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# Analysis: Inorganic unknowns

## Flame test for metal cations

|  |  |
| --- | --- |
| **Metal ion** | **Color of flame when burnt** |
| Li+ | **Red** |
| Na+ | **Yellow** |
| K+ | **Lilac** |
| Mg2+ | **No color** |
| Ca2+ | **(Yellow / Brick) Red** |
| Sr2+ | **(Crimson) Red** |
| Ba2+ | **Green** |

Apparatus: Pt wire

Procedure: Clean with HCl, dip wire to solution & place in Bunsen flame

Observation: Colors as followed

Note: A paste can also be formed with strong acid (HCl) and solids

Reason: e- dropping from higher energy level, releasing energy

## Basic tests

No idea what will the observation be for the question’s test? Answer “no reaction”!

**H2** Lighted splint burns with squeaky pop sound

|  |  |  |
| --- | --- | --- |
| **Species** | **Observation** | **Hazard & Minimizing risk** |
| NO2(g) | Brown gas | - |
| Cl2(g) | White fumes | Toxic, fume cupboard |
| Ba2+ | - | Toxic, fume cupboard |
| HCl(l/g) | Alkaline **B** 🡪 **R** | Toxic, fume cupboard |
| NH3 | Pungent smell | - |
| ROH | - | Flammable, no flames |
| H2O | Colorless vapor, condenses to liquid | |
| CO2 | Acidic **R** 🡪 **B** | |
| MCO3 | White solid | |

**O2** Relights a glowing splint

**CO2**Lime water 🡪 Milky / cloudy solution

**H2O** CuSO4 🡪 Turns **B**

### Ammonia NH3

Reagent: HCl(g)

Observation: **White** fumes (NH4Cl ammonium chloride)

### Ammonium NH4+

Reagent: Warm aqueous NaOH

Observation: Gas given off, litmus paper **R** 🡪 **B** (NH3 alkaline)

### Sulfate SO42- / Barium Ba2+

Reagent: Acidified Ba2+

Observation: **White** pp (BaSO4 insoluble in dilute acids)

### Carbonates CO32-

Reagent: Ba2+

Observation: **White** pp (BaCO3 soluble in dilute acids)

Reagent: H+ from HCl / HNO3

Observation: Bubbles (CO2 milky lime water)

Reason:

### Iodine I2

Reagent: Starch indicator

Observation: **Black**

Note: For iodine clock titrations, indicator turns colorless as I2 🡪 2I-

## Group 7 anions

### Method 1: Silver nitrate AgNO3

Procedure: Add dilute nitric acid HNO3 first, then the reagent

Reagent: Silver nitrate AgNO3

Observation: {Cl-, Br-, I-} : {**White**, **Cream**, **Yellow**} pp

Note: **Silver nitrate** is acidified to remove anions that would interfere with the test. Precipitate is a **Silver Halide** (HX)

### Method 2: Conc. H2SO4

Reagent: Conc. H2SO4

Observation: {Cl-, Br-, I-} : {**Misty** HCl, **Brown** Br2, **Purple** I2} fumes

# Analysis: Organic unknowns

### C=C group

Reagent: Bromine water Br2

Observation: Decolorized (from **Brown**)

Reason: C2H4 + X2 → C2H4X2 (C=C removed)

Note: **Br2 only reacts with alkanes or alkenes**, if reacted with other solution 🡪 no change

Reagent: Acidified Potassium Manganate K2MnO4

Observation: Decolorized

Reason: (C=C) removed

Procedure: Burn

Observation: Smoky flame

Reason: Unsaturated

## Alcohols and it’s derivatives

### -OH hydroxyl group (-OOH included)

Reagent: PCl5(s)

Observation: **White** fumes (HCl)

Reagent: Na

Observation: Bubbles (H2)

*These tests can’t be conducted in the presence of H2O as reagents react with H2O instead*

### -OOH group

Reagent: Alcohol under acidic solution (H2SO4)

Observation: Fruity smell

Reagent: CO32-

Observation: Bubbles (CO2 milky lime water)

### 3° or 1° / 2° Alcohols

Apparatus: Pear shaped flask (reflux setup)

Reagent: Acidified Potassium Dichromate K2Cr2O7

Observation: 3° Remains **orange** 1° / 2° 🡪 **green**

Reason: Only 1° / 2° alcohols can be oxidized

### Aldehydes apart from ketones

Reagent: Fehling’s / Benedict’s reagent

Observation: **B** 🡪 **R**

### CH3C=O group

Reagent: Warm alkaline sol. I2

Observation: **yellow** p.p. (CHI3)

# Essential knowledge

### Decomposition of carbonates

### Ammonium carbonate: (NH4)2CO3 → 2NH3 + H2O + CO2

Metal carbonate: MCO3 → MO + CO2

Metal hydrogen carbonate: 2MHCO3 → M2CO3 + H2O + CO2

Carbonates are white solids. CO2 is acidic.

