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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.

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(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.

19/3/2021

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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-$Pb(N_3)_2$

Lead trinitroresorcinate- Lead Styphnate

LOX- Liquid Oxygen Explosive

Mercury Fulminate-(Hg (ONC)2

NC- Nitrocellulose

NG-Nitroglycerine

PETN- Pentaerythritol tetra nitrate

RDX- Cyclotrimethelenetrinitramine

Picric Acid -2,4,6-trinitro phenol

SEM-Scanning Electron Microscopy

TATP- Triacetonetriperoxide

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 substancesthat 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/Industrialand 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 energyin 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 areproducedby 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 classifiedin 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_2) group. They are sub divided according to the atom to which the NO_2 group is attached.

Nitro compounds	-	C-NO_2group,
Nitrate esters	-	C-O-NO_2 group
Nitramines	-	C-N-NO_2 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)

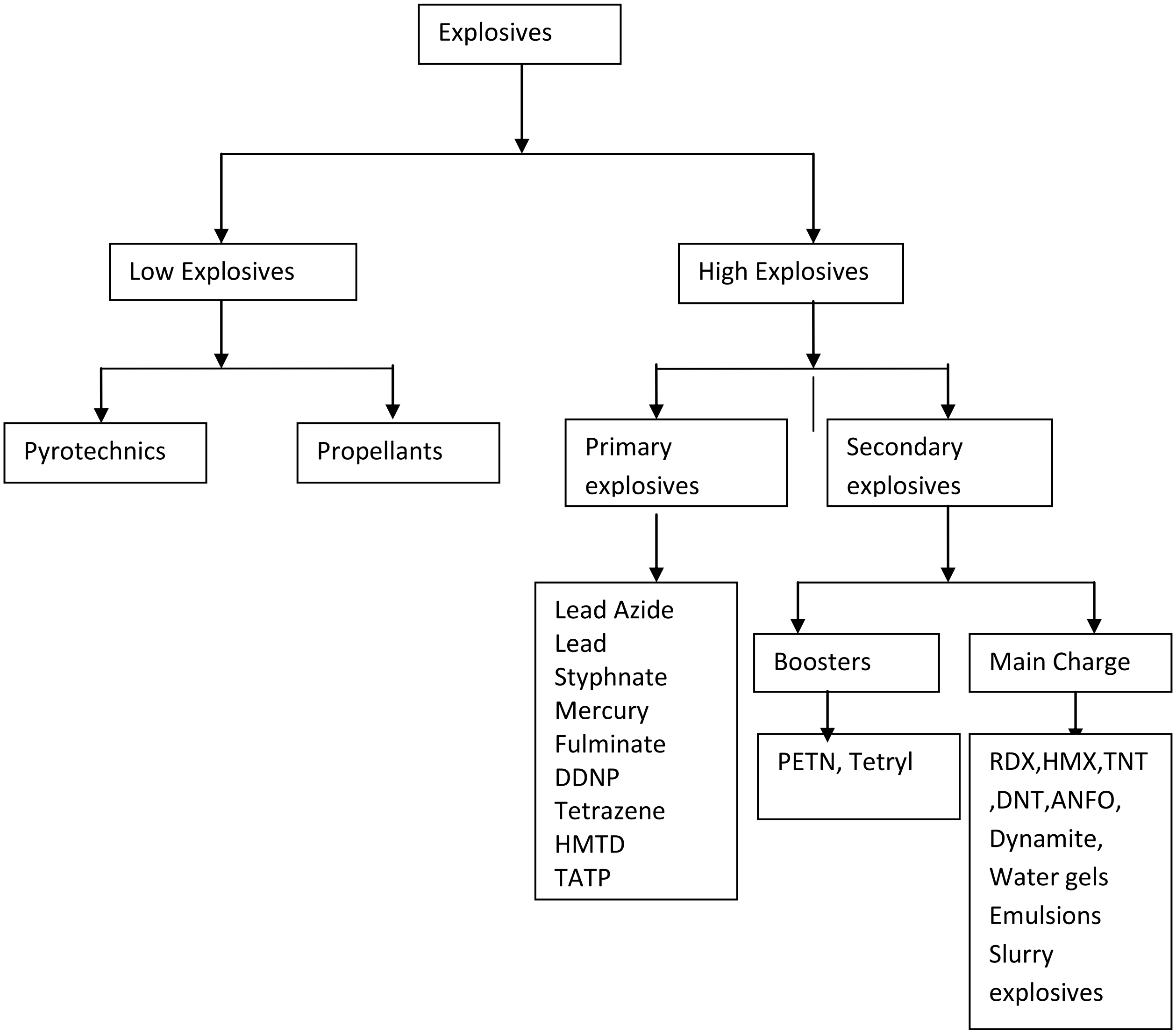

Figure-1 Classification of explosives

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^{-1}	Few centimeters to meter s^{-1}
4.	Velocity of flame front	1.5 to 10 km s^{-1}	0.5 to 1.5 km s^{-1}
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^{-1}	Less than 1000 m s^{-1}

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 ($Hg(ONC)_2$ 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	$C_2N_2O_2Hg$
Molecular weight	284.6
