $\alpha$ -naphthylamine |
| iii) Acetic acid    | iv) Zinc dust               |
| v) Sulphamic acid   | vi) Ferrous sulphate        |
| vii) Sulphuric acid |                             |

**Preparation of Reagents:**

Reagent A: 1% sulphanilic acid in 30% acetic acid. Dissolve 1 g of sulphanilic acid in 100ml of warm 30% acetic acid.

Reagent B:  $\alpha$  – naphthylamine Boil 0.3 g of  $\alpha$ -naphthylamine with 70ml of distilled water, filter or decant and mix with 30ml of glacial acetic acid.

**Procedure:-**

1. Place 1drop of neutral or acidic extract in a clean spot plate. Add 1 drop of the reagent A and then 1 drop of reagent B. Then add a pinch of zinc dust. Pink to red colour indicates the presence of Nitrate. (Griess test).
2. Place a crystal of ferrous sulphate on a spot plate. Add a drop of extract and allow a drop of conc. sulphuric acid to run at the side of the drop. A brown ring forms round the crystal of ferrous sulphate indicating presence of nitrate.

**N.B:** *If nitrite is present in the extract, then at first it is removed from the solution by sulphamic acid or by sodium azide and then test for nitrate is performed.*

*The above tests must be carried out along with the reference standards as well as blank. (ref 1, pages 334-337 )*

### **Additional test**

#### **Requirements**

- i) Brucine
- ii) Conc. H<sub>2</sub>SO<sub>4</sub>

#### **Procedure**

Place a few drops of aqueous solution on a clean spot plate. Add one drop of conc. sulphuric acid and small crystal of Brucine and stir the mixture with a glass rod . A blood red colour is produced.

**Note-Chlorate and Nitrites will interfere hence these ions to be eliminated before testing for nitrates if they are suspected in exhibit as per 4.4.12.1 and 4.4.12.2**

### **4.4.5 Test for Thiocyanate (SCN<sup>-</sup>)**

#### **Requirements:**

- i) Cobalt nitrate
- ii) Acetone
- v) Chloroform
- iii) Cupric sulphate
- iv) Pyridine

#### **Preparation of Reagents**

Reagent A: 0.5M Co (NO<sub>3</sub>)<sub>2</sub>. (Dissolve 146 g of cobalt nitrate in water and dilute to 1lit.)

Reagent B: 0.25 M CuSO<sub>4</sub>. (Dissolve 62.42 g of CuSO<sub>4</sub>.5 H<sub>2</sub>O in water and dilute to 1lit.)

#### **Procedure:**

I Mix one drop of the test solution in a micro-crucible with a very small drop of a reagent A and evaporate to dryness. The residue whether thiocyanate is present or not is coloured violet and the colour slowly fades. Add a few drops of acetone, A blue green or green colouration is obtained.

II Add a few drops of pyridine to 3-4 drops of reagent B. Then introduce about 2ml of chloroform, followed by 5-6 drops of the extract. Shake the mixture vigorously. The chloroform layer will acquire a green colour (ref 1, pages 318-319)

III The above tests must be carried out along with the reference standards as well as blank.

#### **Additional test :**

**Requirement:** Ferric Chloride

**Preparation of reagent:** 5% solution of ferric chloride is freshly prepared.

**Procedure:** Place a few drops of water extract on a clean spot plate. Add 2 drops of ferric chloride solution. A red precipitate or colouration is produced. (Ref 1 page 318)

#### 4.4.6 Test for Chlorate : ( $\text{ClO}_3^-$ )

##### Requirements:

- |                                |                      |
|--------------------------------|----------------------|
| i) Concentrated Sulphuric acid | ii) Aniline sulphate |
| iii) Manganoussulphate         | iv) Phosphoric acid  |
| v) Diphenylcarbazine           | vi) Ethanol          |

##### Preparation of reagents:

Reagent A:- 1% Aniline sulphate (Dissolve 1g aniline sulphate in 100ml of water.)

Reagent B: -Manganese(II) sulphate phosphoric acid

It is prepared by mixing equal volumes of saturated manganese sulphate solution and syrupy phosphoric acid.

Reagent C: 1% Alcoholic DiphenylCarbazide solution.

##### Procedure:

I 1ml of extract is dried in a spot plate. After cooling add 2-3 drops of conc. Sulphuric acid. Then add 2-3 drops of reagent A. Blue colour indicates the presence of chlorate ions.

II Place a drop of the extract in a micro crucible and add 1drop of the reagent B. Warm rapidly over a micro burner and allow to cool. A violet coloration appears. Very pale coloration may be intensified by adding a drop of reagent C. When a deep violet colour, due to an oxidation product of the diphenylcarbazine is obtained.

**NB:***The interference due to peroxodisulphates, nitrites, bromates, iodates can be removed by adding sulphuric acid solution with a little silver nitrate as catalyst. (ref 1, pages 337-339 )*

##### Additional test:

##### Requirement:

- i) Indigo
- ii) conc. Sulphuric acid
- iii) sodium sulphite or sulphurous acid

##### Preparation of reagent :

Reagent A : 1% Indigo in concsulphuric acid

Reagent B: 10% sodium sulphite solution in water

Procedure: Add a few drops of reagent A in 1 ml of water extract until the colour changes to pale blue. Add Reagent B or dilute sulphurous acid drop by drop. The blue colour disappears.

#### 4.4.7:-Test for Thiosulphate : ( $S_2O_3^{2-}$ )

##### Requirements:

- |                      |                        |
|----------------------|------------------------|
| i) Mercuric Chloride | ii) Potassium Chloride |
| iii) Nickel nitrate  | iv) Ethylene diamine   |

##### Preparation of Reagents:

Reagent A: 2% Mercuric Chloride solution (Dissolve 2 g of mercuric chloride in 100 ml of distilled water.)

Reagent B: Nickel ethylene diamine nitrate reagent.

It is prepared as and when required by treating a little nickel nitrate solution with ethylenediamine until a violet colour (due to the formation of the complex  $[Ni(en)_3]^{+2}$  ion) appears.

##### Procedure:

- I. Take 1 drop of the water extract in a clean test tube. Add 2 drops of reagent A and mix it. To it add little KCl. The change of blue litmus paper to red indicates the presence of thiosulphate. (ref 2, pages 319-320 )
- II. Take 1ml of the neutral or slightly alkaline water extract in a clean test tube. Then add 2-3 drops of the reagent B to it. A crystalline violet ppt indicates the presence of thiosulphate. (ref 1, pages 305-307 ).
- III. The above tests must be carried out along with reference standard as well as blank.

#### 4.4.8 Test for Perchlorate ( $ClO_4^-$ )

##### Requirements :

- i) Methylene blue indicator
- ii) Zinc sulphate.
- iii) Absolute Alcohol

##### Preparation of Reagents:

Reagent A: 1N Zinc sulphate solution.(Dissolve 28.8 g zinc sulphateheptahydrate in 100 ml of water.)

Reagent B: 1N Potassium nitrate solution. (Dissolve 10.1 g potassium nitrate in 100 ml of water.)

Reagent C: 0.05 % Methylene blue solution. (Dissolve 0.05 g methylene blue in 100 ml of water.)

**Procedure:**

- I. Apply a drop of water extract on a piece of filter paper impregnated with Reagent A - Reagent B and spray the paper with Reagent C. Violet spot indicates the presence of perchlorate.

NB:- Persulphates gives an analogous reaction. When they are present the test is carried out on a TLC plate which is then heated for over 1 hour at 110 °C to decompose the persulphates. Violet spots obtained, following such treatment indicates the presence of perchlorates. (ref 3, page 102-103 )

- II. The above tests must be carried out along with reference standard as well as blank.

**4.4.9 Test for Sulphite ( $\text{SO}_3^{2-}$ )**

**Requirements:**

- i)  $\text{ZnSO}_4$
- ii) Potassium Ferrocyanide
- iii) Sodium Nitroprusside

**Preparation of Reagents:**

Reagent A: Saturated solution of zinc sulphate.

Reagent B: 1N potassium ferrocyanide (Dissolve 10.4 g potassium ferrocyanide dissolved in 100 ml water)

Reagent C: 1% Sodium nitroprusside. (Dissolve 1g sodium nitroprusside in 100 ml water.)

**Procedure:**

- I. A drop of reagent B is added to a drop of reagent A and then a drop of reagent C is added. White zinc ferrocyanide precipitates. A drop of neutral water extract is added. Red precipitate appears.
- II. The above tests must be carried out along with reference standard as well as blank. (ref 3, page 308 )



### **Additional tests**

#### **Requirements :**

i) fuchsin:

#### **Preparation of reagent**

ReagentA:- 0.015 g fuchsin dissolved in 100ml water.

#### **Procedure:**

Place a drop of reagent A on a spot plate. Add one drop of neutral test solution. The reagent is decolourised. (Ref 1 page 304)

### **4.4.10 Test for Phosphate ( $\text{PO}_4^{3-}$ )**

#### **Requirements:**

- i) Ammonium molybdate
- ii) Benzidine
- iii) Conc. Nitric acid
- iv) Glacial Acetic acid
- v) Saturated solution of Sodium acetate
- vi) Conc. Hydrochloric acid.
- vii) Sodium hydroxide

#### **Preparation of Reagents:**

Reagent A: Dissolve 0.5g of Ammonium molybdate in 10ml of water. Add 3ml conc.  $\text{HNO}_3$ .

Reagent B: Dissolve 6.5g Benzidine in 10ml glacial acetic acid and dilute to 35ml with water.

Reagent C: Dissolve 16 g sodium hydroxide in 100 ml water.

#### **Procedure:**

##### **Test for Phosphate:**

- I. Place one drop of water extract in a spot plate. Add one drop of reagent A, wait for 30 seconds. Add one drop of reagent B, wait for 30 sec. Again add 3 drops of saturated solution of sodium acetate. Blue gray colour indicates the presence of phosphate.
- II. The above tests must be carried out along with reference standard as well as blank.

### **4.4.11 Test for Elemental Phosphorus:**

#### **Requirements:**

- i) Ammonium molybdate
- ii) Benzidine

- iii) Conc. Nitric acid
- iv) Glacial Acetic acid
- v) Saturated solution of Sodium acetate
- vi) Conc. Hydrochloric acid.
- viii) Sodium hydroxide

**Preparation of Reagents:**

Reagent A: Dissolve 0.5g of Ammonium molybdate in 10ml of water. Add 3ml conc.  $\text{HNO}_3$ .

Reagent B: Dissolve 6.5g Benzidine in 10ml glacial acetic acid and dilute to 35ml with water.

Reagent C: Dissolve 16 g sodium hydroxide in 100 ml water.

phosphorus is oxidized to phosphate by adding few drops of  $\text{HNO}_3\text{-HCl}$  (mixture 1:1), boil for 5 minutes, cool and wait for 5 minutes. Place one drop of acidic sample solution on spot plate and one drop of reagent C and one drop of reagent A and one drop of reagent B. wait for 30 sec. And then add 5 drops of saturated solution of sodium acetate. A blue or blue gray colour indicates presence of phosphorus. (ref 3, pages 104-105 )

**4.4.12 Special tests for mixtures of anions**

**4.4.12.1 Nitrate in the presence of Nitrite**

The nitrite is readily detected by the change of colour or starch potassium iodide paper to blue. The nitrite cannot be detected in presence of Nitrate. The nitrite is therefore completely decomposed first by adding some sulphamic acid to the solution of the sample. The Nitrate can then be tested for its presence.

**4.4.12.2 Nitrate in presence of Chlorate**

The chlorate obscure the test for nitrate. The nitrate is reduced to ammonia by boiling the sample solution with zinc test and sodium hydroxide. The chlorate is also reduced to chloride which is tested by silver nitrate solution .

**4.4.12.3 Chloride ,chlorate and perchlorate in presence of each other**

Acidify the sample solution with dilute nitric acid and boil. Add silver nitrate solution. A white precipitate of silver chloride indicates the presence of chloride.

Filter off the precipitates of silver chloride. Introduce a little chloride free sodium nitrite (which reduces chlorate to chloride) and more silver nitrate solution into the filtrate. A curdy white precipitate of silver chloride indicates the presence of chlorate perchlorate. Pass excess sulphur dioxide into the solution to reduce chlorate to chloride, boil off the excess sulphur dioxide and precipitate the chloride with silver nitrate solution. Afterwards, remove the excess silver with a solution of sodium carbonate. Evaporate the resulting solution to dryness and

heat to dull redness better in presence of allied free lime to convert perchlorate to chloride. Extract the residue with water and test for chloride.

