

Experiment no. 1

Aim: Analysis of low explosives/ Identification of inorganic explosives by chemical method (Anions)

Requirements:

- a) Test Sample: Water samples spiked or if explosives samples available (Sodium chloride, calcium sulphate, sodium nitrate, potassium nitrite, sodium thiosulphate, sodium phosphate)
- b) Materials: Beaker(s), test tube(s), test tube stand, test tube holder, dropper, pipette, glass rod, measuring cylinder, spatula, spot plate, marker, volumetric flask.
- c) Instruments: Hot plate, water bath, weighing balance.
- d) Chemicals: alpha-naphthylamine, ammonium molybdate, barium chloride, benzidine, concentrated and dilute hydrochloric acid, concentrated sulphuric acid, ethylene diamine, ferrous sulphate, glacial acetic acid, hydrogen peroxide, ferric chloride, nitric acid, lead acetate, potassium dichromate, potassium permanganate, silver nitrate, sodium rhodizonate, sulphanilic acid, zinc dust.

Theory:

Low explosives are compounds that deflagrate. Low explosives are mechanical mixtures of individual ingredients. They are used mainly as propellants because they tend to exert a rapid pushing effect. Propellants are mixtures of one or more energetic materials, plasticizers, stabilizers, and inorganic additives. Black powder (gun powder) and smokeless powder are most commonly used low explosives. The composition of black powder are sodium/potassium (rarely ammonium) nitrate, charcoal, sulphur / sulphurless powder. The composition of smokeless powder are single, double, and triple base with nitrocellulose, nitroglycerine and/or nitroguanidine with stabilizer diphenylamine (DPA). The materials used in pyrotechnique includes oxidizing agent such as potassium chlorate, sodium nitrate, etc; fuels viz. sulphur, charcoal, phosphorous, aluminium powder, etc; color producing agents such as salts of different metals like sodium, strontium barium, etc; smoke producing substances like white phosphorous, hexachloroethane, etc; binding agent and stabilizers.

Procedure:**a. Preparation of reagents:**

Sr. No	Reagent	Concentration	Preparation
1	Nitric acid	2 M	1.28 ml concentrated nitric acid made up to the mark with water in 10 ml volumetric flask.
2	Silver nitrate	0.1 M	0.1699 g of AgNO ₃ made up to the mark with water in 10 ml volumetric flask.
3	Lead acetate solution	0.25 M	95 mg lead acetate trihydrate with 100 µl glacial acetic acid solution made up to the mark with water in 10 ml volumetric flask.
4	Barium chloride	0.25 M	61.1 mg barium chloride dihydrate made up to the mark with water in 10 ml volumetric flask.
5	Sodium rhodizonate	0.5 %	0.05 g of sodium rhodizonate made up to the mark with water in 10 ml volumetric flask. Reagent should be freshly prepared.
6	Potassium permanganate	0.02 M	3.16 mg Potassium permanganate made up to the mark with water in 10 ml volumetric flask.
7	Hydrogen peroxide solution	3 %	3 ml commercially available 30% H ₂ O ₂ made up to the mark with water in 10 ml volumetric flask.
8	Sulphanilic acid	1 %	0.1 g of sulphanilic acid made up to the mark with warm 30% acetic acid in 10 ml volumetric flask
9	α-Naphthylamine	0.3 %	0.03 g of α-naphthylamine boiled with 7 ml distilled water, filter/ decant and mix with 3 ml of glacial acetic acid.
10	Ferric Chloride	1 M	0.16 g of ferric chloride made up to the mark with water in 10 ml volumetric flask
11	Saturated solution of sodium acetate	--	Excess amount of sodium acetate added to distilled water (till the point of insolubility)
12	Ammonium molybdate	0.2 M	0.5 g Ammonium molybdate + 10 ml water + 3 ml concentrated nitric acid
13	Benzidine	1 m	1.625 g Benzidine with 2.5 ml glacial acetic acid made up to the mark with water in 10 ml volumetric flask.

b. Chemical test:

Sr. No	Test for Anion	Procedure
1	Chloride (Cl ⁻)	Step 1: 1ml water extract + 2-3 drops reagent 1 + 2-3 drops reagent 2 Step 2: 1ml extract + 2-3 drops reagent 3
2A	Sulphate (SO ₄ ²⁻)	Step 1 1 ml of extract + 2-3 drops reagent dilute hydrochloric acid kept in a water bath at 100°C for 5 minutes, + 2-3 drops reagent 4 Step 2 1 drop of reagent 4 on filter paper, + 1 drop of reagent 5 ---- the reddish-brown spot + 1 drop acidified extract
2B	Sulphate (SO ₄ ²⁻)	3 drops extract + 2 drops reagent 6 + 1 drop reagent 5. ---pink precipitate + few drops reagent 7 and shake well.
3	Nitrite (NO ₂ ⁻) (Griess test).	1 drop neutral/ acid extract on a spot plate. + 1 drop reagent 8 + 1 drop reagent 9
4A	Nitrate (NO ₃ ⁻)	1 drop neutral/ acid extract on a spot plate. + 1 drop reagent 8 + 1 drop reagent 9 + zinc dust
4B	Nitrate (NO ₃ ⁻)	a crystal of ferrous sulphate on a spot plate + a drop of extract + a drop of concentrated sulphuric acid to run at the side of the drop.
5	Thiosulphate (S ₂ O ₃ ²⁻)	Step 1 1 drop water extract + 2 drops reagent 10 and mix
6	Phosphate (PO ₄ ³⁻)	1 drop water extract on spot plate + 1 drop reagent 12 Stand time -30 seconds + 1 drop of reagent 13 Stand time -30 seconds + 3 drops reagent 11.