Deflagration point	165^0C
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^{0}C
Impact sensitivity	0.25-0.4 kpm

1.10.1.3Lead 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 azideand 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.

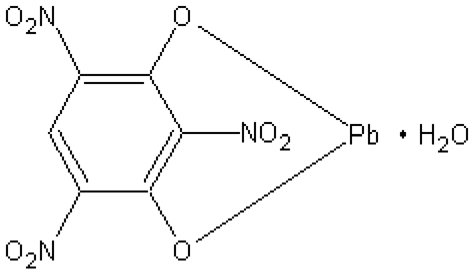

Figure-2- Chemical structure of compound

Table-4- Properties of Lead Styphnate

Colour	Reddish brown crystals
Solubility	insoluble in water
Empirical formula	$C_6HN_3O_8Pb$
Molecular weight	468.3
Deflagration point	275 - 280^{0}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.

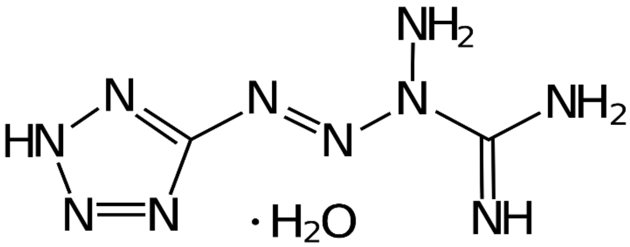

Figure-3- Chemical structure of compound

Table-5- Properties of Tetrazene

Colour	Pale yellow crystals
Solubility	insoluble in water
Empirical formula	$C_2H_6N_{10.}\ H_2O$
Molecular weight	188.2
Deflagration point	140^{0}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.

1.10.2.1Picric 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.

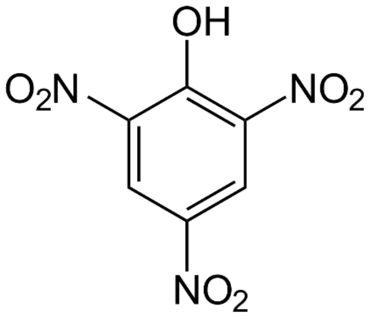

Figure-4- Chemical structure of Picric Acid

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	300^0C
Impact sensitivity	0.75kpm
Solidification point	122.5^0c

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 bombsused 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

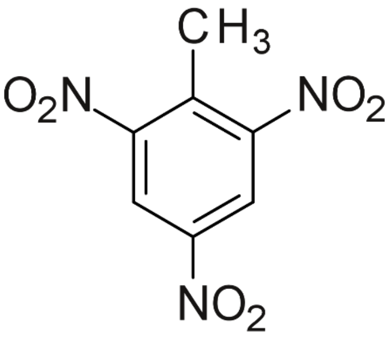

Figure-5- Chemical structure of TNT

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	300^0C
Impact sensitivity	1.5kpm
Solidification point	80.8^0c

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.

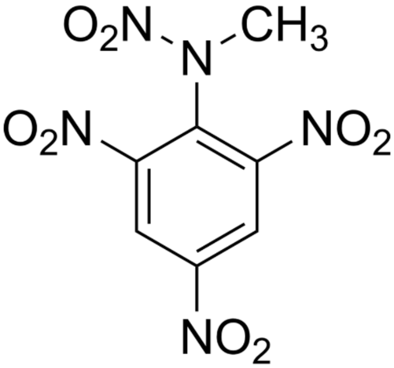

Figure-6- Chemical structure of Tetryl

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	185^0C
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.