#### 4.4.12.4 Sulphide, sulphite, sulphate and thiosulphate

Shake the sample solution with excess of freshly precipitated cadmium carbonate and filter

| <u>Residue</u>   | <u>Filtrate</u>  |                 |                 |   |  |
|--|--|-----------------|-----------------|---|--|
| CdS and excess of CdCO <sub>3</sub> . Wash and Reject the washings. Digest the residue With dilute acetic acid to remove Excess carbonate. Yellow residue Indicates sulphide. Confirm by warming With dilute HCl and test the evolved H <sub>2</sub> S with lead acetate paper (Sulphide | Add strontium nitrate solution in slight excess. Allow to stand overnight and filter.  |                 |                 |   |  |
|  | <table border="1"> <thead> <tr> <th><u>Residue</u></th> <th><u>Filtrate</u></th> </tr> </thead> <tbody> <tr> <td>treat with dil. HCl and filter. Test the residue for sulphate (sodium nitropruside test) and filtrate for Thiosulphate and sulphite</td> <td>Acidify with dil. HCl and boil SO<sub>2</sub> is evolved and sulphur is slowly precipitated</td> </tr> </tbody> </table> | <u>Residue</u>  | <u>Filtrate</u> | treat with dil. HCl and filter. Test the residue for sulphate (sodium nitropruside test) and filtrate for Thiosulphate and sulphite | Acidify with dil. HCl and boil SO <sub>2</sub> is evolved and sulphur is slowly precipitated |
|  | <u>Residue</u>   | <u>Filtrate</u> |                 |   |  |
| treat with dil. HCl and filter. Test the residue for sulphate (sodium nitropruside test) and filtrate for Thiosulphate and sulphite  | Acidify with dil. HCl and boil SO <sub>2</sub> is evolved and sulphur is slowly precipitated   |                 |                 |   |  |
|  |  |                 |                 |   |  |

### 4.5 Chemical tests for Cations

#### 4.5.1 Test for Sodium ion (Na<sup>+</sup>)

##### Requirements:

- i) Zinc acetate
- ii) Uranyl acetate
- iii) Glacial Acetic acid

##### Preparation of reagents:

Reagent A: Uranyl zinc acetate reagent:

Dissolve 10 gms of uranyl acetate dihydrate in a mixture of 5ml glacial acetic acid and 20ml of water and dilute to 50ml (Solution a). In a separate vessel stir 30 gms of zinc acetate dihydrate with 5ml glacial acetic acid and 20ml water and dilute to 50ml (solution b). Mix the two solution a & b and add 0.5g of sodium chloride. Allow to stand for 24 hours, and filter from the precipitated sodium zinc uranyl acetate Alternatively, The reagent is prepared by dissolving 10 g of uranyl zinc acetate in 50ml water or in 1M acetic acid.

##### Procedure :

- I. Place a drop of neutral water extract on a black porcelain plate and add 8 drops of reagent A. Stir with a glass rod when a yellow turbidity or ppt appears. In case of smaller amounts of sodium, place the porcelain plate under UV light. A bright fluorescence indicate the presence of sodium.

- II. The tests must be carried out along with reference standard as well as blank ( ref 2, page 229 )

#### 4.5.2 Test for Potassium ion ( $K^+$ ):

##### Requirements:

- i) Sodium cobalti nitrite
- ii) Dipicrylamine reagent
- iii) 0.1N Nitric acid
- iv) Sodium carbonate

##### Preparation of reagents:

Reagent A: Dipicrylamine (or hexanitro-diphenylamine) reagent. Dissolve 0.2 g of dipicrylamine in 2ml 2N sodium carbonate and 15 ml water. Soak strips of filter paper in the reagent and dry it. This paper should be freshly prepared.

Reagent B: 0.167 M sodium cobaltinitrite solution (Dissolve 6.73g sodium cobaltinitrite in 100 ml water.)

##### Procedure:

I Place a drop of the water extract upon reagent A paper and add 2 drops of 0.1N  $HNO_3$ . A red stain at the site of the spot appears while all other parts of the reagent paper turn bright yellow.

*NB: First test the suspected material for the presence of ammonium ion. If the extract contain ammonium, evaporate it to dryness in a micro crucible. After cooling the residue moisten the residue with water and carry out the test as above. Ammonium ion may be eliminated by boiling the solution with dilute NaOH solution. (ref 3, pages 105-106 )*

II. Take 5 drops of water extract in watch glass, evaporate to about 2 drops and cool, then add 4 drops of reagent B and warm if necessary. Yellow precipitate insoluble in dilute acetic acid, .indicates  $K^+$  (ref 1, page 289).

- a) Test for Potassium ion in presence of ammonium ion, add water extract to a few drops of perchloric acid white ppt. appears (Ref 1)
- b) Add water extract to 1 ml of 10% (W/V) Sodium hydroxide solution and boil. The evolution of pungent smelling gas occurs, which produces deme white fume on contact with

a glass rod moistened with conc. Hydrochloric acid, indicating the presence of ammonium ions.

III. The above tests must be carried out along with the reference standard as well as blank.

#### 4.5.3 Test for Barium ion ( $Ba^{2+}$ )

##### Requirements:

- i) Sodium rhodizonate.
- ii) Ethylene diaminedihydrochloride
- iii) Hydrochloric acid

##### Preparation of reagent:

Reagent A: 0.2 % aqueous sodium rhodizonate.

Dissolve 0.2 g of sodium rhodizonate in 100ml of distilled water. The reagent does not keep well so only small quantities must be prepared at a time for long.

Reagent B: 2% ethylene diaminedihydrochloride solution.

Reagent C: 0.5M HCl. Dilute 4.5ml conc.HCl with water to 100ml.

##### Procedure:

I. Place one drop of neutral or slightly acidic water extract on a filter paper and then add a drop of reagent A. A red-brown spot indicates the presence of  $Ba^{+2}$ . On adding reagent C a brilliant red acid salt is formed. The red colour disappears if barium is absent. (ref 1, page 280)

II. Prepare a relatively stable rhodizonate reagent paper. Impregnate with reagent B, sprayed with reagent A and dry at a temp less than  $80^{\circ}C$ . The resulting test paper is stable for at least six months when kept in a dark container (ref 3,page 82). Barium in presence of Lead a relatively stable rhodizonate reagent paper has been developed for barium and lead screening. The filter paper is impregnated with reagent B sprayed with reagent A and dried at a temperature less than  $80^{\circ}C$ . the resulting test paper is stable for at least six months when kept in a dark container. (ref 3, page 82 )

III. The above test must be carried out along with reference standard as well as blank.

#### 4.5.4 Test for Calcium ion ( $Ca^{2+}$ )

##### Requirements:

- i) Sodium Rhodizonate
- ii) 0.5 N Sodium Hydroxide.

##### Preparation of reagents:

Reagent A: 0.2% aqueous solution of Sodium rhodizonate. Dissolve 0.2 g of Sodium rhodizonate in 100ml of distilled water.

**Procedure:**

Place one drop of neutral or weakly acidic water extract on a spot plate. Add one drop freshly prepared reagent A and then add one drop of reagent B. Mix the solution by blowing air briskly through a pipette. A violet precipitate indicates the presence of calcium. (ref 2, pages 222-223)  
The above tests must be carried out along with the reference standard as well as blank.

**4.5.5 Test for Strontium ion ( $\text{Sr}^{2+}$ )****Requirements:**

- i) Potassium chromate
- ii) Strontium rhodizonate

**Preparation of reagents:**

Reagent A: 0.2% aqueous solution of sodium rhodizonate.

Dissolve 0.2 g of sodium rhodizonate in 100ml of distilled water.

Reagent B: Saturated solution of potassium chromate.

**Procedure:**

- I. Place a drop of neutral extract of sample on a filter paper or in a spot plate. Add a drop of reagent A. A brownish red coloration or precipitate is produced.  
If barium is also present proceed as follow. Impregnate the filter paper with reagent B and dry it. Place a drop of extract on this paper and after a minute place a drop of reagent A on the moistened spot. Brownish red spot or ring is formed. (ref 1, pages 281-282 )
- II. The above test must be carried out along with the reference standard as well as blank.

**4.5.6 Flame test for Sodium, Potassium, Barium, Calcium & Strontium.**

Take small part of the water extract in a beaker, evaporate it to dryness and cool. Take conc. HCl in a watch glass. Perform flame test by platinum wire in the non-luminous flame of the burner. (ref 1, page 466 )

Table-15. Colour of flame

| IONS             | COLOUR OF FLAME                                   |
|------------------|---|
| Na <sup>+</sup>  | Golden Yellow                                     |
| K <sup>+</sup>   | Violet (lilac), Crimson through cobalt blue glass |
| Ca <sup>2+</sup> | Brick-red   |
| Ba <sup>2+</sup> | Yellowish-green                                   |
| Sr <sup>2+</sup> | Crimson   |

### 3.5.7 Test for ammonium ion (NH<sub>4</sub><sup>+</sup>)

#### Requirements:

- |                          |                       |
|--------------------------|-----------------------|
| i) Potassium Iodide      | ii) Mercuric Chloride |
| iii) Potassium Hydroxide | iv) Hydrochloric Acid |
| v) Sodium Nitrite        | vi) Calcium Oxide     |

#### Preparation of reagents:

Reagent A: Nessler's Reagent

Dissolve 10 g of potassium iodide in 10ml of ammonia free water (Solution A). Dissolve 6 g of mercuric chloride in 100ml water (Solution B). Dissolve 45 g of KOH in water and dilute to 80 ml (Solution C). Add solution B to solution A drop wise until a slight permanent precipitate is formed. Then add solution C, mix and dilute with water to 200ml. Allow to stand overnight and decant clear liquid.

Reagent B: Para-nitrobenzene-diazonium chloride reagent.( Riegler's solution)

Dissolve 1 g of p-nitro aniline in 25ml of 2M. HCl and dilute with water to 160 ml. Cool and add 20 ml of 5% sodium nitrite solution with vigorous shaking. Continue the shaking until the precipitate dissolves. The reagent becomes turbid on keeping, but can be employed again after filtering.

#### Procedure:

- I. Place a drop of neutral or weakly acidic water extract in a spot plate and add a drop of reagent. An Orange brown precipitate indicates the presence of ammonium ion.

- II. Place a drop of the neutral or slightly acidic extract on a spot plate. Cover the drop with a fine granules of Calcium oxide before adding a drop of reagent B. Lay on adding a drop of reagent B a red zone forms around calcium oxide, carry out blank test side by side.(ref 1, page 295 )
- III. The above tests must be carried out along with the reference standard as well as blank.

#### 4.5.8 Test for Magnesium ion ( $Mg^{2+}$ )

##### Requirement:

- i) Quinalizarin
- ii) Ethanol
- iii) p-Nitrobenzene-azo-resorcinol ( or Magneson I ) Reagent
- iv) Sodium hydroxide

##### Preparation of reagent

Reagent A: Dissolve 0.02 g of Quinalizarin in 100ml of ethanol.

Reagent B: Dissolve 0.5 g of Magneson I in a mixture of 10 ml 2M sodium hydroxide and 10ml water. Dilute the solution with water to 100 ml

##### Procedure:

##### Post explosion Test Procedure:

- I. Place a drop of water extract in the spot plate and add 2 drops of reagent A. (In an acidic medium solution is orange colour). Add 2N NaOH until colour changes to violet. Appearance of blue precipitate or tint that intensifies on standing indicates the presence of Magnesium. (ref 3, pages 97-98 )
- II. Place a drop water extract on a spot plate and add 1-2 drops of reagent B. Add one drop of 2M NaOH. Blue precipitate is formed indicating the presence of magnesium. (ref 1, page 288 )
- III. The above tests must be carried out along with the reference standard as well as blank.

##### Pre explosion Test Procedure:

Dissolve the unknown sample in 2N hydrochloric acid prior to performing the procedure above (ref 3, page 98)

#### 4.5.9 Test for Aluminium ( $Al^{3+}$ )

##### Requirements:

- i) 1N acetic acid
- ii) Morrin reagent (3,5,7,2',4'-pentahydroxy flavanol )
- iii) Aluminon (Ammonium salt of aurinetricarboxylic acid)
- iv) Alizarin-S



- v) Sodium hydroxide
- vi) Acetic acid
- vii) Quinalizarin

**Preparation of Reagents:**

Reagent A: Prepare saturated solution of Morrin reagent in 5ml of methyl alcohol in a small bottle.

Reagent B: Aluminon reagent.(Dissolve 0.25 g of aluminon in 250ml of water.)

Reagent C: Quinalizarin (1,2,5,8-Tetra hydroxyanthraquinone)

Prepare the reagent paper by soaking filter paper in a solution of quinalizarin obtained by dissolving 0.01 g of quinalizarin in 2ml of pyridine and then diluting with 18 ml of acetone.

**Post explosion Test Procedure:**

- I. Add a small amount of 1N acetic acid to the water extract to make it slightly acidic. Place 3 drops of the acidified solution in a black spot plate and then add 2 drops of reagent A. Observe the spot under UV light. Green fluorescence indicates presence of aluminium. (ref 3, page 90)
- II. Take 1ml of the acidified water extract, add 1 ml 10M ammonium acetate solution and 2 ml 0.1% aqueous solution of reagent B. Shake, allow to stand for 5 minutes and add excess of ammoniacal ammonium carbonate solution to decolourise excess dyestuff and lakes due to traces of chromium hydroxide and silica. A bright red precipitate or colouration persisting in alkaline solution indicates the presence of aluminium. (ref 1, page 252 )
- III. Place a drop of water extract on a reagent paper (reagent C). Hold it for a short time over a bottle containing conc. ammonia solution and then over glacial acetic acid until the blue colour just disappears and the unmoistened paper regains the brown colour of free quinalizarin. A red-violet or red spot is formed. Blank test should also be performed.(ref 1, page 254 )
- IV. The above tests must be carried out along with the reference standard as well as blank.

**Pre explosion Test Procedure:**

- I. Dissolve the unknown metallic powder in 2 N NaOH and carry out the above tests.(ref 3, page 90)

#### 4.5.10 Test for sugars.

##### Preparation of Reagents:

Reagent A: 15% ethanolic solution of 1-naphthol.