Observation (on blank side):

Sr. No	Test for		Observation	Inference
1	Chloride	Cl ⁻	white precipitate	Indicates the presence of chloride ion, [soluble in ammonium hydroxide and insoluble in nitric acid]
2a	Sulphate Step 1	SO ₄ ²⁻	white crystalline precipitate	Indicates the presence of sulphate
2a	Step 2		red color spot disappears	Indicates the presence of sulphate
2b		SO ₄ ²⁻	pink coloured precipitate becomes clearly visible	Indicates the presence of sulphate
3	Nitrite	NO ²⁻	pink to red colour	Indicates the presence of nitrite
4A	Nitrate	NO ³⁻	pink to red colour	Indicating presence of nitrate
4B		NO ³⁻	brown ring forms round the crystal of ferrous sulphate	Indicating presence of nitrate
5	Thiosulphate	S ₂ O ₃ ²⁻	crystalline violet visible	Indicates the presence of Thiosulphate
6	Phosphate	PO ₄ ³⁻	blue gray colour	Indicates the presence of phosphate

Conclusion:

Thus, based on the inference from chemical test carried out, chloride, sulphate, nitrate, nitrite, thiosulphate, and phosphate identified in the extract of test sample.

Experiment no. 2

Aim: Analysis of low explosives/ Identification of inorganic explosives by chemical method (Cations)

Requirements:

- a) Test Samples: Water samples spiked (with lead acetate trihydrate, calcium carbonate, ammonium chloride, mercuric chloride, bismuth nitrate, arsenic pentahydrate, copper sulphate, antimony trioxide) or if explosives samples available.
- b) Materials: Beaker(s), test tube(s), test tube stand, test tube holder, dropper, pipette, glass rod, measuring cylinder, spatula, spot plate, filter paper, marker, volumetric flask, funnel, copper strip, iron nail.
- c) Instruments: Hot plate, weighing balance.
- d) Chemicals: concentrated hydrochloric acid, sodium rhodizonate, sodium hydroxide, potassium iodide, mercuric chloride, potassium hydroxide, potassium dichromate

Theory:

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. 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.

Colour testing is a presumptive technique that indicates the presence or absence of particular chemical using chemical methods. Lead on reaction with potassium iodide and potassium dichromate forms yellow precipitate of lead iodide and lead dichromate that settles down. Calcium on reaction with sodium rhodizonate solution in presence of sodium hydroxide forms purple precipitate of calcium rhodizonate that settles down. Ammonium on reaction with Nessler's reagent forms a brown precipitate of iodide of Millon's base that settles down. Reactivity series of metals is an empirical and analytical progression of a series arranged from highest to lowest, i.e. they displace the lower placed metal in the order and form the a new compound.

Procedure

a. Preparation of reagents:

Sr. No	Reagent	Concentration	Preparation
1	Sodium rhodizonate	0.2 %	0.02 g sodium rhodizonate made up to the mark with water in 10 ml volumetric flask.
2	Sodium hydroxide	0.5 N	0.2 g sodium hydroxide made up to the mark with water in 10 ml volumetric flask.
3	Nessler's reagent		A: 1 g potassium iodide + 1ml distilled water (1:1) B: 0.6 g mercuric chloride + 10 ml distilled water C: 4.5 g potassium hydroxide + 8ml distilled water. Solution B + Solution A drop wise (till a permanent precipitate forms) + Solution C, mix. Made up to 20 ml with water. Left overnight.

b. Chemical test:

Sr. No	Test for Cation	Procedure
1	Lead (Pb^{2+})	1ml water extract + 2-3 drops concentrated hydrochloric acid --- white precipitate formed --- filter and 2 parts divide Part I: 1 ml filtrate + small amount potassium iodide Part II: 1 ml filtrate + small amount potassium dichromate
2	Calcium (Ca^{2+})	2-3 drops extract on spot plate + 2-3 drops reagent 1 + 2-3 drops reagent 2 -- mix, while blowing briskly with pipette
3	Ammonium (NH_4^+)	2-3 drops extract on spot plate + 2-3 drops Nessler's reagent
4	Mercury (Hg^{2+})	12 ml extract in beaker
5	Antimony (Sb^{3+})	+ 3 ml concentrated hydrochloric acid

6	Bismuth (Bi^{2+})	<ul style="list-style-type: none"> - then ceramic granules added to reduce bumping - kept on hot plate (60-70°C) until boiling starts - copper strip (prewashed with hydrochloric acid) placed - iron nail (prewashed with distilled water) placed - wait for some time to check deposition on nail & strip.
7	Arsenic (As^{2+})	
8	Copper (Cu^{2+})	

Observation (on blank side):

Sr. No	Test for	Observation	Inference
1	Lead (Pb^{2+})	yellow precipitate	Indicates the presence of lead ion.
2	Calcium (Ca^{2+})	violet precipitate	Indicates the presence of calcium ion.
3	Ammonium (NH_4^+)	orange brown precipitate	Indicates the presence of ammonium ion.
4	Mercury (Hg^{2+})	silver deposition	Indicates the presence of mercury ion
5	Antimony (Sb^{3+})	purplish-black deposition	Indicates the presence of antimony ion
6	Bismuth (Bi^{2+})	jet black deposition	Indicating presence of bismuth ion
7	Arsenic (As^{2+})	dull black deposition	Indicating presence of arsenic ion
8	Copper (Cu^{2+})	copper color on nail	Indicates the presence of copper ion

Conclusion:

Thus, based on the inference from chemical test carried out, it is concluded that cations of lead, calcium, ammonium, mercury, bismuth, arsenic, antimony and copper identified in the extract of test sample.