

SCHEME FOR IDENTIFICATION OF PVC

PRELIMINARY TESTS

1. Appearance test:

Visual appearance is noted	Granules or Powders	May be thermoplastic
----------------------------	---------------------	----------------------

2. Bounce test:

A small piece of the sample is dropped	It doesn't bounce	may be plastic
--	-------------------	----------------

3. Effect of heat:

A few granules or a bit of powder is heated in a spatula	It softens	thermoplastic material.
--	------------	-------------------------

4. Beilstein test:

A bright copper wire (about 5cm long with one end embedded in a cork) is cleaned by heating to redness in a colourless bunsen flame. The sample under test is touched by the hot wire and then placed in the flame.	presence of green flame.	presence of halogens.
---	--------------------------	-----------------------

5. cutting test:

A piece of sample is cut with a sharp knife	Easily pared off	thermo plastics except PS & PMMA.
---	------------------	-----------------------------------

6. Drop test:

A piece of sample is dropped in water. If the sample sinks drop on a hard surface.	Sinks dull sound.	May be PC, ABS, PS, Nylon, PVC, PET etc. May be, Nylon, PVC, PET etc.
---	--------------------------	--

7. Heating test:

A small piece of the sample is taken in a clear spatula and heated without ignition until it begins to fume. The sample is then removed, odour of the fume and its nature to wet blue litmus paper is noted. Then it is heated to burning or ignited and the nature of the flame is also noted.	The material burns but extinguishes itself on removal from the flame yellow with green base, acidic smell, acidic fumes.	May be PVC
--	---	------------

ELEMENTAL ANALYSIS (LASSAIGNE'S TEST)

Preparation of sodium fusion extract

A small piece of clear sodium (about 0.02g) is placed in an ignition tube and a small quantity of the sample (about 0.1g) to be tested is added. The tube is gently heated until the reaction subsides. Then heated strongly till it becomes red hot. Then the tube is plunged into distilled water taken in a clear china dish. It is stirred well with a glass rod, the mixture is heated to boiling and filtered. The filtrate is divided into four equal portions and the following tests are carried out.

1. Test for Nitrogen

The test portion is boiled with 3 drops of freshly prepared aqueous ferric sulphate solution (approx 5%) and cooled. After acidification with 5N H_2SO_4 , a drop of 0.5N ferric chloride solution is added.

No, characteristics
observation.

Absence of
Nitrogen.

2. Test for sulphur.

3 drops of freshly prepared sodium

nitro prusside is added to the test portion.

No characteristics observation

Absence of sulphur.

3. Test for chlorine and bromine:

The test portion is acidified with 5N nitric acid and 0.1N silver nitrate solution is added.

A white precipitate soluble in ammonia
yellow precipitate insoluble in ammonia

Presence of chlorine.
presence of bromine.

4. Test for fluorine

An aqueous solution of zirconium nitrate (0.1%) and alizarin red (0.1%) is prepared. Filter paper is immersed in the solution and then allowed to dry. Then it is moistened with acetic acid solution (5%). The test portion is neutralised with 5N HCl and a drop is placed on the moistened filter paper.

NO characteristic
observation

Absence of fluorine.

FINAL IDENTIFICATION

Group-III (plastics containing chlorine).

Test for Poly Vinyl Chloride (PVC)

A sample (about 0.05gm) is dissolved in pyridine (5ml) by heating in a water bath. A solution (0.5ml) of NaOH in methanol (2%) is added to the hot polymer solution.

A brown colouration is produced and a brown precipitate results.

Presence of PVC confirmed.

RESULT

PVC is identified

SCHEME FOR IDENTIFICATION OF NITRILE

RUBBER

PRELIMINARY TESTS OR INITIAL TESTS

I. EFFECT OF HEAT

A small piece of sample about 0.1gm is taken in a spatula and warmed.

It softens

Presence of unvulcanised rubber.

2. Beilstein test

A bright copper wire (about 5cm long with one end embedded in a cork) is cleaned by heating to redness in a colourless bunsen flame. The sample under test is touched by the hot wire and then placed in the flame.

presence of green flame

presence of halogens.

3. Specific gravity test

About 0.02g of the sample is properly wetted and pushed below the surface of water in a test tube with a glass rod and released.

The sample floats on water

specific gravity less than one.
presence of NR, NBR, BR, EPR, SBR, IIR etc.

4. Bounce test

small piece of the sample is dropped

It bounce

presence of all rubber except butyl rubber

5. Odour test

odour of the sample is noted.

Fruity smell

presence of nitrile rubber.

6. Heating test

A small piece of the sample is taken in a clear spatula and heated without ignition until it begins to fume. The sample is then removed, colour of the fume and its nature to wet blue litmus paper is noted. Then it is heated to burning or ignited and the nature of the flame is also noted.