Reagent B: 0.5% solution of 2,3,5-triphenyltetrazolium chloride

Reagent C: 0.1 N sodium hydroxide.

##### Post explosion Test Procedure:

- I. Place 2 drops of the test solution on a spot plate add 1 drop of reagent A and 2 drops of conc. sulphuric acid. A blue or purple blue colour indicates the presence of sugar. (ref 3, page 106)
- II. A drop of the test solution is mixed with reagent B and 1 drop of reagent C in a test tube. The mixture is boiled for 1-2 min. A red colour or precipitate indicates reducing sugars. (ref 6, page 338). The above tests must be carried out along with the reference standard as well as blank.

##### Pre explosion Test Procedure:

- I. The procedure is same as above ( I) but a purple colour formation indicates a positive result.

#### 4.5.11. Test for Arsenic ( $As^{3+}$ )

##### Requirements:

- i) Dilute Sulphuric acid
- ii) Arsenic free Zinc granules
- iii) Mercuric Chloride
- iv) Cu foil
- v) Conc. Hydrochloric acid

##### Procedure:

##### I Gutzeit Method:-

Mix a drop of alkali extract with a few grains of Zn and few drops of dilute sulphuric acid in the micro test tube with a flat rim and place a small piece of filter paper moistened with 20% silver nitrate solution on the flat surface. Gray stain will be obtained. (ref 1, page 231)

## II Reinsch's test:

If a bright copper foil is boiled with extract, acidified with at least one tenth of its bulk of conc. HCl, the arsenic is deposited upon the copper as a gray film of copper arsenide.

Antimony, Mercury, Silver and other metals are also precipitated under similar condition. It is therefore necessary to test for arsenic in the deposit in the dry way as follow. Wash the copper strip having deposits with distilled water, dry between filter paper and then heat gently in a test tube. A white sublimate of arsenic oxide appears which is identified by examining under microscope when the sublimate appears as octahedral and tetrahedral crystals (ref 1, Page 231).

### 4.5.12 Test for Antimony ( $Sb^{3+}$ )

#### Requirements:

- i) Rhodamine-B
- ii) Sodium Nitrite
- iii) Conc. HCl

#### Preparation of Reagents:

Reagent A: Add 0.01g Rhodamine-B in 100ml of distilled water.

#### Procedure:

##### Post explosion Test Procedure:

- I. Take a small portion of NaOH extract in a spot plate. To it add 1-2 drops of conc. HCl and a few mg sodium nitrite. Add 10 drops of reagent A and mix it. A change of colour from bright red to violet indicates the presence of Antimony. (ref 3, page 92)
- II. The test must be carried out along with reference standards as well as blank.

##### Pre explosion Test Procedure:

- I. Take a small portion of NaOH extract in a spot plate and add few mg sodium nitrite. Add 10 drops of reagent A and mix it. A change of colour from bright red to violet indicates the presence of Antimony. (ref 3, page 92)

### 4.5.13 Test for Sulphide ions ( $S^{2-}$ )

#### Requirements:

- i) p-Amino dimethylaniline
- ii) Ferric chloride
- ii) Hydrogen sulphide
- iv) Sodium nitroprusside

**Preparation of Reagents:**

Reagent A: 0.1% Methylene blue. Dissolve 0.1 g Methylene blue in 100 ml water.

Reagent B: 1% Sodium nitroprusside. Dissolve 0.1 g Sodium nitroprussidedihydrate in 10 ml water. (use freshly prepared solution)

Reagent C: 0.5M Ferric chloride. Dissolve 135.2 g Ferric chloride hexa hydrate in water. Add a few ml conc. HCl, if necessary and dilute with water to 1lit. If the solution turns dark add more HCl.

**Procedure:**

- I. Place a drop of alkali extract on a spot plate, add a drop of conc. HCl, mix. Add 2-3 drops of reagent A to it. Add a drop of reagent C. A clear blue coloration appears after a short time (2-3 minutes). (ref 1, page 310)
- II. Mix on a spot plate a drop of the alkaline extract with a drop of reagent B. A violet colour indicates the presence of sulphide. (Ref 1, page 309)  
Alternatively filter paper impregnated with 2M ammoniacal solution of sodium nitroprusside may be employed.  
The tests must be carried out along with reference standards as well as blank.

**4.5.14 Test for Sulphur****Requirements:**

- i) 2M Sodium hydroxide

**Procedure:**

- I. Take a small portion of pyridine extract in a small test tube and boil it for one minute. Add a drop of 2M sodium hydroxide (add in a hot condition). A blue to green colour for low and red to brown for high concentrations of sulphur is observed. (ref 3, page 107)
- II. The above tests must be carried out along with the reference standard as well as blank.

**4.5.15 Ion Chromatography**

The instrument Ion Chromatograph is calibrated and kept ready for analysis as per the instrument manual.

**Analysis of Anions**

Column : Polystyrene/divinylbenzene copolymer, 250mm length, 4.6mm ID  
Suppressor : Metrohm Suppressor Module (MSM, 20m mol/L H<sub>2</sub>SO<sub>4</sub>)

Eluent : 1.7 Mmol/L NaHCO<sub>3</sub>/1.8Mmol/L Na<sub>2</sub>CO<sub>3</sub>

Flow rate : 1ml/min

Sample volume: 10 µL

pH range : 1 - 13

**NB:** Any other validated methods may be used for analysis.

### **Analysis of Cations**

Column : Spherical Silica gel with polybutadiene maleic acid groups, 125mm length, 4mm ID

Eluent : 4 Mmol/L Tartaric acid/0.75 Mmol/L Dipicolinic acid : 3 Mmol/L Tartaric acid/0.5Mmol/L Oxalic acid for basic cations and nickel, zinc, cobalt : 8 Mmol/L Tartaric acid/10% acetone for basic cations.

Flow rate : 1ml/min

Sample volume: 10 µL

pH range : 2 - 7

**NB:** Any other validated methods may be used for analysis.

### **4.5.16 Capillary Electrophoresis.**

The Capillary Electrophoresis is a complimentary technique to Ion chromatography for the analysis of inorganic ions.

The instrument is calibrated and kept ready for analysis as per the instrument manual.

#### **Conditions:**

Column : Fused silica capillary with 75µm I D (Prepared for use by flushing for 2 minutes with 100 mMNaOH.)

Detector: Scanning UV detector, dual wavelength mode at 280 nm and 205 nm.

Operating Voltage: 20 KV

Buffer: Add 0.53 g of potassium dichromate, 0.76 g of sodium tetraborate, and 2.47 g of boric acid to 1 liter of de-ionised water, adjust to 7.65 with diethylenetriamine (DETA) and filter through 0.45 µ m nylon filter. The resultant buffer solution is 2mM in borate , 40 mM in boric acid and 1.8 mM in dichromate.

Injection: Hydro dynamic injection

Injection time: 5 sec

Ions identified: Cl<sup>-</sup>, NO<sub>2</sub><sup>-</sup>, ClO<sub>3</sub><sup>-</sup>, NO<sub>3</sub><sup>-</sup>, SO<sub>4</sub><sup>-</sup>, SCN<sup>-</sup>, ClO<sub>4</sub><sup>-</sup>, HCO<sub>3</sub><sup>-</sup>

(ref 4, page 135 )

#### 4.5.17 High Performance Liquid Chromatography for Sugars

##### Conditions:

Column: LC-NH<sub>2</sub>, 250mm x 4.6mm(I<sub>d</sub>)

Mobile phase: Acetonitrile: water (80:20)

Flow rate: 2 ml/ min

For fructose, glucose and sucrose.

Detector: Refractive Index (RI)

#### 4.5.18 EDXRF data of explosive residues.

XRF Technique based on the emission of characteristic X-ray radiation when a sample is exposed to exciting radiation from more energetic X-ray. The spectra are displayed as intensity versus energy or wavelength.

- The sample is irradiated with a beam of primary X-ray knocks out an electron from the K shell. An electron in the L shell drops into the K shell with concurrent emission of radiation of characteristic wavelength, the secondary or fluorescent radiation. An electron in the M shell drops into the vacancy in the L shell with the emission of radiation. The process is repeated as an electron in the N shell drops into the newly created vacancy in the M shell. Hence obtain spectrum consist of well defined peaks due to these transitions, which are called  $K\alpha$ ,  $K\beta$ ,  $K\gamma$ ,  $L\beta$ , and  $L\gamma$  for the transition  $L\rightarrow$ ,  $M\rightarrow K$ ,  $N\rightarrow K$ ,  $M\rightarrow L$  and  $N\rightarrow L$ . These peaks may have shoulders, because the electron in the L or M shell can have one of three or five energies depending whether it is in a, s, p or d sub shell but quantum mechanical selection rule allows two  $K\alpha$  and two  $K\beta$  transitions.
- The energy differences within a shell are very small compared with the differences between various shells and these transitions show up. If at all, as extra peaks on the main peaks, they are labeled  $K\alpha_1$ ,  $K\alpha_2$  and  $K\beta_1$  etc. The X-ray spectrum is thus highly characteristic of an element.
- According to Moseley for a series of the same type the observed wave length is related to the atomic number of the element. This leads to two conclusion of analytical importance: (1) A spectrum of unknown sample will have a series of peaks whose maximum wavelength is directly related to atomic number of the element in the sample, i.e. quantitative analysis is simple. (2) The peaks are so characteristic of an element that quantitative analyses can be carried out without prior separation, i.e. method is specific.
- Furthermore, because the transitions are between inner shells, the valence state of the element is of little consequence so that the total amount of an element can be readily determined regardless of its chemical form.

### Detection limits

- Detection limits depends upon the fluorescence efficiency of the desired element, the apparatus and the matrix in which the particular element is to be determined. Detection limits are there for different elements and matrices and range from 0.1% for very high elements in heavy matrices to 0.00001% or 0.1ppm heavy element in light matrices.

- Table-16 ENERGY TABLE OF INORGANIC ELEMENT OF EXPLOSIVES

| ELEMENT | K $\alpha$ | KB1    | K $\beta$ | L $\alpha$ 1 | LB1    | LB2    | Ly1    | Llab   | Lllab  | Llllb  | M $\alpha$ 1 |
|---------|------------|--------|-----------|--------------|--------|--------|--------|--------|--------|--------|--------------|
| C       | 0.277      |        | 0.284     |              |        |        |        |        |        |        |              |
| Na      | 1.041      | 1.067  | 1.072     |              |        |        |        |        |        |        |              |
| Mg      | 1.254      | 1.296  | 1.303     |              |        |        |        |        |        |        |              |
| Al      | 1.487      | 1.553  | 1.56      |              |        |        |        |        |        |        |              |
| S       | 2.308      | 2.464  | 2.471     |              |        |        |        |        |        |        |              |
| Cl      | 2.622      | 2.816  | 2.82      |              |        |        |        |        |        |        |              |
| k       | 3.313      | 3.59   | 3.608     |              |        |        |        |        |        |        |              |
| Ca      | 3.691      | 4.013  | 4.038     | 0.341        | 0.345  |        |        |        |        |        |              |
| Fe      | 6.4        | 7.058  | 7.111     | 0.705        | 0.718  |        |        |        |        |        |              |
| As      | 10.532     | 11.727 | 11.865    | 1.282        | 1.317  |        |        |        |        |        |              |
| Sr      | 14.142     | 15.837 | 16.108    | 1.807        | 1.872  |        |        |        |        |        |              |
| Sb      | 26.279     | 29.725 | 30.486    | 3.605        | 3.844  | 4.101  | 4.348  | 4.699  | 4.382  | 4.132  |              |
| Ba      | 32.071     | 36.381 | 37.458    | 4.466        | 4.828  | 5.157  | 5.531  | 5.996  | 5.623  | 5.247  |              |
| Hg      | 70.167     | 80.249 | 83.1      | 9.989        | 11.823 | 11.924 | 13.83  | 14.843 | 14.251 | 12.287 | 2.195        |
| Pb      | 74.243     | 84.921 | 87.995    | 10.552       | 12.614 | 12.623 | 12.623 | 15.855 | 15.205 | 13.041 | 2.346        |

**4.6 Table-17 Summary of Techniques used for Explosive Trace Analysis  
(ref 7 page 20)**

| Detection Technique  | Target Analytes   | Specificity   | Typical LOD    |
|--|---|---|----------------|
| Visual microscopy  | All   | Low   | µg             |
| UV/Vis absorption  | All   | Medium  | ng- µg         |
| Fluorescence   | Fluorescent organics and Inorganics   | Low-Medium  | ng- µg         |
| Spot test/Chemical colour test   | Several relevant targets e.g. nitrates, chlorates, peroxides, sulphur, sugars | Low-Medium  | µg/ml          |
| Immunoassay  | Organics  | Medium  | ng             |
| Ion Mobility Spectrometry (IMS)  | Organics  | Medium-High   | pg-ng          |
| Mass spectrometry (MS)   | All (depending on ionization used)  | Medium-unit mass resolution<br>High-High resolution | pg-ng          |
| Scanning Electron Microscopy/Energy Dispersive Spectroscopy (SEM/EDS)                      | Elements Z >10  | High  | pg             |
| X-ray diffraction (XRD)  | Crystalline organics and inorganics   | High (pure compounds)<br>Medium (Mixtures)          | mg             |
| Fourier Transform- Infrared (FTIR)   | Infrared active organics and inorganics                                       | High (pure compounds)<br>Medium(Mixtures)           | mg<br>µg (ATR) |
| Raman  | Raman active organics and inorganics  | High(pure compounds)<br>Medium(mixtures)            | µg<br>ng       |
| X Ray Fluorescence (XRF)   | Elements Z>10   | High  | µg             |
| Inductively Coupled Plasma mass spectrometry or optical emission spectrometry (ICP-MS/OES) | Elements Z>7  | High  | ng             |
| Thermal Energy Analyser (TEA)  | Nitro- containing compounds   | High  | pg             |
| Electron Capture Detector (ECD)  | Organics  | Low   | pg             |
| Conductivity   | Inorganics, Sugars  | Low   | ng             |