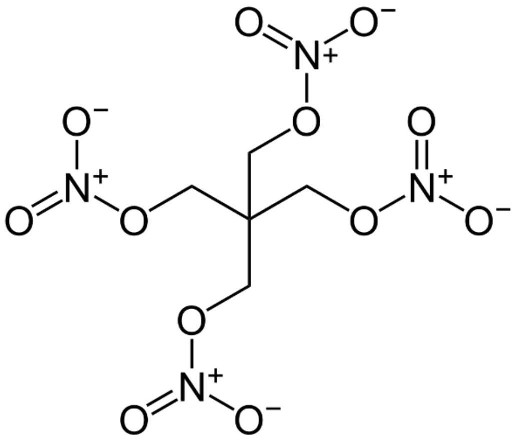

Figure-7- Chemical structure of PETN

Table-10- Properties of PETN

Colour	colourless crystals
Solubility	insoluble in water, soluble in acetone and methyl acetate
Empirical formula	$C_5H_8N_4O_{12}$
Molecular weight	316.1
Deflagration point	202^0C

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 fordetonation. 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.

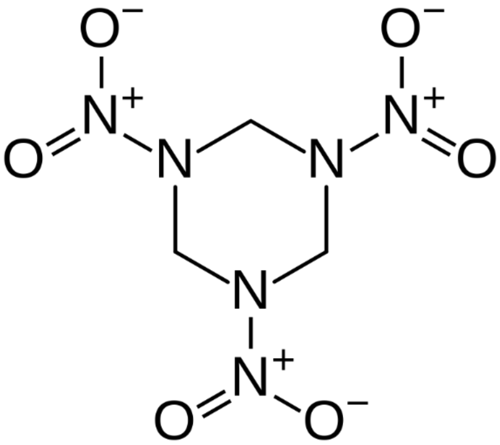

Figure-8- Chemical structure of RDX

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	204^0C
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 propellent.HMX exists as α (orthorhombic), β (monoclinic), γ (monoclinic) and δ (hexagonal) forms, of which 'β' 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.

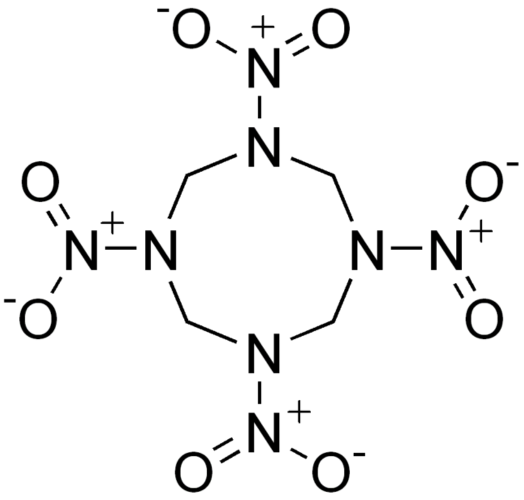

Figure-9- Chemical structure of HMX

Table-12- Properties of HMX

Colour	colourless crystals
Solubility	insoluble in water, spiranglysoluble in acetone and ether.
Empirical formula	$C_4H_8N_8O_8$
Molecular weight	296.2
Deflagration point	287^0C
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.

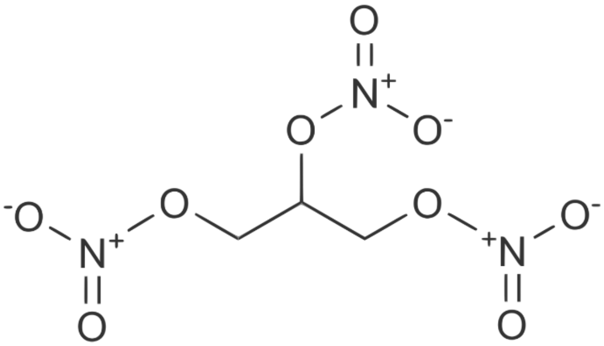

Figure-9- Chemical structure of NG

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	13.2^0C
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, pages7-8).