The material burns and continues to burn on removal from the flame.

yellow smoky flame,
sickly sweet smell

Presence of
nitrolic rubber

ELEMENTAL ANALYSIS (LASSAIGNE'S TEST)

Preparation of sodium fusion extract

A small piece of clear sodium (about 0.02g) is placed in an ignition tube and a small quantity of the sample (about 0.1g) to be tested is added. The tube is gently heated until the reaction subsides. Then heated strongly till it becomes red hot. Then the tube is plunged into distilled water taken in a clear china dish. It is stirred well with a glass rod, the mixture is heated to boiling and filtered. The filtrate is divided into four equal portions and the following tests are carried out.

1) Test for nitrogen.

The test portion is boiled with 3 drops of freshly prepared aqueous ferric sulphate solution (approx 5%) and cooled. After acidification with 5N H_2SO_4 , a drop of 0.5N ferric chloride solution is added.

A prussian blue precipitate of ferric ferro cyanide is formed.

Presence of nitrogen.

2.) Test for sulphur

3 drops of freshly prepared sodium nitro-prusside is added to the test portion.

No characteristics observation

Absence of sulphur

3) Test for chlorine and Bromine.

The test portion is acidified with 5N nitric acid and 0.1N silver nitrate solution is added.

No characteristics observation

Absence of chlorine & bromine.

4) Test for Fluorine.

An aqueous solution of zirconium nitrate (0.1%) and alizarin red (0.1%) is prepared. Filter paper is immersed

No characteristics observation

Absence of Fluorine.

in the solution and then allowed to dry. Then it is moistened with acetic acid solution (50%). The test portion is neutralised with 5N HCl and a drop is placed on the moistened filter paper.

FINAL IDENTIFICATION

GROUP I (Rubbers containing Nitrogen)

1. Test for polyurethane Rubber

conc. sulphuric acid (1ml) is carefully added to water (4ml) in a test tube and a small piece of rubber, about 0.05g is placed in the mixture and boiled for a few minutes.

The sample did not disintegrate

Absence of polyurethane rubber.

2. Test for vinyl pyridine Rubber

A sample (about 0.1g) is fused with KOH (about 0.2g) in a heat resistant test tube. The mixture is cooled

and then shaken with water (2ml). The aqueous layer is poured off and just acidified with 5N HCl and then an excess of acd (2ml) is added. 5 drops of Mayer's reagent is also added.

No characteristics
observation

Absence of
vinyl pyridine
Rubber.

[Mayer's reagent is prepared by dissolving mercuric chloride (3g) and potassium iodide (10g) in 200 mL water.]

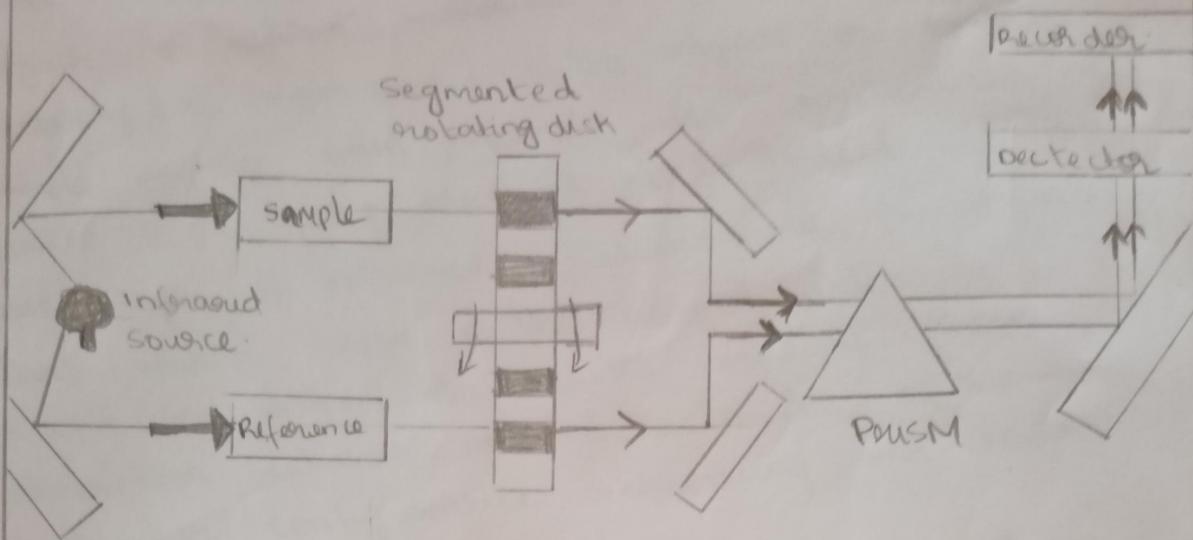
3. Test for Nitrile Rubber

Negative results from tests 1 & 2 indicate the presence of Nitrile Rubber.