**References:**

1. A.I Vogel, (1982). *Text Book of Macro and Semi Micro Qualitative Inorganic Analysis*, 5<sup>th</sup> edition, Longman London.
2. Fritz Fiegl. (1965). *Spot Tests in Inorganic Analysis*, Elsevier Publishing Company, Amsterdam.
3. Ervin Jungreis. (1997). *Spot Test Analysis*, 2<sup>nd</sup> edition, A Wiley-Interscience publication, N.York
4. Yinon. J. *Advances in Analysis and Detection of Explosives*,. Proceedings of the 4<sup>th</sup> international Symposium, Ion analysis and Detection of Explosives, Sep 7- 10, 1992, Jerusalem, Israel. Kluwer Academic Publishers, London.
5. K.E.Hall and B.R.Mc Cord (1993). " *The analysis of mono and divalent cations present in Explosives residues using ion chromatography with conductivity detection*",J.Foren.Sci. Vol 38, No 4, pages 928-934.
6. Fritz Fiegl . (1966). *Spot Tests in Organic Analysis*, Elsevier Publishing Company, Amsterdam.
7. *Best Practice Manual for the Forensic Recovery, Identification and Analysis of Explosive Traces* (ENFSI-BPM-EXP-01) Version 01 November 2015 by the European Network of Forensic Science Institutes (ENFSI)

## CHAPTER – 5

### **IDENTIFICATION OF PRIMARY HIGH EXPLOSIVES (INITIATORY COMPOSITION)**

**5.1 Title:** -Identification of Primary High Explosives (Initiatory Composition)

**5.2 Scope:** Chemical tests and chromatographic methods for identification of Primary High Explosives (Initiatory Composition)

**5.3 Purpose:** Detection and identification of Primary High Explosives (Initiatory Composition) in pre blast and post blast residues.

#### **5.4 Identification of Primary High explosives (Initiatory Composition)**

##### **5.4.1 SPOT TEST**

###### **5.4.1.1 Mercury Fulminate**

Add an equal amount of Vanadium pentoxide to the sample on a spot plate and allow one drop of concentrated hydrochloric acid to run into the mixture. Mercury fulminate produces a violet coloration.

###### **5.4.1.2 Lead Azide**

Add one drop of freshly prepared ferric chloride solution to the sample dissolved in saturated ammonium acetate solution. A blood red colouration indicates Azide or Thiocyanate. In case of azide the colour is discharged by hydrochloric acid but retained in case of thiocyanate.

The presence of Lead styphnate is indicated by its characteristic yellow green colour.

##### **5.4.2 Thin layer chromatography**

###### **Eluents:**

1. Methanol/2N hydrochloric acid 80/20
2. Methanol/2N hydrochloric acid/acetone 65/15/10
3. Acetone/concentrated ammonium hydroxide 75/25

**TLC Conditions:**

Plate coating: MN Silica gel N 0.25mm (thickness)

Solvent: 20% aqueous ammonium acetate:ethanolamine

Sample aliquot : 2ul.

**Spray reagent for visualization**

1. 2% aqueous Potassium iodide
2. 2% aqueous ferric chloride
3. 2% aqueous acidified sodium sulphide.( ref : 11 page 248 )

Table-18. Rf Values with various solvents and colour of spot

| Substance | Rf with various eluents |      |      | colour with various visualisers |     |               |
|-----------|-------------------------|------|------|---------------------------------|-----|---------------|
|           | 1                       | 2    | 3    | 1                               | 2   | 3             |
| MercurylI | -                       | -    | 0.14 | Orange                          | -   | Grey<br>Brown |
| Lead II   | 0.37                    | 0.36 | 0.00 | Yellow                          | -   | Brown<br>Grey |
| Styphnate | 0.73                    | 0.71 | 0.89 | Yellow without visualization    |     |               |
| Fulminate | 0.64                    | 0.63 | 0.95 | -                               | -   | Brown<br>Grey |
| Azide     | -                       | 0.49 | 0.62 | -                               | Red | -             |

**References :-**

1.Yallop.H.J. *Explosion Investigation* 1980. Foren. Sci. Soc. Academic Press, U.K.

## CHAPTER – 6

### IDENTIFICATION OF SECONDARY HIGH EXPLOSIVES (MAIN CHARGE)

**6.1 Title:** -Identification of Secondary High Explosives (Main Charge)

**6.2 Scope:** -Chemical tests and chromatographic methods for Identification of Secondary High Explosives (Main Charge)

**6.3 Purpose:** Detection and identification of Identification of Secondary High Explosives (Main Charge) in pre blast and post blast residues.

#### 6.4 Identification of Secondary High Explosives (Main Charge) by colour tests

##### Requirements:

- |                         |                                     |
|-------------------------|-------------------------------------|
| i) Diphenylamine        | ii) Sulphanilic acid                |
| iii) Acetic acid        | iv) $\alpha$ -Naphthylamine         |
| v) Brucine Sulphate     | vi) Ethanol                         |
| vii) Thymol crystals    | viii) Nitrogen free conc. $H_2SO_4$ |
| ix) Potassium hydroxide | x) Tetramethylammonium hydroxide    |
| xi) J-acid.             | xii) conc. Sulphuric acid           |

##### Preparation of Reagents:

Reagent A: Diphenylamine (DPA). Dissolve 1 g of DPA in 10 ml of conc. Sulphuric acid.

Reagent B: 1% Sulphanilic acid in 33% acetic acid. Dissolve 1 g of sulphanilic acid in 100 ml of 33% acetic acid.

Reagent C:  $\alpha$ -Naphthylamine in acetic acid, Dissolve 1 g of  $\alpha$ -Naphthylamine in 100ml of boiling distilled water, cool and decant the colourless supernatant liquid and mix with 110ml of glacial acetic acid.

Reagent D: Brucine Reagent. Dissolve 5 g brucinesulphate in 100ml of 33% acetic acid.

Reagent E: Alcoholic potassium hydroxide.

Dissolve 10gms of potassium hydroxide in 100ml of absolute alcohol.

Reagent F: 0.1 g J-acid in 100ml of conc. Sulphuric acid.

**Procedure:**

I Take 1ml of the extract in a clean test tube and add 2 drops of Reagent A. (ref 1, pages 41-42 and ref 4) and observe the colour for conclusion.

NG -- blue to blue black      RDX -- blue      PETN -- blue  
NC -- blue black              HMX -- blue      Tetryl -- blue  
NS -- blue black              TNT -- no color

II Add 1 drop of Reagent B followed by 2 drops of Reagent C to 1ml of extract in a spot plate. (ref 2, page 683 and ref 4)

NG -- pink to red              RDX -- pink to red  
NC -- pink                      Tetryl -- pink to red  
NS -- pink                      TNT -- No colour      PETN -- pink to red

III To 1ml of extract in a test tube add 2 drops of Reagent D. (ref 4) and observe the colour for conclusion.

NG -- orange to red              RDX -- orange to red  
NC -- orange to red              Tetryl -- orange to red  
NS -- orange to red              PETN -- orange to red      TNT -- no reaction

IV Add 1 ml of extract to 2-3 drops of Reagent E in a clean test tube (ref 5, page 108).

TNT -- purple red              Tetryl -- red violet

*NB: Add one drop of a 1;1 acetone -alcohol mixture and one drop of 25% aqueous tetramethylammonium hydroxide to 5-10 mg of test material. In the presence of 2,4-dinitrotoluene a blue colour appears, and in the presence of TNT, a dark red colour. Since the colour will change with time, the first observation is the decisive one.*

V Take 200 mg crystals of thymol in a test tube and add 0.3 ml of conc. sulphuric acid (nitrogen free) and 1ml of the sample extract, mix well, warm the tube for 5 minutes at 100 C and add 5-10 ml of ethanol.(ref 5, pages 95-96). Observe the colour for conclusion.

RDX --blue HMX -- pale blue green tint

NB: If the same test is repeated at 150 C RDX will produce blue colour whereas HMX results in olive colour. Thus RDX can be distinguished from HMX.

VI Place a drop of reagent F on a glass filter paper followed by a drop of the test solution in Acetone (ref 5, page 94) and observe the colour for conclusion.

RDX -- Yellow fluorescence under UV light

## 6.5 Identification of Secondary High Explosives (Main Charge) by Thin Layer Chromatography (TLC)

### 6.5.1 For separation of general Secondary High explosives

#### Requirements:

TLC plates (20x20cm or 10x10cm) of Silica gel G layer thickness 250 µm.

TLC developing chamber, Sprayer, Capillary tubes.

Developing solvents:

- i) Chloroform
- ii) Acetone
- iii) Toluene
- iv) Cyclohexane
- v) Ethylacetate
- vi) Trichloroethylene

Control samples: Reference standard of high explosives

#### Procedure:

1. Activate the pre-coated TLC plates by keeping it in air oven at a temperature of 110°C for 30 minutes.
2. Prepare reference standards of high explosives by dissolving small amount of explosives in acetone separately.
3. Take 100ml of eluent in a developing chamber (for 20x20 cm TLC plate), cover with a lid and allow it for saturation for at least 30 minutes.
4. The concentrated acetone extract of sample is spotted on the pre coated TLC plate along with reference standard of high explosives, by leaving 2 cm from one edge of the TLC plate and maintaining minimum distance of 1.5cm between two spots of applications.
5. The TLC plate is placed in the developing chamber vertically and allowed to develop up to 10cm from the spots by ascending method.
6. Then the plate is removed and kept aside for the eluent to evaporate at room temperature.

**7. Solvent system for Developing Chromatogram:**

1. Trichloroethylene: Acetone (4:1) (ref 7)
2. Chloroform: Acetone(1:1) (ref 11, page 241)
3. Toluene: Cyclohexane (7:3) (ref 3, page 35)
4. Toluene : Ethylacetate (9:1) (ref 6, page 323)

**8. Spray (Detection) Reagents**

For visualization of the spots after separation.

- A. Spray the TLC plate with 5% Diphenylamine (DPA) in 95% ethanol and note the colour developed, (II) Then keep the TLC plate under UV light for some time note the colour, (III) The plate is again sprayed with conc. sulphuric acid and the colour developed is noted.
- B. Spray the TLC plate with 1N NaOH in ethanol and keep at 100°C for 10 minutes in an air oven. Cool and spray with Griess reagent (0.25% sulphanilic acid and 0.1%  $\alpha$ -Naphthylamine in 1:1 aqueous acetic acid and observe the colour developed as given in table-1.
- C. KOH and Griess as spray reagent. Brown red for TNT, orange brown for tetryl

Table-19.Rf with various solvents and colour of spot

| Compound | Colour Development |               |                     |        | Rf Values in Different Solvent Systems. |                           |                             |                               |
|----------|--------------------|---------------|---------------------|--------|---|---------------------------|-----------------------------|-------------------------------|
|          | DPA                | UV light      | Sulphuric Acid      | Griess | Trichloroethylene : Acetone (4:1)       | Chloroform: Acetone (1:1) | Toluene : Cyclohexane (7:3) | Toluene : ethyl acetate (9:1) |
| NG       | No colour          | Gray to green | Blue-gray           | Pink   | 0.47                                    | --                        | 0.42                        | 0.66                          |
| RDX      | No colour          | gray          | Blue-gray           | Red    | 0.15                                    | 0.47                      | 0.03                        | 0.13                          |
| PETN     | No colour          | Gray to green | Blue-green          | Red    | 0.55                                    | 0.69                      | 0.45                        | 0.78                          |
| TNT      | Orange brown       | Orange brown  | Colour fades        | Brown  | 0.59                                    | -                         | 0.57                        | 0.88                          |
| TETRYL   | brown              | brown         | Yellow to Blue-gray | Red    | 0.40                                    | 0.66                      | 0.25                        | 0.61                          |
| HMX      | No colour          | No colour     | No colour           | Red    |   | 0.40                      |                             |                               |

**6.5.2 For differentiation of RDX & HMX:** Solvent system: Chloroform : Acetone (2:1)

Spray reagent: Griess reagent,

Rf Value = 0.72 (RDX), 0.35 (HMX)

**6.5.3 For Separation of NC:** Solvent system: Acetone: Methanol (3:2)

Spray reagent: Griess reagent, Rf value = 0.64

**6.5.4 For separation of mono methyl amine nitrate (MMAN):**

Solvent system: Chloroform: methanol: water (100:90:14)

Spray reagent: DPA followed by UV light and then sulphuric acid

Rf value = 0.60 (ref 8)

**6.6 Identification of Minor components of propellants by Thin Layer Chromatography (TLC).**

**6.6.1 Identification of Stabilisers**

Plate coating: Silica gel G (0.25 mm thick), Sample aliquot: 5µl.