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% 0, 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 of plastic bubble. Because the emulsions are neither gelled nor cross linked, their storage time is limited. Their relative high detonation velocity compensate 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+$Ba(NO_3)_2$ + 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+DimethylFormanide

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 – Nitroguandine+ NC + nitroglycerine and a stabilizer (DPA).

1.10.3.3Pyrotechnic 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.
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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 thosewhich 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*2nd edition 2nd priniting 1977 charles Thomas Publisher, Spring field Illinois USA.
2. BroadieT *Bombs and Bombings* 3rd Printing Publisher Charles C Thomas Spring field 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)

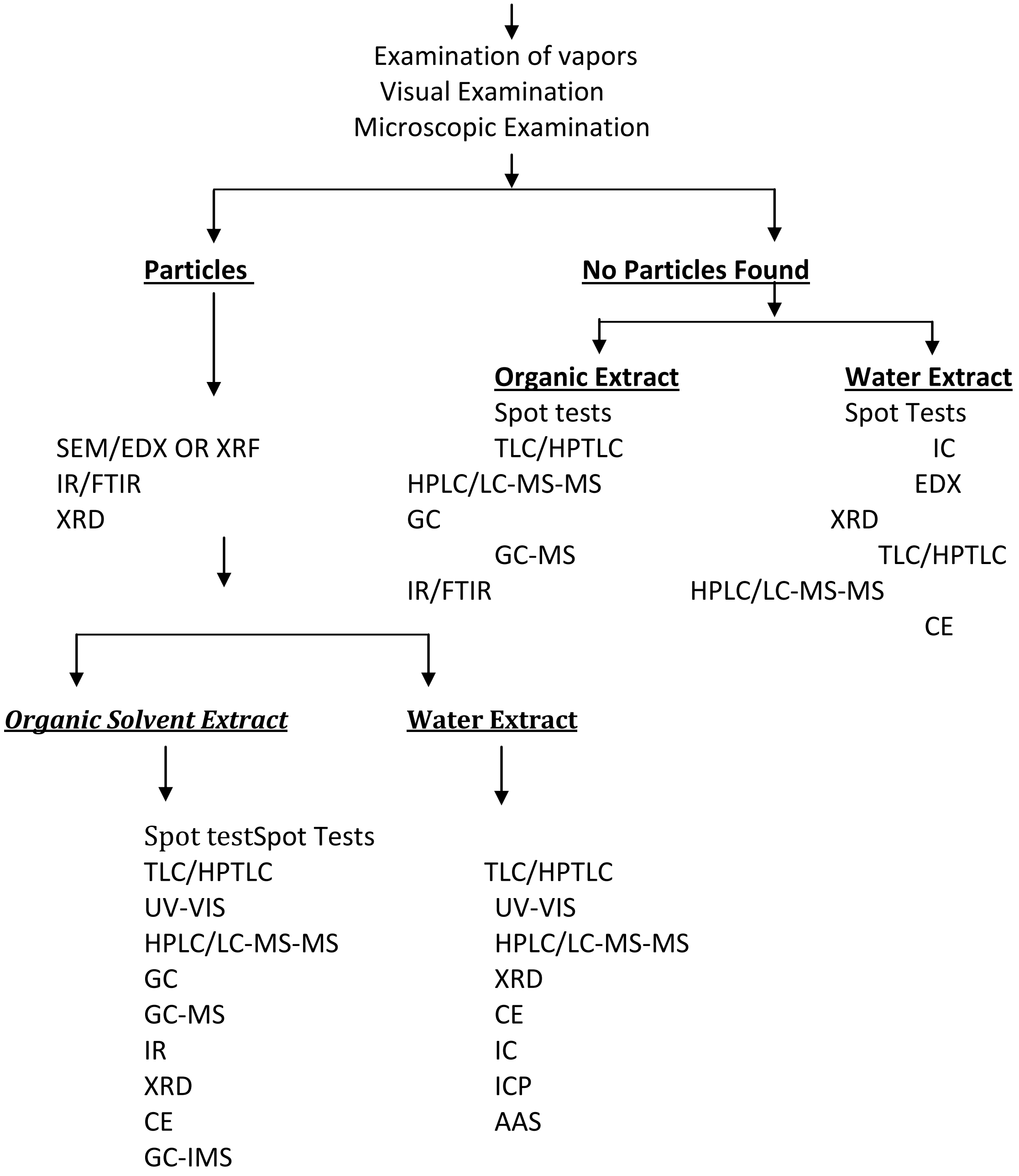
3.5.2.4 Pyridine Extract:

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,*5th 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 cationsin pre blast and post blast inorganic explosives/ pyrotechnic compositions.