RESULT

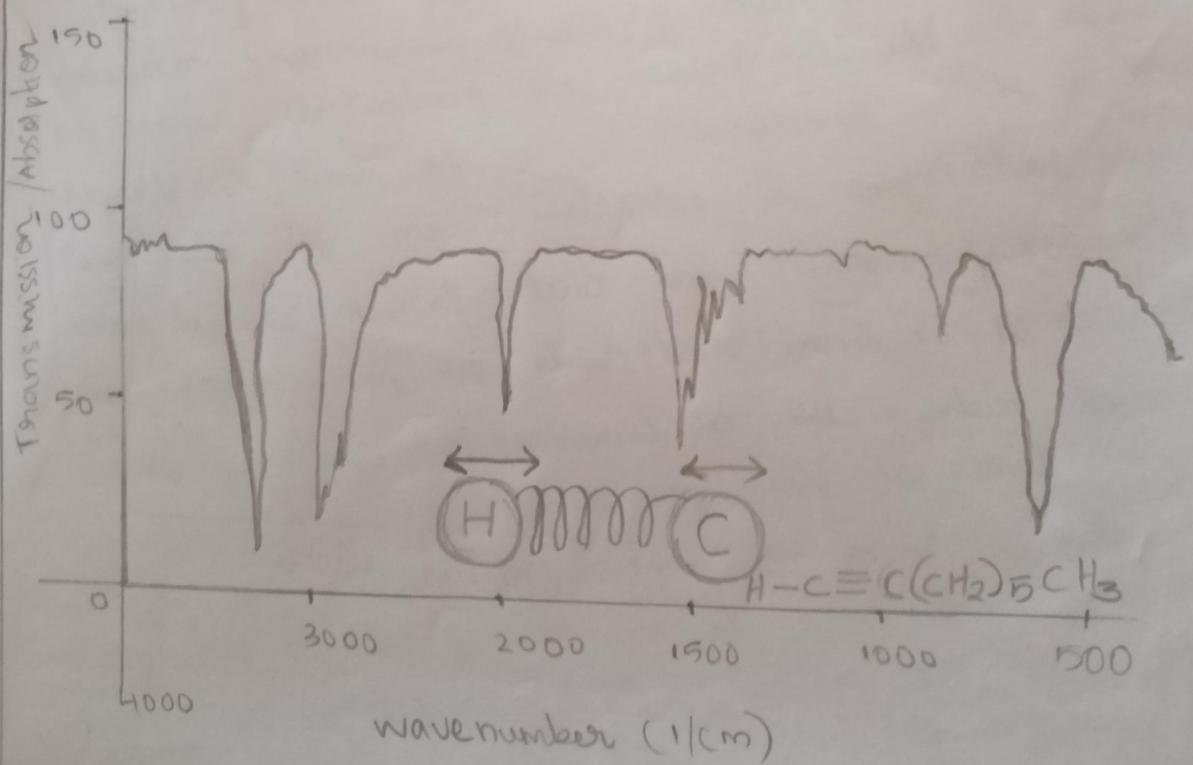
Nitrile rubber is identified.

INFRARED (IR) SPECTROSCOPY OF POLYMER



The infrared spectrum of a polymer or rubber is a profile of its absorption characteristics plotted against infrared wave number (or frequency).

INFRARED SPECTRUM OF 1-Octyne



It is generated when infrared radiation interacts with the molecular moieties that constitute the polymer or rubber material. It is a distinctive property (a "finger print") of the polymer or rubber sample in the form, manner and environment in which it is being examined. It is based on the absorption of infrared radiation at frequencies, that match those of the normal modes of vibration within the macro molecule. These absorption features are characteristic of the molecular configuration, sequencing and conformation and state of order.

Absorptions by internal vibrations predominate in the mid-infrared region (ca 4000-400 cm^{-1}); they involve a few selected atoms in a molecular (functional) grouping, which is a sub-set of those consulting the macro molecule external vibrations, such as lattice vibrations, which involve segments of macro molecules in crystalline regions tend to occur at low wave numbers ($< 400 \text{ cm}^{-1}$). The intensity of an absorption band is, to a first approximation, proportional to the number density of vibrating species giving rise to that band. The intensity of an absorption band is related to the dipole moment change associated with the molecular vibration; if it is large then the band intensity will be high. Infrared spectroscopy is

highly specific. For some quantitative analyses, it can be very precise and sensitive ($<0.1\%$). It is complemented with Raman spectroscopy for which the selection rules are different and relate band intensity to the change of polarizability occurring during a molecular vibration.

APPLICATIONS | USES

Infrared spectroscopy can be used for identify and analyse the molecular structure, composition, order and morphology.

Both qualitatively and quantitatively of polymers, copolymers and rubbers and their products.

REFERENCE

<https://onlinelibrary.wiley.com/abs>

Text Polymer science VR Grawariker.