Eluents:

1. Benzene
2. Benzene/Petroleum Ether (40/60)/methanol 27/9/4
3. Benzene/Petroleum Ether (40/60) 1/1
4. Benzene/Ethyl acetate 85/15.

Elution distance: 16 cm

Visualizing reagent :-Potassium dichromate solution



Table-20. Rf values with various eluents

| Substance                   | 1    | 2    | 3    | 4    | Colour    |
|-----------------------------|------|------|------|------|-----------|
| Resorcinol                  | 0.00 | 0.32 | 0.00 | 0.16 | Brown     |
| 2-nitrodiphenylamine        | 0.71 | 0.86 | 0.41 | 0.66 | Red       |
| Methyl centralite           | 0.01 | 0.69 | 0.00 | 0.20 | Red       |
| Ethyl centralite            | 0.02 | 0.75 | 0.01 | 0.33 | Red       |
| p-nitro-N-methylaniline     | 0.25 | 0.58 | 0.05 | 0.37 | No change |
| N-Nitrosodiphenylamine      | 0.58 | 0.82 | 0.24 | 0.69 | Blue      |
| 4-nitrodiphenylamine        | 0.39 | 0.66 | 0.08 | 0.55 | Violet    |
| 2,4-dinitrodiphenylamine    | 0.37 | 0.78 | 0.08 | 0.57 | No change |
| 2,4-dinitro-N-methylaniline | 0.27 | 0.67 | 0.05 | 0.38 | No change |
| Diphenylamine               | 0.74 | 0.78 | 0.48 | 0.69 | Blue      |

### 6.6.2 Identification of Phthalates:

Plate coating: Silica gel G (0.25mm thick).

Sample aliquot: 50µ l.

Eluents:

1. Dichloromethane

2. Benzene: Ethyl acetate :: 85:15.

Visualising reagent:

1% resorcinol in concentrated sulphuric acid. After elution, the plate is air-dried and sprayed. After heating at 140-150°C for 15-20 minutes a yellow-green colour is produced.

Table-21. Rf values with various eluents

| Substance              | 1    | 2    |
|------------------------|------|------|
| Dimethyl Phthalate     | 0.38 | 0.47 |
| Diethyl phthalate      | 0.44 | 0.54 |
| Dibutyl phthalate      | 0.50 | 0.65 |
| Amyl phthalate         | 0.59 | 0.68 |
| Dicyclohexyl phthalate | 0.53 | 0.66 |

## 6.7 High Performance Liquid Chromatography (HPLC)-Acetone extract

1. The instrument HPLC is calibrated and kept ready for analysis as per the operating instructions described in instrument manual.
2. A solution of standard of High Explosives is prepared in Acetonitrile of concentration of about 1mg/ml each and filtered.
3. A solution of Test Sample of High Explosives is prepared in Acetonitrile of concentration of about 2 mg/ml each and filtered. The concentration of test sample may vary depending upon purity.

### Method 1: For TNT, RDX, HMX, PETN, NG, EGDN, Tetryl (ref 9)

Column : C-18, 25cm x 4.6mm ID  
Mobile phase : Acetonitrile: Water (70:30)(HPLC Grade Solvents)  
Flow rate : 1ml / min  
Detection : UV at 214 nm

### Method 2: For NG, EGDN, RDX, PETN, TNT(ref 3, pages 84-85)

Column :  $\mu$ Bondapak CN, 10 $\mu$  m particle size  
Mobile phase : Isooctane: dichloromethane: methanol (165:35:100)(HPLC Grade Solvents)  
Flow rate : 1.5 ml / minute  
Detection : TEA , catalytic pyrolyzer operation temperature at 550 °C, reaction chamber pressure 1.8 torr, ozone flow rate 5 ml/minute, carrier gas nitrogen flow rate 20 ml/minute, cryogenic trap was maintained at - 78 °C with slush bath of ethanol and solid carbon dioxide.

### Method 3: For NG, EGDN(ref 3, page 70)

Column : C-18, 25cm x 4.6mm ID, 10  $\mu$ m Particle size  
Mobile phase : Acetonitrile: Water (50:50) (HPLC Grade Solvents)  
Flow rate : 1ml/ min  
Detection : UV at 200 nm

## 6.8 Gas Chromatography - Mass spectrometry (GC-MS)-Acetone extract

The instrument GC-MS is calibrated and kept ready for analysis as per the operating instructions given in instrument manual.

**Method 1** : For NG, DNT, TNT, PETN, RDX, Tetryl(ref 10)

Column : fused silica capillary, 15m x 0.25 mm ID with 0.25 mm coating of DB-5.

Temperature : Programmed from 70 °C to 270 °C at the rate of 15 °C/ min.

Injector Temp : 180 °C

Ion source temp: 140 °C

Carrier gas : Helium

Scan range : 30-500 or 40-500 mass units

Scan rate : 1 scan/ sec

Electron energy: 70 eV in electron impact (EI) and chemical ionization (CI) modes

**NB:** Any other validated methods may be used for analysis.

## 6.9 Infra-Red Spectrometry

Comparison of an IR spectrum of an unknown compound with a library file of IR spectra (thus taking advantage of the finger print value of an IR spectrum) requires the unknown compound to be relatively pure. This poses a severe limitation on the use of IR for post explosion analysis. The extracted residues seldom contain a pure compound. Thus IR finger print identification is useful only in cases when the extracts are relatively pure or when unexploded particles of the explosives are collected from the debris or when the IR is preceded by chromatographic separation. The latter case is rare because the amount of residual explosives in debris is usually insufficient for the semi-preparative chromatographic separation needed for recording an IR spectrum. (ref 1, page 154). The inference of IR spectra can be drawn as :-

General – Absorbing bands in IR range  $4000-1300\text{ cm}^{-1}$  for specific functional groups. Below  $1300\text{ cm}^{-1}$  fingerprint region

$\text{NO}_2$  – Symmetric and asymmetric stretching vibrations of group give rise to two distinct absorption bands – a highly diagnostic value.

Nitro aromatic compounds bands – 1390 – 1320  $\text{cm}^{-1}$  and 1590 – 1510  $\text{cm}^{-1}$

Nitrate esters –  $\text{NO}_2$  stretching vibrations 1285 – 1270  $\text{cm}^{-1}$  and 1660 – 1640  $\text{cm}^{-1}$

Two stretching vibrations in Nitra amines i.e., 1310 – 1270  $\text{cm}^{-1}$  and 1590 – 1530  $\text{cm}^{-1}$ ( Ref 15)

### 6.10:-HPLC-MS/MS (Tandem Mass Spectrometric Method) (ref 16)

1. The instrument HPLC MSMS is calibrated and kept ready for analysis as per the operating instructions described in instrument manual.
2. A solution of standard of High Explosives Acetate adducts are prepared for RDX, HMX, Tetryl, TNT, PETN in Acetonitrile of concentration of about 0.1 $\mu\text{g}/\text{ml}$  each and filtered.
3. A solution of Test Sample of High Explosives acetate adducts is prepared in Acetonitrile of concentration of about 0.1 $\mu\text{g}/\text{ml}$  each and filtered. The concentration of test sample may vary depending upon purity.
4. Ammonium acetate (2mM- 5mM) is used to prepare the adducts of Explosives.
5. The Samples were analyzed by Multiple Reaction Monitoring and Product Ion Scan modes.

**Table 22:** LC-MS/MS operational parameters

| LC conditions    |  | MS conditions                                 |                        |
|------------------|--|---|------------------------|
| Column           | C18 (150mmx2.1mmx5 $\mu$ ) with C18 guard column | Ion spray voltage                             | -4500V                 |
| Mobile Phase     | Acetonitrile: water (60:40: v/v).                | Heater temperature                            | 250 $^{\circ}\text{C}$ |
| Flow Rate        | 200 $\mu\text{l}/\text{min}$                     | Curtain gas                                   | $\text{N}_2$           |
| Injection volume | 20 $\mu\text{l}$                                 | Curtain gas pressure                          | 25 psi                 |
| Run time         | 8 minutes  | Collision activated dissociation (CAD) gas    | $\text{N}_2$           |
|                  |  | Collision activated dissociation gas pressure | 6 psi.                 |

**Table 23:** Mass spectral parameters (LC-MS/MS)

| Mass Parameters                       | Explosive compound        |                              |   |   |  |
|---------------------------------------|---------------------------|------------------------------|---|---|--|
|                                       | TNT<br>[M-H] <sup>-</sup> | Tetryl<br>[M-H] <sup>-</sup> | RDX<br>[M+CH <sub>3</sub> COO] <sup>-</sup> | HMX<br>[M+CH <sub>3</sub> COO] <sup>-</sup> | PETN<br>[M+CH <sub>3</sub> COO] <sup>-</sup> |
| MRM transition                        | 226/46                    | 286/240                      | 281/46                                      | 355/46                                      | 375/62                                       |
| Declustering Potential (V)            | -28.00                    | -44.00                       | -15.00                                      | -17.00                                      | -14.00                                       |
| Entrance Potential (V)                | -5.00                     | -3.00                        | -3.00                                       | -4.00                                       | -3.00  |
| Collision cell entrance potential (V) | -16.00                    | -10.00                       | -15.00                                      | -17.00                                      | -14.00                                       |
| Collision Energy (V)                  | -51.00                    | -12.00                       | -23.00                                      | -34.00                                      | -18.00                                       |
| Collision cell exit potential (V)     | -6.00                     | -2.00                        | -6.00                                       | -7.00                                       | -7.00  |

**References:**

1. Yinon.J. and Zitrin .S. (1981).*The Analysis of explosives*, Pergamon press, New York.
2. Fritz Fiegl . (1966).*Spot Tests in Organic Analysis*, Elsevier Publishing Company, Amsterdam.
3. Yinon.J. and Zitrin .S. (1993).*Modern Methods and Application in Analysis of Explosives*, John Wiley and Sons, New York
4. Parker.R.G. Stephenson .M.O., McOwen. J.M. and Cherolis J.A. (1975). " *Analysis of Explosives and Explosive Residues Part-1. Chemical tests*, Jr. Foren. Sci., 20, 3, 133.
5. Ervin Jungreis. (1997). *Spot Test Analysis*, 2<sup>nd</sup> edition. Wiley.Interscience.

6. Alexander Beveridge. (1998). *Forensic Investigation of explosions*, Taylor and Francis Ltd, Bristol.
7. Kaplan.M.A. and Zitrin.S.(1977). *Identification of Post Explosion Residues*. J.A.O.A.C, 60,3,619.
8. Parker .R.G.(1975). " *Analysis of Explosives and Explosive residues Part-3. Mono Methyl Aamine Nitrate*, Jr. Foren Sci. 20, 2, 257-260.
9. Lyter.A.H. III.(1983). *A high performance liquid chromatographic (HPLC) Study of Seven Common Explosive Materials*, J.Foren.Sci. 28, 2, 446.
10. Tamiri.T. and Zitrin.S. (1986). *Capillary column Gas chromatography/ Mass spectrometry of Explosives*. J. Energetic materials, 14, 215.
11. Yallop.H.J. *Explosion Investigation* 1980. Foren. Sci. Soc. Academic Press, U.K.
12. J.S.Gharia, R.K.Sinha, U.R.Nair, S.R.Nair \*& R.M. Roy (2000) " Tagging of Explosives for Detection", Defence Science Journal, 50, 1, 45.(PP 45-49)
13. Yehuda, Yinon, *Forensic and Environmental Detection of Explosives*, (1999) J Wiley & sons, N. York.
14. Krishnamurthy R ; Malve M K., Shinde B M , Analytical Report on Bombay Blast cases . March 1993. Journal of Indian Academy of Forensic science 1996 (35) page 54-55.
15. Krishnamurthy .R, Bakre SM , Shinde BM , Simultaneous detection of High Explosives in Post explosion debris by HPTLC with two successive mobile phases . Journal of Planar Chromatography Vol 12 Sept –Oct 1999 Page 394-396.
16. Atul Bajaj, Cijo John, Meenu Singh, *Explosive post blast analysis: A case study*, European Journal of Forensic Sciences, Issue 2, Vol 3, 2016, 1-5.

## CHAPTER – 7

### EVALUATION AND INTREPRETATIONS

**7.1 Title:** -Evaluation and Interpretation of result

**7.2 Scope:** -Guidance for evaluation of test results and expression of opinions/interpretation in Test Reports

**7.3 Purpose:** To have a uniform guide lines for interpretation of results in Test Reports and while presenting the evidence and the limitations involved.