4.4 Chemical tests for anions

4.4.1 Test for Chloride (Cl^-)

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 $AgNO_3$ 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_4OH and insoluble in HNO_3.
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^-
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_2SO_4
- 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_2SO_4. Heat the mixture gently. A red vapour of Chromyl chloride evolves which turns diphenylcarbizide spot on filter paper violet. (ref1page 326).

4.4.2 Test for Sulphate: ($SO_4{}^{2-}$).

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 $100^0{}_C$ for 5 minutes, then add 2-3 drops of reagent B to it. A white crystalline precipitate indicates the presence of $SO_{4.}{}^{2-}$

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:

ReagentA;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 colouredppt becomes clearly visible. (Ref1 page 348).

4.4.3: Test for Nitrite (NO_2^-)

Requirement:-

i) Sulphanilic acid
ii) α -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:-α-naphthylaminesolution.Take 0.3 g of α -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. Place1 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 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 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 (NO_3^-)

Requirements:

i)	Sulphanilic acid	ii)	α-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: α – naphthylamine Boil 0.3 g of α-naphthylamine with 70ml of distilled water, filter or decant and mix with 30ml of glacial acetic acid.

Procedur*e*:-

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_2SO_4

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 Thiocynate (SCN^-)

Requirements:

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

Preparation of Reagents

Reagent A: 0.5M Co $(NO_3)_2$. (Dissolve 146 g of cobalt nitrate in water and dilute to 1lit.)

Reagent B: 0.25 M $CuSO_4$. (Dissolve 62.42 g of $CuSO_4.5\,H_2O$ 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 : (ClO_3^-)

Requirements:

i)	Concentrated Sulphuric acid	ii)	Aniline sulphate
iii)	Manganoussulphate	iv)	Phosphoric acid
v)	Diphenylcarbazide	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 diphenylcarbazide 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.

4.4.9Test for Sulphite (SO_3^{2-})

Requirements:

i) $ZnSO_4$
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 of 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:

Preperation 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 (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. 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. 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 HNO_3–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

4.4.12.1Nitrate 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.

4.4.12.2Nitrate 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 .

4.4.12.3Chloride ,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

<table>
<tr><th><u>Residue</u></th><th colspan="2"><u>Filtrate</u></th></tr>
<tr><td rowspan="3">CdS and excess of $CdCO_3$. 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_2S with lead acetate paper (Sulphide</td><td colspan="2">Add strontium nitrate solution in slight excess. Allow to stand overnight and filter.</td></tr>
<tr><td><u>Residue</u></td><td><u>Filtrate</u></td></tr>
<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_2 is evolved and sulphur is slowly precipitated</td></tr>
</table>

4.5 Chemical tests for Cations

4.5.1 Test for Sodium ion (Na^+)

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.

<u>Procedure :</u>

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 pptappears.In case of smaller amounts of sodium, place the porcelain plate under UV light. A bright fluorescenceindicate 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) Sodiium 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 80^0C. 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 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 (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^+	Golden Yellow
K^+	Violet (lilac), Crimson through cobalt blue glass
Ca^{2+}	Brick-red
Ba^{2+}	Yellowish-green
Sr^{2+}	Crimson

3.5.7 Test for ammonium ion (NH_4^+)

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 Quinializarin 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 aceticacid until the blue colour just disappears and the unmoistened paper regains the brown colour of free quanalizarin. 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-triphenlytetrazolium 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 position 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. Diisolve 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_2SO_4)

Eluent : 1.7 Mmol/L $NaHCO_3$/1.8Mmol/L Na_2CO_3

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^-, NO_2^-, ClO_3^-, NO_3^-, SO_4^-, SCN^-, ClO_4^-, HCO_3^-**

(ref 4, page 135)

4.5.17High Performance Liquid Chromatography for Sugars

Conditions:

Column: LC-NH2, 250mm x 4.6mm(Id)
Mobile phase: Acetonitrile: water (80:20)
Flow rate: 2 ml/ min
For fructose, glucose and sucrose.
Detector: Refractive Index (RI)

