Preparation of ammonium chloride

Ammonium chloride is a white crystalline, very well soluble in water, but less soluble in ethanol. It also sublimes very easy. Ammonium chloride can be prepared by the protolytic reaction of ammonia with hydrochloric acid, as described by the chemical equation

$$\mathbf{NH_3(aq) + HCl(aq) \longrightarrow NH_4Cl(aq)}$$

In water solution of ammonium chloride the hydrolysis of ammonium cation occurs and the concentration of oxonium cations increases, $c(H_3O^+) > c(OH^-)$, therefore pH < 7, and the solution is of acidic nature.

$$NH_4^+(aq) + H_2O(l) \rightleftharpoons NH_3(aq) + H_3O^+(aq)$$

Work

Prepare ammonium chloride by the protolytic reaction of NH_3 with HCl.

Chemicals

- ammonia, NH_3, concentrated water solution, $w(NH_3) = 0{,}26$
- hydrochloric acid, HCl, concentrated, $w(HCl) = 0{,}36$

Procedure

Warning! Ammonia and hydrogen chloride very strongly irritate human respiration system. In addition, they also react together in gaseous phase forming dense white fume therefore, we must work with them in a fume hood.

Measure calculated volumes of concentrated ammonia and concentrated hydrochloric acid in a graduated cylinder and then pour them separately into another two beakers. It is recommended to use a higher beaker for hydrochloric acid. Using a glass rod add slowly ammonia solution to the hydrochloric acid in small portions. After adding each portion stir shortly the final solution and quickly cover it with a watch glass or a Petri dish (Fig. 1) to avoid the escape of white fume of ammonium chloride. When entire ammonia solution is added tune the resulting pH to be basic by adding another portion of ammonia solution (universal litmus paper changes its colour to dark blue). Because the commercial hydrochloric acid contains ferric cations (responsible for yellow colour of solution), sufficient excess of ammonia is necessary to cause their precipitation in form of brown-red hydrated iron(III) oxide (rust), $Fe_2O_3 \cdot x\ H_2O$, in accord with the equation

$$2\ Fe^{3+}(aq) + 6\ NH_3(aq) + (3 + x)\ H_2O(l) \longrightarrow Fe_2O_3 \cdot x\ H_2O(s) + 6\ NH_4^+(aq)$$

Warning! Do not add very great excess of ammonia. It could complicate next steps of the procedure, such as increasing evaporation time and changing solubility of the product.

The resulting solution with floating rust (photo in Fig. 1) boil for 2 minutes to remove the excess of ammonia and let it stand for a while. If necessary, ammonia can be also exhausted using filtration funnel connected to the water (not vacuum!) pump (Fig. 2). After cooling to room temperature filter the solution using a fluted filter paper directly to an evaporating dish.

Put the evaporating dish with the pale pink solution on a water bath and wait until crystallisation film is formed (saturated solution). Turn off a gas burner and measure the temperature of the solution. Pour the solution from the evaporating dish into a crystallization dish and add two drops of concentrated ammonia.
Place the crystallization dish to the coolest part of the laboratory (or use an ice bath). After cooling to room temperature measure the temperature of the solution with crystals formed. Filter out the crystals on a Büchner funnel under vacuum and place wet crystals of the product on a watch glass into an oven heated over 100 °C to dry them up. Weigh the final dry white crystals of ammonium chloride.

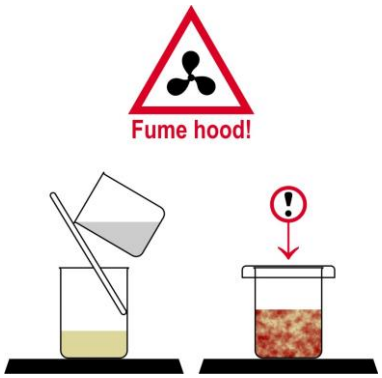
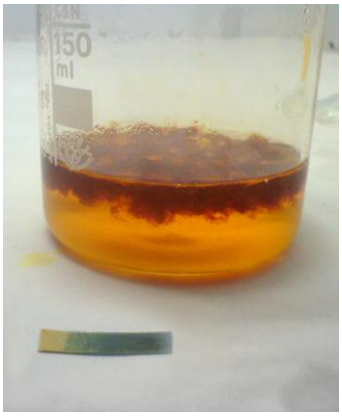


Fig. 1 Neutralisation



Precipitated hydrated iron(III) oxide (rust) in basic solution. Litmus paper changed its color to dark blue.

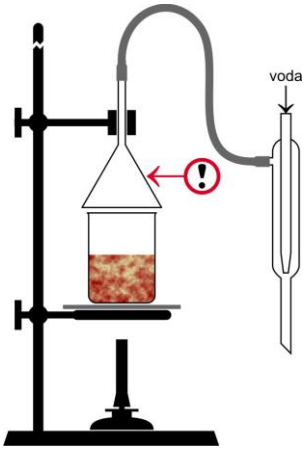


Fig. 2 Short boiling of a basic solution

Safety instructions

***Ammonia* – NH_3**

R10	Flammable.
R23	Toxic by inhalation.
S16	Keep away from sources of ignition – No smoking
S38	In case of insufficient ventilation wear suitable respiratory equipment
S7/9	Keep container tightly closed and in a well-ventilated place.

***Hydrochloric acid* – HCl**

R34	Causes burns.
R37	Irritating to respiratory system.
S2	Keep out of the reach of children
S26	In case of contact with eyes, rinse immediately with plenty of water and seek medical advice

***Ammonium chloride* – NH_4Cl**

R22	Harmful if swallowed.
R36	Irritating to eyes.
S22	Do not breathe dust