#### **7.4 General**

The explosive experts are required to provide in Test Reports the details of explosives or otherwise based on the analysis result. For bulk materials which are suspected to be explosives the analysis and interpretations are easy. However, for post explosion cases the prediction of original explosive substance (s) based on trace analysis pose challenges due to various factors namely, protection of explosion scene, use of large quantity of water during firefighting in case of fire associated with explosion, sample collection, handling, packing, labelling, preserving, transportation, avoidance of cross contamination and maintenance of chain of custody from the scene of explosion to the laboratory. In the laboratory, also proper care has to be taken to ensure for proper chain of custody, storage of exhibits, cases are to be opened under supervision and exhibits are to be handled with utmost care to ensure the sample integrity, avoidance of cross contamination, proper cleaning of surfaces of case opening area and working space, unambiguous labelling of exhibits as well as reagents and good laboratory practices. The expert has to deal with very small amount of post explosion residues which many a time deny the possibility of repetition. Also, re-examination of post explosion debris by same laboratory or another laboratory with same exhibits often leads to inconclusive and misleading results as most of the available material might have been utilized by the original laboratory by using repeated solvent extraction process to extract the maximum available residues. Care has to be taken while prioritization and sequence of examination, handling of items, selection of test procedures etc. (ref 1)

#### **7.5 Interpretation of Results**

**7.5.1** In case of organic explosives normally residues of original explosives substances are detected in trace level. The selection of test methods are decided by the experts based on the back ground data provided by the forwarding authority, availability of samples, target analytes, matrix effects, control samples and availability of resources both internal and external. Hence, the methods selected to be validated and ensured that it is capable of detecting the

expected trace level of such explosives. It is also important to know about the case history, place of explosion and surroundings. This will help the expert to eliminate the interferences due to surroundings.

**7.5.2** In case of low explosive mixtures and pyrotechnic compositions, for pre blast samples the ingredients can be detected by the methods mentioned in Chapter 4 of this manual. The ingredients are identified in ionic forms and elemental form. It is well known that the low explosives contain mixture of oxidizer and fuel along with flame modifiers, binders, stabilizers etc. (ref 2,3). The post explosion residues of low explosives may contain ions of original ingredients or reaction products depending up on nature of explosion and stoichiometric proportion of oxidizer and fuel.

**Table 24. Some Common Inorganic Improvised Explosive Devices and the Target Analytes in post blast residues (ref 3 & 4)**

| Device   | Composition  | Characteristic anions  | Characteristic cations          | Other ingredients           |
|--|--|--|---------------------------------|-----------------------------|
| ANFO   | Ammonium Nitrate-Fuel oil  | $\text{NO}_3^-$ , $\text{NO}_2^-$  | $\text{NH}_4^+$                 | Hydrocarbons                |
| Black powder                                       | Potassium nitrate, #Sulphur, Charcoal  | $\text{NO}_3^-$ , $\text{NO}_2^-$ , $\text{SO}_4^{2-}$ , $\text{S}_2\text{O}_3^{2-}$ | $\text{K}^+$                    | Sulphur                     |
| Throw down (Jute Bomb)                             | Potassium #Chlorate Arsenic Sulphide   | $\text{ClO}_3^-$ , $\text{Cl}^-$ , $\text{S}^{2-}$ , $\text{SO}_4^{2-}$              | $\text{K}^+$ , $\text{As}^{3+}$ |                             |
| Cracker* (potassium perchlorate+ Aluminium Powder) | Potassium perchlorate, Aluminium powder                                      | $\text{ClO}_4^-$ , $\text{Cl}^-$   | $\text{K}^+$ , $\text{Al}^{3+}$ | Al                          |
| Chlorate/Sugar                                     | Potassium # Chlorate/Sugar   | $\text{ClO}_3^-$ , $\text{Cl}^-$   | $\text{K}^+$                    |                             |
| Perchlorate/Sugar                                  | Potassium Perchlorate*/Sugar   | $\text{ClO}_4^-$ , $\text{Cl}^-$   | $\text{K}^+$                    |                             |
| Chlorate/Sulphur/ Aluminium                        | Potassium Chlorate#/Sulphur/ Aluminium                                       | $\text{ClO}_3^-$ , $\text{Cl}^-$ , $\text{SO}_4^{2-}$ , $\text{S}_2\text{O}_3^{2-}$  | $\text{K}^+$ , $\text{Al}^{3+}$ | Al                          |
| Slurry and Emulsion                                | Ammonium Nitrate Mono Methyl Amine Nitrate/jelling agent,/Higher Hydrocarbon | $\text{NO}_3^-$ ,  | $\text{NH}_4^+$                 | Higher Hydrocarbon fraction |

\*Instead of potassium perchlorate Ammonium perchlorate, or Sodium perchlorate and



**Potassium Chlorate or Sodium Chlorate are also used as oxidizer in Homemade IEDs and anions and cations may change accordingly**

**#Sodium nitrate or Sodium Chlorate is also used in Homemade IEDs and cation may change accordingly**

Some inferences can also be drawn in respect of composition of inorganic anions and cations in mixtures.

1. The presence of ammonium and phosphate can normally be due to ammonium phosphate since this is the only phosphate likely to be present in an industrial blasting explosive.

2. The presence of ammonium and nitrate can normally be due to ammonium nitrate since this is a very common constituent of industrial blasting explosives.

3. The presence of sodium and chloride can normally be due to sodium chloride since this is a common major ingredient of coal mining explosives.

4. If ammonium, nitrate, sodium and chloride are all present this may mean ammonium nitrate and sodium chloride or, alternatively, the reversed ion mixture i.e. ammonium chloride and sodium nitrate. The former is far more likely since reversed ion mixture is very uncommon. The point may be resolved, however, by extracting a portion of explosive with ether and acetone and examining the residue microscopically for the characteristic cubic crystals of sodium chloride(ref 5).

5. The presence of barium normally denotes barytes since this is the only barium salt normally used as a constituent of industrial blasting explosives. By close observation and keen attention in details, experienced operators can make other deductions with varying degrees of certainty. After taking into account any known or suspected contaminants, the observations are then compared with the specified compositions of explosives by manufacturers whose products are normally used in the country in which the investigation is taking place in some other country, if this appears relevant. This may be quickly and conveniently done if the components of the various explosives are recorded on punched cards. It will often be found that only one explosive has the particular combination of ingredients. When there are two or more observations of colour, consistency will often resolve the question. But, if this is not so; then a quantitative determination must be made. It is often not necessary, however, to determine all the ingredients since normally there will be one ingredient that is present in markedly different proportions in the possible explosives of a particular class.

## References

1. *Best Practice Manual for the Forensic Recovery, Identification and Analysis of Explosive Traces* (ENFSI-BPM-EXP-01) Version 01 November 2015 by the European Network of Forensic Science Institutes (ENFSI)
2. KPS Kartha (1991) Study on the Sensitiveness of Pyrotechnic Compositions with Slight Change in Oxidiser Fuel Ratio J. Standards India Vol 5 No.6 pp 192-195 (1991)
3. Joseph P Hutchinson, Christopher J Evenhuis, Cameron Johns, Artaches A. Kazarian, Michael C Breadmore, Miroslav Macka, Emily F Hilder, Rosanne M. Guijt, Greg W. Dicoski and Paul R. Haddad (2007) *Identification of Inorganic Improvised Explosive Devices by Analysis of Post blast Residue Using Portable Capillary Electrophoresis instrumentation and Indirect Photometric Detection with a Light- Emitting Diode*, J of Analytical Chemistry, Vol. 79, No. 18, 2007
4. Mohamad Afiq Mohamed Huri, Umi Kalthomahmad, Roliana Ibrahim, Mustafa Omar (2017) *A Review of Explosive residue detection from forensic chemistry perspective*, Malaysian Journal of Analytical Sciences, Vol 21 No.2 (2017) pp 267-282
5. E. Suresh Babu and Sukhminder Kaur *A DSC Analysis of Inverse Salt-pair explosive Composition* Journal of Propellants, Explosives, Pyrotechnics 29 (2004) No.1 p 50-55

## CHAPTER – 8

### IDENTIFICATION OF MISCELLANEOUS EXPLOSIVES

**8.1 Title:** -Identification of Miscellaneous Explosives

**8.2 Scope:** -To identify explosives which are not commonly encountered

**8.3 Purpose:** To have a procedure to identify explosives which are not commonly used and to keep the manual open document so as include procedures for new explosive materials. Some of the miscellaneous explosive materials and their test procedures are mentioned below

#### **8.3.1 PEROXIDE EXPLOSIVES**

The peroxide explosives TATP and hexamethylenetriperoxidediamine (HMTD) have become popular with terrorists because they are easily prepared from readily obtainable ingredients, although the synthesis is fraught with danger. Although they do not contain NO<sub>2</sub> groups, the O–O bond is a source of oxygen available for potentially rapid self-oxidation and explosion. Although neat hydrogen peroxide (H<sub>2</sub>O<sub>2</sub>) is detonable, most of the common industrial peroxides, which contain only one O–O functionality per molecule, have insufficient oxygen to gasify the majority of the C and H atoms in the molecule. These peroxides are not usually considered explosives, even though some have a reported “TNTequivalence”. As TATP and HMTD contain three peroxide linkages per molecule, their explosive output is much higher than most organic peroxides. TATP is estimated as 88% and HMTD as 60% of TNT blast strength.

The unusual danger to public safety in the case of peroxide explosives is not their explosive performance but their ease of initiation and the ease with which terrorists have acquired and used the materials for their synthesis, although synthesis is actually quite hazardous. Both TATP and HMTD are classed as primary explosives. Hydrogen peroxide at the correct concentration is also detonable. (ref 3 p21). The detection of post explosion residues of peroxide is very difficult as most of the material will be consumed during explosion.

##### **8.3.1.1 TATP-Triacetone triperoxide (TATP)**

Triacetone triperoxide (TATP) is a white crystalline solid material. It is one of the most sensitive explosive known. Being extremely sensitive to impact, temperature change and friction, it is very easy to detonate. It is an organic peroxide containing neither metallic elements nor nitro group. Therefore the presence of peroxide group cannot be detected by standard security procedure like transmission and vapor detector. (ref 1&3).

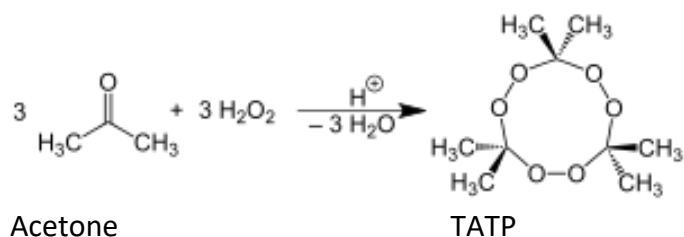
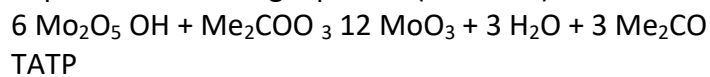


Table-25- Properties of TATP

|                   |   |
|-------------------|---|
| Colour            | White crystalline   |
| Solubility        | Insoluble in water,   |
| Empirical formula | C <sub>9</sub> H <sub>18</sub> O <sub>6</sub>                 |
| Molecular weight  | 222.2   |
| Melting Point     | 98 <sup>0</sup> C   |
| VOD               | 5300 <u>m/s</u> at maximum density (1.18 g/cm <sup>3</sup> ), |

#### 8.3.1.1.1 Colorimetric detection of TATP

The dark blue color of molybdenum hydrogen bronze suspension is changed to yellow upon oxidation with TATP. The same reagent can also be used for quick neutralization of the sensitive explosive: a lasting final blue color indicates complete neutralization. The reaction with TATP is depicted in following equation (ref 4 P51)



#### 8.3.1.1.2 Thin Layer Chromatography (TLC) (ref 5)

Conditions-

Plate-Merck pre-coated silicagel 60 F254,0.25mm on glass

Sample- in ethyl acetate

Development Solvent- Toluene

Spray Reagent -1%diphenylamine in conc.H<sub>2</sub>SO<sub>4</sub>

Rf value -0.57

#### 8.3.1.1.3 High Performance Liquid Chromatography (HPLC) (ref. 6)

Conditions-

Instrument-HPLC-DAD-Fluorescence

Column-Merck LiChrospher RP 18: Particle size 5µm Pore size 300 Å, Column dimensions – 250 mm x 3 mm.

For UV irradiation- low pressure mercury lamp with 6 W

Mobile Phase-Acetonitrile:Water-60:40

Flow Rate- 0.6mL/min

Post column derivatization-

Immediately after the separation of analytes the latter irradiated with UV light of 254 nm. A 25 m knitted Teflon tubing (inner diameter 0.3 mm) wrapped around the UV lamp and used as reaction loop. After decomposition of the analytes to hydrogen peroxide, two HPLC pumps are introduced to deliver the derivatization reagents 1 (a solution of 205mg 91.18 mmol) of  $K_2HPO_4$  and 7.6 mg (0.05 mmol) of *p*-hydroxyphenylacetic acid (*p*HPAA) in 100 mL of water) and 2 (a solution of 8.4 mg of horseradish peroxidase in 100 mL of water). Both reagents are added with a flow rate of 0.2mL/min. giving an overall flow rate of 1 mL/min. The following reaction loop of 8m knitted Teflon tubing coil (inner diameter 0.3 mm) which is heated to 40 °C. The excitation wavelength of the fluorescence detector set to 324 nm and the emission wavelength 405 nm.

#### **8.3.1.1.4 FT-IR (ref 10)**

Mode- Attenuated Total Reflectance (ATR)

Number of background scans 64

Number of sample scans 64

Resolution 4  $cm^{-1}$

Spectral range ( $cm^{-1}$ ) 4000-650

Final data spacing 1.928  $cm^{-1}$

Detector MCT/A

Optical velocity (cm/s) 1.898

Aperture 69

Principal wave numbers at 3946, 2998, 1376, 1361, 1177,884,784  $cm^{-1}$

#### **8.3.1.1.5 GC-MS**

Conditions

Injector: SSL injector in split mode (ratio 1:10)

Carrier gas: Helium @1.2 mL/min

Injection Volume: 0.2 $\mu$ L

Capillary column: TG-5MS (15m $\times$ 0.25mm $\times$ 0.25 $\mu$ m)

Oven: 50°C hold for 3 min.