4.5.18 EDXRFdataof 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 verses 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 florescent 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-16ENERGY TABLE OF INORGANIC ELEMENT OF EXPLOSIVES

ELEMENT	**Kα**	**KB1**	**Kab**	**Lα1**	**LB1**	**LB2**	**Lγ1**	**Llab**	**Lllab**	**Llllb**	**Mα1**
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 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) Medium (Mixtures)	mg
Fourier Transform- Infrared (FTIR)	Infrared active organics and inorganics	High (pure compounds) Medium(Mixtures)	mg µg (ATR)
Raman	Raman active organics and inorganics	High(pure compounds) Medium(mixtures)	µg 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

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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
MercuryII	-	-	0.14	Orange	-	Grey Brown
Lead II	0.37	0.36	0.00	Yellow	-	Brown Grey
Styphnate	0.73	0.71	0.89	Yellow without visualization		
Fulminate	0.64	0.63	0.95	-	-	Brown Grey
Azide	-	0.49	0.62	-	Red	-

References :-

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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 forIdentification 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) α-Naphthlyamine
v) BrucineSulphate
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: α -Naphthaylamine in acetic acid, Dissolve 1 g of α -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^{0}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^{0}C for 10 minutes in an air oven. Cool and spray with Griess reagent (0.25% sulphanilic acid and 0.1%. α-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**	**Trichloro ethylene : Acetone (4:1)**	**Chloro-form: Acetone (1:1)**	**Toluene : Cyclo-hexane (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)

6.6Identification of Minor components of propellantsbyThin 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) I/I
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^{0}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 solutionof 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 : μBondapak CN, 10μ 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, reactionchamber 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 μm 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 cm^{-1} for specific functional groups. Below 1300 cm^{-1} fingerprint region

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 cm^{-1} and 1590 – 1510 cm $^{-1}$

Nitrate esters – NO2 stretching vibrations 1285 – 1270 cm^{-1} and 1660 – 1640 cm^{-1}

Two stretching vibrations in Nitra amines i.e., 1310 – 1270 cm^{-1} and 1590 – 1530 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 0.1ug/ml each and filtered.
3. A solution of Test Sample of High Explosives acetate adducts is prepared in Acetonitrile of concentration of about 0.1ug/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 (150mmx2.1mmx5µ) with C18 guard column	Ion spray voltage	-4500V
Mobile Phase	Acetonitrile: water (60:40: v/v).	Heater temperature	250^{0}C
Flow Rate	200µl/min	Curtain gas	N$_2$
Injection volume	20µl	Curtain gas pressure	25 psi
Run time	8 minutes	Collision activated dissociation (CAD) gas	N$_2$
		Collision activated dissociation gas pressure	6 psi.

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

Mass Parameters	Explosive compound				
	TNT $[M-H]^-$	Tetryl $[M-H]^-$	RDX $[M+CH_3COO]^-$	HMX $[M+CH_3COO]^-$	PETN $[M+CH_3COO]^-$
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

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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.

7.4General

The explosive experts are required to provide in Test Reports thedetails of explosivesor otherwise based on the analysis result. For bulk materials which are suspected to be explosives the analysis and interpretationsare 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 ofcustody 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, un ambiguous 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)

7.5Interpretation 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 expectedtrace 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**	NO_3^-, NO_2^-	NH_4^+	**Hydrocarbons**
Black powder	**Potassium nitrate, #Sulphur,Charcoal**	NO_3^-, NO_2^-,SO_4^{2-}, $S_2O_3^{2-}$	K^+	**Sulphur**
Throw down (Jute Bomb)	**Potassium #Chlorate Arsenic Sulphide**	ClO_3^-,Cl^- S^{2-}, SO_4^{2-}	K^+ As^{3+}	
Cracker* (potassium perchlorate+ Aluminium Powder)	**Potassium perchlorate, Aluminium powder**	ClO_4^-,Cl^-	K^+ Al^{3+}	**Al**
Chlorate/Sugar	**Potassium # Chlorate/Sugar**	ClO_3^-,Cl^-	K^+	
Perchlorate/Sugar	**Potassium Perchlorate*/Sugar**	ClO_4^-, Cl^-	K^+	
Chlorate/Sulphur/ Aluminium	**Potassium Chlorate#/Sulphur/ Aluminium**	ClO_3^-,Cl^- SO_4^{2-}, $S_2O_3^{2-}$	K^+ Al^{3+}	**Al**
Slurry and Emulsion	**Ammonium Nitrate Mono Methyl Amine Nitrate/jelling agent,/Higher Hydrocarbon**	NO_3^-,	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.