### Determining empirical formula

1. Find mass of all elements

|  |  |  |
| --- | --- | --- |
| Corrosive | Oxidising | Flammable |
| *Explosive* | *Oxidizing* | *Flammable* |
| Corrosive |  | Toxic |
| *Corrosive* | *Health hazard* | *Toxic* |

1. Find mol of all elements
2. Using simplified mol ratio to determine empirical formula

### Br2 and alkenes

Mol ratio of decolorization of Br2 and alkene = number of C=C bonds in alkene

### Rate of reaction of halogenoalkanes

* Rate is inversely proportional to time
* Reaction speed: I > Br > Cl 3° > 2° > 1°

### Mass spectrometry & Infrared

Mass spectrum is consistent with the compound when there’s a peak at Ar

Mass spectrometer sequence: Vaporization – Ionization – Acceleration – Deflection – Detection

Mass peak =

Structure of the ion causes peaks at different positions, by breaking C-C bonds.

# Common experiments & methods

### Drying agents

Most compounds can be dried with **MgSO4** (anhydrous salt & doesn’t react). Alternatively, use silica gel.

## Find: Enthalpy change of reaction

*Graphical method to find*

*(extrapolation)*

### Key concepts

Latter part of the graph represents the heat transferred to system from surroundings.

If a spirit burner is used, needs to be measured to calculate

### Common questions

* Excess solution used: Ensure all primary reactant used up
* ΔH can’t be measured directly: Hard to measure of solid

Some substance might evaporate

* Cleaning with sandpaper purpose: Remove oxidized layers to ensure purity of metal
* Fast reaction of material: Reduce heat lost
* Glass beaker effect on : Glass worse insulator ( less exothermic
* Lower than accurate value: Heat lost to surroundings
* w/o procedure: Greater mass solute, smaller volume of solvent
* Increase accuracy by procedure: Lid to reduce heat lost

Use more precise thermometer

### Assumptions

* Solution density
* Specific heat capacity is the same as water

## Find: Concentration / purity of solution

Procedure: Standard solution 🡪 titration

### To: Titrate

*Common procedure if asked for it:*

1. Prepare solution of known conc.
2. Titrate with HCl of known conc.
3. Use methyl orange indicator
4. Repeat to obtain titres

### To: Prepare standard solution

Procedure:

1. Dissolve solid in conical flask with distilled water
2. Pour the solution into volumetric flask with washings
3. Add water up to the mark & shake

### Key concepts

Mean titre = Mean of titres within range

### Common questions

**Standard solution**

* Reason for washing: Ensure all solution is transferred

**Titration**

* Reduce % uncertainty: Use more dilute titrate, larger titre
* Why burette rinsed with titrate: Remove remaining water
* Effect on titre if no solution in tip: More solution in flask, larger titre
* Necessary addition of compound: Remove an ion that might react with indicator

## Find: Percentage yield of halogenoalkane

### To: Prepare organic solutions with reflux / distillation

Procedure: Prepare solution 🡪 reflux & distillation 🡪 transfer to separating funnel 🡪 final distillation

|  |  |
| --- | --- |
|  |  |
| *Reflux setup – ensures complete oxidation* | *Distillation setup – prevents further oxidation* |

### Common questions

*Often given as “Drop by drop”*

**Prepare solution**

* Slow addition of acid with cooling: Reaction exothermic, prevent boiling over

**Reflux**

* Powdered solid: Increase surface area, faster reaction
* Anti-bumping granules: Reduce size of bubbles formed

Prevent mixture from overflowing to condenser

**Separating funnel**

* Observations physical properties: Density comparison, insolubility
* Effervescence: Remove stopper to release built-up pressure
* Collect top layer: Run off lower layer, run of top layer
* Species added when running off: Remove excess acid
* Anhydrous species added: Remove water, solution goes clear

**Final distillation**

* Distillate collected in temp range: Only target species boil within this range, above this range others will be collected

## To: Collect gas

### Common questions

* Wool with anhydrous solid in tube: Absorb water as would otherwise react with product
* Wool with reagent in tube: Holds reagent in place, wool does not react
* Heating stop, remove delivery tube: Suck-back occurs, lower pressure in test tube, test tube will crack

## Miscellaneous methods

* Reducing % uncertainties: Increase measured value by different methods e.g. reducing concentration
* Ensuring full reactions: No production / reactant observations are seen anymore
* Using granules instead of powder: Allow gas to pass through
* Species in collection apparatus: Remove unreacted reactants
* Obtain dry crystals: Heat to crystallization, leave to crystallize, dry in oven
* Delay between adding gas and heat: Ensure pure gas in container

## Common Indicators

|  |  |  |  |
| --- | --- | --- | --- |
| ***Indicator*** | ***Acidic*** | ***Initial*** | ***Alkaline*** |
| Methyl orange | **Red** | **Orange** | **Yellow** |
| Phenolphthalein | **Colorless** | | **Pink** |
| Litmus paper | **Red** | **N/A** | **Blue** |
| Universal | **Red** | **Green** | **Blue** |

Initial color: pH of initial solution

End color: pH of final solution

Make sure to check with Brønsted–Lowry acid–base theory!