10°C/min to 200°C and hold for 2.0 min

Total run time: 20 min

Detector: ISQLTMS

Ionisation Source: EI @70eV

Transfer Line Temperature: 200°C

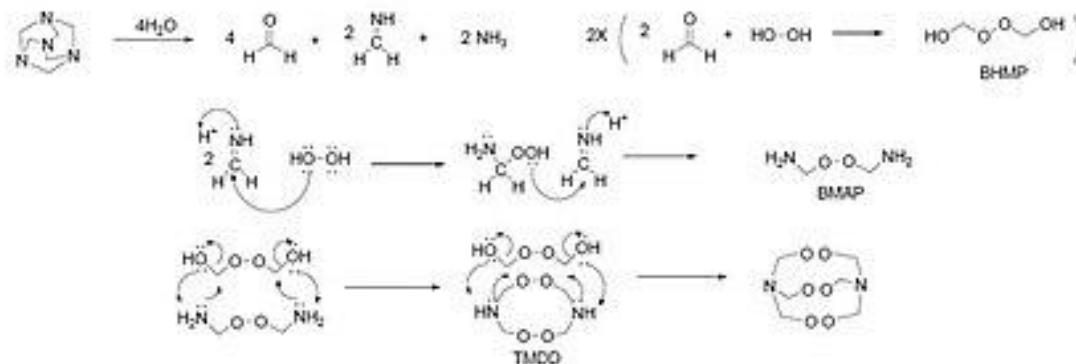
Source Temperature: 2000C

Data acquisition mode: Full scan (identification):35 to 350m/z

SIM masses (Quantification) : 43,58,75 m/z

### 8.3.1.2 Hexamethylene tri peroxide diamine (HMTD)

Hexamethylenetriperoxydiamine is a high explosive organic compound. HMTD is an organic peroxide, a heterocyclic compound with a cage-like structure. It is a primary explosive. At 40°C HMTD begins to break up into volatile pungent compounds. (ref 1)



HMTD

Table-26- Properties of HMTD

|                   |   |
|-------------------|---|
| Colour            | White crystalline                             |
| Solubility        | Insoluble in water,                           |
| Empirical formula | $\text{C}_6\text{H}_{12}\text{N}_2\text{O}_6$ |
| Molecular weight  | 208.2   |
| Melting Point     | Decomposes at 75°C                            |
| VOD               | 5100 m/s at around 1.1 g/cm <sup>3</sup>      |

#### 8.3.1.2.1 Colorimetric detection of HMTD (ref 1)

To water solution, 1-2 drops of 1% TSS (N,N-diethyl-p-phenyldiaminesulphate) added cherry red colour indicates the presence of HMTD.

#### 8.3.1.2.2 High Performance Liquid Chromatography (HPLC) (ref. 6)

Conditions-

Instrument-HPLC-DAD-Fluorescence

Column-Merck LiChrospher RP 18: Particle size 5µm Pore size 300 Å, Column dimensions – 250 mm x 3 mm.

For UV irradiation- low pressure mercury lamp with 6 W

Mobile Phase-Acetonitrile: Water-60:40

Flow Rate- 0.6mL/min

Post column derivatization-

Immediately after the separation of analytes the latter irradiated with UV light of 254 nm. A 25 m knitted Teflon tubing (inner diameter 0.3 mm) wrapped around the UV lamp and used as reaction loop. After decomposition of the analytes to hydrogen peroxide, two HPLC pumps are introduced to deliver the derivatization reagents 1 (a solution of 205mg 91.18 mmol) of  $K_2HPO_4$  and 7.6 mg (0.05 mmol) of *p*-hydroxyphenylacetic acid (*p*HPAA) in 100 mL of water) and 2 (a solution of 8.4 mg of horseradish peroxidase in 100 mL of water). Both reagents are added with a flow rate of 0.2mL/min. giving an overall flow rate of 1 mL/min. The following reaction loop of 8m knitted Teflon tubing coil (inner diameter 0.3 mm) which is heated to 40 °C. The excitation wavelength of the fluorescence detector set to 324 nm and the emission wavelength 405 nm.

### 8.3.1.2.3 GC-MS

#### Conditions

Injector: SSL injector in split mode (ratio 1:10)

Carrier gas: Helium @1.2 mL/min

Injection Volume: 0.2µL

Capillary column: TG-5MS (15m x 0.25mm x 0.25µm)

Oven: 50°C hold for 3 min.

10°C/min to 200°C and hold for 2.0 min

Total run time: 20 min

Detector: ISQLTMS

Ionisation Source: EI @70eV

Transfer Line Temperature: 200°C

Source Temperature: 2000C

Data acquisition mode: Full scan (identification): 35 to 350m/z

SIM masses (Quantification) : m/z 42 ( $C_2H_3N^+$ ), 73 ( $C_2H_3NO_2^+$ ), 176 (HMTD- $O_2^+$ )

### 8.3.1 Urea Nitrate (Uronium nitrate)

Urea Nitrate is a powerful explosive frequently made and used by terrorists. It can be prepared quickly and easily by adding Nitric Acid to a cooled aqueous solution of urea. The white precipitate thus formed is filtered, washed with cold water and dried in air (ref 4 p- 52)

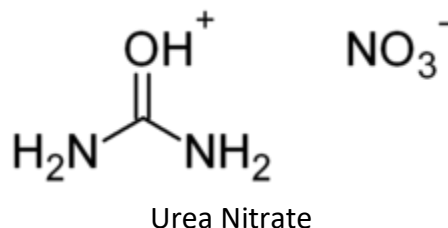


Table-27- Properties of Urea Nitrate

|                   |   |
|-------------------|---|
| Colour            | White crystalline                             |
| Solubility        | Soluble in water and alcohol                  |
| Empirical formula | CH <sub>5</sub> N <sub>3</sub> O <sub>4</sub> |
| Molecular weight  | 123.1   |
| Melting Point     | 163 <sup>0</sup> C                            |
| VOD               | 4700 m/s                                      |

### 8.3.2.1 Colorimetric detection of Urea Nitrate (ref 4 p52)

1. Urea nitrate treated with ethanolic solution of p-dimethylaminocinnamaldehyde (p-DMAC) under neutral conditions. **A red pigment is formed within 1 min from contact.** The limit of detection on filter paper is  $\approx 0.1$  mg/cm<sup>2</sup>. Urea itself, which is the starting material for urea nitrate, does not react with p-DMAC under the same conditions. Other potential sources of false-positive response such as common fertilizers, medications containing the urea moiety and various amines, do not produce the red pigment with p-DMAC.

2. p-Dimethylaminobenzaldehyde (p-DMAB) which is one vinyl shorter than p-DMAC, also reacts with urea nitrate under similar conditions, to produce a typical yellow pigment of analogous structure. The essence of the proposed technique lies in the fact that urea nitrate, as opposed to urea, is strongly acidic (pH 1–2) because of the presence of the nitric acid moiety in its molecule. Thus, although neutral urea does not react with p-DMAC, urea nitrate does, as it provides the necessary acidity for the reaction to occur. p-DMAC does not detect urea, but uronium ion.

### 8.3.2.2 FTIR (ref.8)

#### Conditions

Detector: DTGS TEC

Beam splitter: KBr

Spectral range: 650 to 4000 cm<sup>-1</sup>

Resolution: 4.0 cm<sup>-1</sup>

Mode: Attenuated Total Reflectance (ATR) for solids

Scans: 32

Principal wave numbers: 3402 cm<sup>-1</sup> (NH<sub>2</sub> asymmetric stretch), 3354 cm<sup>-1</sup> (NH stretch), 3198 cm<sup>-1</sup> (NH<sub>2</sub> symmetric stretch), 1704 cm<sup>-1</sup> (CO stretch), 1568 cm<sup>-1</sup> (N-H), 1426 cm<sup>-1</sup> (C-N), 1298 cm<sup>-1</sup> (NO<sub>3</sub><sup>-</sup>)



### 8.3.2.3 GC-MS (ref.9)

#### Conditions

Column: Fused silica capillary Column 30mX 0.25 mm (id) coated with HP-5 MS (0.25µm film)

Injector Temperature: 180<sup>0</sup>C

Column Temperature: programmed from 50<sup>0</sup>C to 180<sup>0</sup>C at a rate of 15<sup>0</sup>C/min,10 min at 180<sup>0</sup>C, 180-300<sup>0</sup>C at a rate of 25<sup>0</sup>C/min and 5 min at 300<sup>0</sup>C.

Transfer Line was held at 230<sup>0</sup>C.

Ion Source Temperature: 180<sup>0</sup>C

Quadrupole Temperature: 120<sup>0</sup>C

Scan range: 29-350 Da

Scan Rate : 2.29 scans/s

Electron energy :70eV

Carrier gas: Helium

Injection: Split less mode

#### Preparation of xanthrolalkylcarbamates (xantylurethanes)

Sample and xanthrol are reacted according to the method described by Mc Cord and de Perre (ref C.de Perre, B McCord, Trace analysis of urea nitrate by Liquid chromatography-UV/fluorescence, Forensic Sci. Int.211(2011) 76-82) using three alcohols as solvents; methanol, ethanol and 1-propanol.The products have been characterized in the reaction mixtures by GC-MS.

### 8.3.3 Quantitative Estimation of Ammonium Nitrate

Vide Notification No. G.S.R. S.O. 1678(E) dated 21/07/2011, Ammonium Nitrate having the chemical formula  $\text{NH}_4\text{NO}_3$  or any combination containing more than 45 percent of ammonium nitrate by weight including emulsions, suspensions, melts or gels (with or without inorganic nitrates) shall be deemed to be an explosive under the meaning of the Explosives Act 1884. Provided that such ammonium nitrate or any combination thereof shall not include those fertilizers from which ammonium nitrate cannot be extracted by any physical or chemical means.(ref.12). Hence, it is important that the purity of ammonium nitrate needs to be estimated to include it under the purview of the act or otherwise.

#### 8.3.1 Determination of Purity of Ammonium Nitrate on dry basis percent by mass

(ref11 Test Method A-13)

The test method is reproduced here with for ready reference

**Outline of the Method-** Ammonia from ammonium nitrate reacts quantitatively with excess of formaldehyde to produce nitric acid



This nitric acid can be titrated against standard alkali.

### 8.3.3. 1.Reagents

**8.3.3.1.1 Sodium Hydroxide:** 0.2 N

**8.3.3.1.2 Hydrochloric Acid:** 0.2 N

**8.3.3.1.3 Phenolphthalein:** 1 percent in alcohol

**8.3.3.1.4 Formaldehyde:** 1:1- Mix equal volumes of AR grade formaldehyde ( 37 percent m/m) and water. Add few drops of phenolphthalein indicator and titrate against standard alkali to its light pink end point.

**8.3.3.2 Procedure-** Weigh exactly 0.4 g of sample into a 250 ml conical flask and dissolve in 20 ml of water. Add 50 ml of formaldehyde 1:1 reagent and warm the flask. Cool to room temperature and titrate against standard alkali till permanent light pink colour appears. Drain another 2 ml of alkali and note down the volume ( $V_1$ ). Keep it for 10 minutes and titrate against standard hydrochloric acid using a calibrated micro burette till the solution becomes colourless and note down the volume ( $V_2$ ). Repeat the same experiment till concordant values are obtained

### 8.3.3.3 Calculations

Volume of sodium hydroxide required ( $V$ ) =  $V_1 - \frac{V_2 \times N_2}{N_1}$

$N_1$

Percent purity of ammonium nitrate =  $\frac{V \times N_1 \times 8.005}{M}$

$M$

**Purity ( on dry basis) percent by mass =  $\frac{100 \times \text{Purity as obtained above}}{(100 - \text{moisture percent})}$**

Where

$N_1$  = Normality of sodium hydroxide

$N_2$  = Normality of Hydrochloric acid, and

$M$  = Mass of the sample in g

## NOTE:

Uncertainty of measurement for the quantitative estimation may be calculated as per ref. 13 by considering all the contributory factors.