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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

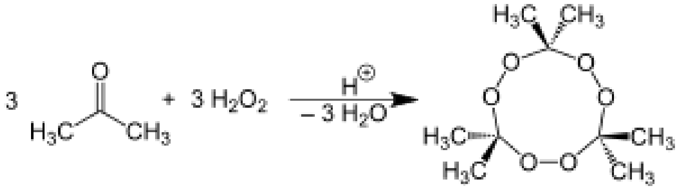
8.3.1 PEROXIDE EXPLOSIVES

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 NO2 groups, the O–O bond is a source of oxygen available for potentially rapid self-oxidation and explosion. Although neat hydrogen peroxide (H2O2) 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.

8.3.1.1TATP-Triacetonetriperoxide (TATP)

Triacetonetriperoxide (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).



Acetone TATP

Table-25- Properties of TATP

Colour	White crystalline
Solubility	Insoluble in water,
Empirical formula	$C_9H_{18}O_6$
Molecular weight	222.2
Melting Point	98^0C
VOD	5300 m/s at maximum density (1.18 g/cm^3),

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)
6 Mo_2O_5 OH + Me_2COO $_3$ 12 MoO_3 + 3 H_2O + 3 Me_2CO
TATP

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_2SO_4
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 Ă, 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-

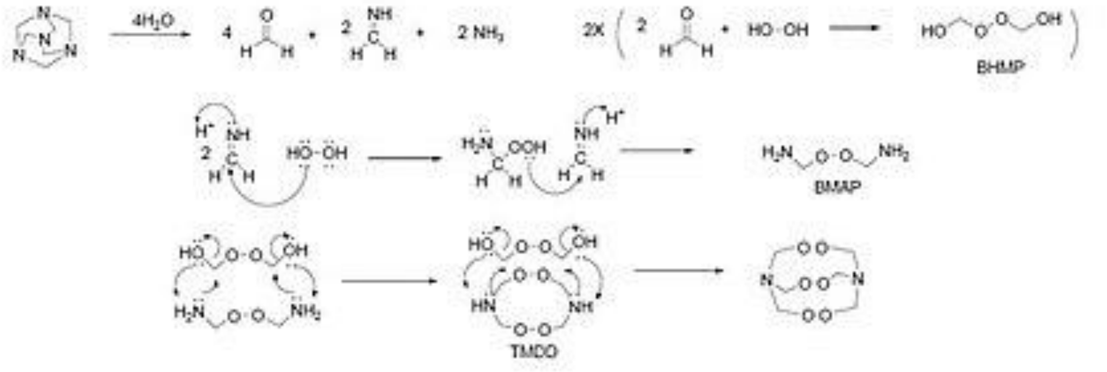
Immediately after the separation of analytes the latter irradiated with UV light of 254 nm. A 25 m knitted Teflon tubing (inner diameter 0.3 mm) wrapped around the UV lamp and used as reaction loop. After decomposition of the analytes to hydrogen peroxide, two HPLC pumps are introduced to deliver the 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 floe 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 ^{0}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: 0.2µL
Capillary column: TG-5MS (15mx0.25mmx0.25µm)
Oven: 50^{0}C hold for 3 min.
10^{0}C/min to 200^{0}C and hold for 2.0 min
Total run time: 20 min
Detector: ISQLTMS
Ionisation Source: EI @70eV
Transfer Line Temperature: 200^{0}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)

Hexamethylenetriperoxidediamine 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 40^0C HMTD begins to break up in to volatile pungent compounds.(ref 1)



HMTD

Table-26- Properties of HMTD

Colour	White crystalline
Solubility	Insoluble in water,
Empirical formula	$C_6H_{12}N_2O_6$
Molecular weight	208.2
Melting Point	Decomposes at 75^0C
VOD	5100 m/s at around 1.1 g/cm^3

8.3.1.2.1 Colorimetric detection of HMTD (ref 1)

To water solution , 1-2 drops of 1% TSS (N,N-diethyl-p-phenylendiaminesulphate) 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 Ǻ, 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 floe 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 ^{0}C. The excitation wavelength of the fluorescence detector set to 324 nm and the emission wavelength 405 nm.