## References

1. Chladek J. (1993) *The identification of organic peroxides. Advances in analysis and detection of explosives*. Proceedings of the 4<sup>th</sup> International symposium on Analysis and detection of Explosives September 7-10: 1992; J Yinon edition. Kluwer academic publishers, 1993;73-76
2. Alvaro J Pena-Quevedo and Samuel P. Hernandez-Rivera (2009) *Mass spectrometry analysis of Hexamethylene Triperoxide Diamine by its Decomposition Products*. Detection and Sensing of Mines, Explosive Objects and Obscured Targets XIV edited by Russell S. Harmon J Thomas Broach, John H. Holloway Jr. Proceedings of SPIE Vol 7303, 730303, 2009.
3. Maurice Marshall and Jimmie Oxley (2009) *Aspects of Explosives Detection* Elsevier 1<sup>st</sup> Edition, 2009
4. J. Almog and S. Zitrin (2009) *Colorimetric Detection of Explosives- Aspects of Explosives Detection* Elsevier 1<sup>st</sup> Edition, 2009
5. Anthony J Bellamy (1999) *Triacetone Triperoxide: Its Chemical Destruction*, Journal of Forensic science 1999, 44(3) p 603-608
6. Rasmus Schulte-Ladbeck, Peter Kolla and Uwe Karst (2003) Trace Analysis of Peroxide- Based Explosives, Journal of Analytical Chemistry, Vol. 75 No. 4, February 15, 2003, p 731-735
7. Jimmie Oxley, James Smith, Joseph Brady, Faina Dubnikova, Ronnie Kosloff, Leila Zeiri and Yehuda Zeiri (2008) *Peroxide- Based Explosives*, Applied Spectroscopy, Vol 62 No. 8, 2008 p 906-915
8. Jimmie C. Oxley; James L. Smitha ; Sravanthi Vadlamannatia ; Austin C. Brown a ; Guang Zhanga ; Devon S. Swanson a ; Jonathan Canino a *Synthesis and Characterization of Urea Nitrate and Nitrourea*. [energetics.chm.uri.edu/system/files/PEPUNNUManuscript11](http://energetics.chm.uri.edu/system/files/PEPUNNUManuscript11).
9. Joseph Almog, Dario Espino, Tsippy Tamiri, Dana Sonenfeld (2013) *Trace analysis of urea nitrate in post-blast debris by GC/MS* Forensic Science International Vol. 224 1-3, 10 January, 2013 p 80-83
10. Clayton S.C. Yang , Barry R. Williams, Ashish Tripathi, Melissa S. Hulet, Alan C. Samuels , Joseph A. Domanico, Joseph May, Ronald W. Miles, Jr., Augustus W. Fountain III (April, 2014) Spectral Characterization of RDX, ETN, PETN, TATP, HMTD, HMX, and C-4 in the Mid- Infrared Region. [apps.dtic.mil/dtic/tr/fulltext/u2/a600508.pdf](http://apps.dtic.mil/dtic/tr/fulltext/u2/a600508.pdf)
11. Bureau of Indian Standards, Indian Standard Specification for Ammonium Nitrate for Explosives (IS :4668-1985, Reaffirmed 2011) First Revision

12. Ammonium Nitrate Rules, 2012 Published in the Gazette of India Extra ordinary Part II dated 11th July,2012.
13. EURACHEM / CITAC Guide CG 4 Quantifying Uncertainty in Analytical Measurement Third Edition [www.eurachem.org/images/stories/Guides/pdf/QUAM...](http://www.eurachem.org/images/stories/Guides/pdf/QUAM...)

## APPENDIX-A

(Page 01 of 08)

Form No.

### WORK SHEET FOR EXPLOSIVES ANALYSIS

1. Case No.

2. Date(s) of examination:

#### I Examination of High Explosives

| Extraction with Ether | Extraction with Acetone |
|-----------------------|-------------------------|
|                       |                         |

#### 1) Chemical Tests for High Explosives : Acetone Extract

| Sample | Alcoholic KOH Test | DPA Test  | Thymol+<br>H <sub>2</sub> SO <sub>4</sub> + Ethanol Test | Ethylene diamine Test |
|--------|--------------------|-----------|--|-----------------------|
| TNT    | Purple Red         | No Colour | No Colour  | Maroon                |
| RDX    | No Colour          | Blue      | Blue   | No Colour             |
| PETN   | No Colour          | Blue      | No colour  | No Colour             |
| Tetryl | Red violet         | Blue      | No colour  | No Colour             |
| NG     | No Colour          | Blue      | No Colour  | No Colour             |
| HMX    | No Colour          | Blue      | Pale blue green  | No Colour             |
| Ex-No. |                    |           |  |                       |
| Ex No  |                    |           |  |                       |
| Ex No. |                    |           |  |                       |
| Ex No  |                    |           |  |                       |

Signature of Test Scientist

Signature of Reporting Officer

Case No.

Form No.

(2) **TLC/HPTLC Test**  
**(a) Experimental Conditions**

Solvent System A:

Solvent System B:

Solvent System C:

Spray Reagent:

HR<sub>f</sub>/λ<sub>max</sub> values

Results

**(3) HPLC with PDA Detector Analysis :**  
**(a) Experimental Conditions**

Mobile Phase:

Column:

Flow Rate:

**(b)Results**

Signature of Test Scientist

Signature of Reporting Officer

Case No.

Form No.

**(4) GC MS Analysis for High Explosives and Hydro carbons:**

**(a) Experimental Conditions**

Column : fused silica capillary, 15m x 0.25 mm ID with 0.25 mm coating of DB-5.

Temperature : Programmed from 70 °C to 270 °C at the rate of 15 °C/ min.

Injector Temp : 180 °C

Ion source temp: 140 °C

Carrier gas : Helium

Scan range : 30-500 or 40-500 mass units

Scan rate : 1 scan/ sec

Electron energy: 70 eV in electron impact (EI) and chemical ionisation (CI) modes

**(b) Results**

**(5) Any other Technique**

**(a) Experimental Conditions**

**(b)Results**

Signature of Test Scientist

Signature of Reporting Officer

Case No.

Form No.

**II. Chemical Examination for anions****Extraction:**

|               |              |                  |
|---------------|--------------|------------------|
| Water extract | NaOH extract | Pyridine extract |
|               |              |                  |

**1. Chemical Tests for anions and observation**

| Name of the Anions             | Name of the Test conducted      | Observation With control                                       | Exhibit No. |  |  |  |  |  |  |  |  |  |
|--------------------------------|---------------------------------|--|-------------|--|--|--|--|--|--|--|--|--|
|                                |                                 |  |             |  |  |  |  |  |  |  |  |  |
| Chloride                       | AgNO <sub>3</sub> Test          | White curdy ppt sol in NH <sub>4</sub> OH                      |             |  |  |  |  |  |  |  |  |  |
| Nitrite                        | Griess Test                     | Pink colour  |             |  |  |  |  |  |  |  |  |  |
| Nitrate                        | (Griess reagent + Zn dust) Test | Pink colour  |             |  |  |  |  |  |  |  |  |  |
| Chlorate                       | Aniline sulphate Test           | Deep Blue colour   |             |  |  |  |  |  |  |  |  |  |
| Perchlorate                    | Methylene blue indicator Test   | Violet spot  |             |  |  |  |  |  |  |  |  |  |
| Sulphate                       | BaCl <sub>2</sub> Test          | White crystalline ppt  |             |  |  |  |  |  |  |  |  |  |
| Sulphide<br>Na OH<br>extract   | Sodium nitroprusside Test       | Violet colour  |             |  |  |  |  |  |  |  |  |  |
| Sulphur<br>Pyridine<br>Extract | Pyridine + NaOH                 | Blue to Green for low conc.<br><br>Red to Brown for high conc. |             |  |  |  |  |  |  |  |  |  |
| Sugar **                       | Fehling Test                    | Brown to Red   |             |  |  |  |  |  |  |  |  |  |

\*\* Optional Tests to be done if these are suspected

Signature of Test Scientist

Signature of Reporting Officer



Case No.

Form No.

**2. Instrumental Analysis (if required):**

**4.7 Ion Chromatography for Inorganic Anions**

**(a) Experimental Condition**

Mobile Phase A:

Mobile Phase B:

Mobile Phase C:

Column :

Flow Rate:

**(b) Results-**

**2.2. Any Other Technique**

**(a) Experimental Condition**

**(b) Results-**

Signature of Test Scientist

Signature of Reporting Officer

Case No.

Form No.

**Chemical Examination for cations**

Extraction:

|               |              |
|---------------|--------------|
| Water extract | NaOH extract |
|               |              |

**1. Chemical Examination for cations:**

| Name of the cation          | Name of the Test conducted                          | Observation with control | Exhibit No. |  |  |  |  |  |  |
|-----------------------------|---|--------------------------|-------------|--|--|--|--|--|--|
|                             |   |                          |             |  |  |  |  |  |  |
| Sodium                      | Zinc Uranyl Acetate Test                            | Yellow ppt               |             |  |  |  |  |  |  |
|                             | Flame Test with Pt wire                             | Golden Yellow            |             |  |  |  |  |  |  |
| Potassium                   | Sodium Cobalti Nitrate Test                         | Yellow ppt               |             |  |  |  |  |  |  |
|                             | Dipicryl amine Test                                 | Orange spot              |             |  |  |  |  |  |  |
|                             | Flame Test with Pt. Wire                            | Violet (lilac)           |             |  |  |  |  |  |  |
| Ammonium                    | Nessler's reagent                                   | Orange brown ppt         |             |  |  |  |  |  |  |
| Magnesium*                  | Para-nitro benzene azo resorcinol (Magneson I Test) | Blue ppt                 |             |  |  |  |  |  |  |
| Barium*                     | Sodium Rhodizonate Test                             | Red-brown spot           |             |  |  |  |  |  |  |
|                             | Flame Test  | Yellowish green          |             |  |  |  |  |  |  |
| Calcium*                    | Sodium Rhodizonate Test                             | Violetppt                |             |  |  |  |  |  |  |
|                             | Flame Test  | Brick Red                |             |  |  |  |  |  |  |
| Strontium*                  | Sodium Rhodizonate Test                             | Brownish red             |             |  |  |  |  |  |  |
|                             | Flame Test  | Crimson                  |             |  |  |  |  |  |  |
| Aluminium<br>(NaOH extract) | Aluminon reagent                                    | Brown flakes or ppt      |             |  |  |  |  |  |  |
|                             | Alizarine – S Test                                  | Red ppt                  |             |  |  |  |  |  |  |

|  |                  |                         |  |  |  |  |  |  |  |  |
|--|------------------|-------------------------|--|--|--|--|--|--|--|--|
| Arsenic<br>(As <sup>+3</sup> ) (NaOH<br>extract) | Gutzeit's Test   | Yellow to Brown<br>spot |  |  |  |  |  |  |  |  |
|  | Reinsch's Test   | White sublimate         |  |  |  |  |  |  |  |  |
| Antimony<br>(Sb <sup>+3</sup> )                  |                  |                         |  |  |  |  |  |  |  |  |
|  | Rhodamine-B Test |                         |  |  |  |  |  |  |  |  |

\* To be conducted if pyrotechnic compositions are suspected

**2. Instrumental Analysis( if required)**

**(2.1) Ion Chromatography test for Inorganic cations**

**(a)Experimental Condition**

Mobile Phase A:

Mobile Phase B

Mobile Phase C

Column :

Flow Rate:

**(b) Result**

Signature of Test Scientist

Signature of Reporting Officer

(Page 07 of 08)

Case No.

Form No.

**3. Any Other Technique**

**(a) Experimental Condition**

**(b) Result**

Note: Tests were carried out as per Working Procedure Manual Document No

**Inference:**

Signature of Test Scientist

Signature of Reporting Officer

LAST PAGE

### Formation of committees at National Level for formulation of SOPs and Manuals:

**Background:** In view of technological advancements in the scientific arena, the Standard Operating Procedures (SOPs) and Working Procedure Manuals, around which the technical and analytical exercise takes place in the laboratory in the examination of crime exhibits, needs periodical review to keep the laboratory updated.

For uniform SOP/Manuals and reporting pattern in all the CFSLs / State FSLs following committees were formed by JS (PM), MHA by including members from Central and State FSLs in the following areas:

| Discipline                        | CFSL Member  | Member  |
|-----------------------------------|--|---|
| Biology/DNA                       | Dr. A. K. Sharma, Director, CFSL, Kolkata/Guwahati   | 1. Sh. Arun Sharma, Director, FSL, HP<br>2. Sh. Srikumar, Director, Chemical Examiner Lab, Thiruvananthapuram.  |
| Chemistry/ Narcotics              | Sh. K. M. Varshney, Coordinator, CFSL, Pune  | 1. Dr. R. K. Gupta, Director, FSL, Chhattisgarh.<br>2. Sh. B Shanmukham, Director, FSL, Puducherry.<br>3. Dr. Harsh Sharma, Director, FSL, Sagar (MP) |
| Explosives                        | Dr. Sukhminder Kaur, Coordinator CFSL, Pune  | 1. One officer from FSL, Delhi<br>2. One officer from FSL, Maharashtra  |
| Toxicology                        | Dr. Vimukti Chauhan, SSO, CFSL, Chandigarh   | 1. Dr. K. V. Kulkarni, Director, DFSL, Maharashtra<br>2. One officer from FSL, Karnataka.   |
| Ballistics                        | Sh. S. S. Baisoya, CFSL Chandigarh   | 1. Dr. D. K. Kaushal, Director, FSL, Haryana<br>2. Sh. N. P. Waghmare, Director, FSL, Goa<br>3. Dr. S. S. Das, Director, FSL, Odisha                  |
| Documents                         | Sh. M. C. Joshi, Dy. Director, CFSL, Chandigarh (Shimla Unit) and Dr. S. Ahmad, DFSS HQs., New Delhi | 1. Ms. Deepa Verma, Director, FSL, Delhi  |
| Psychology, Computer, Audio-Video | Dr. S. K. Jain, Director, CFSL, Chandigarh and Sh. M. Krishna, AD, CFSL, Hyderabad                   | Officers from FSL: HP, Delhi, Gujarat and Maharashtra   |
| Crime Scene                       | Dr. M. Baskar, Dy. Director (Physics), CFSL, Chandigarh  | 1. Dr. Harsh Sharma, Director, FSL (MP)<br>2. Sh. R. K. Gupta, Jt. Director, FSL, Chhattisgarh  |

The officers of CFSLs will coordinate with the experts of State FSLs for convening of meeting(s) in the state and Central FSLs and finalization of SOPs and manuals.