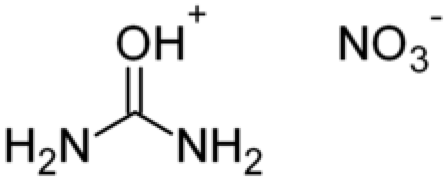
8.3.1.2.3 GC-MS

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 (15mx0.25mmx0.25µm)
Oven: 50^{0}C hold for 3 min.
    10^{0}C/min to 200^{0}C and hold for 2.0 min
Total run time: 20 min
Detector: ISQLTMS
Ionisation Source: EI @70eV
Transfer Line Temperature: 200^{0}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 $(HMTD\text{-}O_2)^+$

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)



Urea Nitrate

Table-27- Properties of Urea Nitrate

Colour	White crystalline
Solubility	Soluble in water and alcohol
Empirical formula	$CH_5N_3O_4$
Molecular weight	123.1
Melting Point	163^0C
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 _0.1 mg/cm2. 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 shorterthan p-DMAC, also reacts with urea nitrate under similar conditions, to producea 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, ureanitrate does, as it provides the necessary acidity for the reaction to occurp-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^{-1}
Resolution: 4.0 cm^{-1}
Mode: Attenuated Total Reflectance (ATR) for solids
Scans:32
Principal wave numbers: 3402 cm^{-1} (NH_2 asymmetric stretch),3354 cm^{-1} (NH stretch), 3198 cm^{-1} (NH_2 symmetric stretch), 1704 cm^{-1} (CO stretch), 1568 cm^{-1} (N-H), 1426 cm^{-1} (C-N), 1298 cm^{-1} (NO_3^-)

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: 180^0C
Column Temperature: programmed from 50^0C to 180^0C at a rate of 15^0C/min,10 min at 180^0C, 180-300^0C at a rate of 25^0C/min and 5 min at 300^0C.
Transfer Line was held at 230^0C.
Ion Source Temperature: 180^0C
Quadrupole Temperature: 120^0C
Scan range: 29-350 Da
Scan Rate : 2.29 scans/s
Electron energy :70eV
Carrier gas: Helium
Injection: Split less mode

Preparation of xanthydrolalkylcarbamates (xantylurethanes)

Sample and xanthydrol 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 NH_4NO_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

$4NH_4NO_3+6HCHO \rightleftharpoons (CH_2)_5\ N_4+4HNO_3+6\ H_2O$

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

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

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

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

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.

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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+ H_2So_{4+} 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_f / λ_{max} 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	$AgNO_3$ Test	White curdy ppt sol in NH4OH								
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	$BaCl_2$ Test	White crystalline ppt								
Sulphide Na OH extract	Sodium nitroprusside Test	Violet colour								
Sulphur Pyridine Extract	Pyridine + NaOH	Blue to Green for low conc. 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 (NaOH extract)	Aluminon reagent	Brown flakes or ppt								
	Alizarine – S Test	Red ppt								

Arsenic (As $^{+3}$) (NaOH extract)	Gutzeit’s Test	Yellow to Brown spot								
	Reinsch’s Test	White sublimate								
Antimony (Sb^{+3})										
	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

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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 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. 2. Sh. B Shanmukham, Director, FSL, Puducherry. 3. Dr. Harsh Sharma, Director, FSL, Sagar (MP)
Explosives	Dr. Sukhminder Kaur, Coordinator CFSL, Pune	1. One officer from FSL, Delhi 2. One officer from FSL, Maharashtra
Toxicology	Dr. Vimukti Chauhan, SSO, CFSL, Chandigarh	1. Dr. K. V. Kulkarni, Director, DFSL, Maharashtra 2. One officer from FSL, Karnataka.
Ballistics	Sh. S. S. Baisoya, CFSL Chandigarh	1. Dr. D. K. Kaushal, Director, FSL, Haryana 2. Sh. N. P. Waghmare, Director, FSL, Goa 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) 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.