PROF. DR. SELEN BILGE KOÇAK CHM356 INORGANIC CHEMISTRY LABORATORY

LABORATORY TECHNIQUES TO BE USED IN ANORGANIC CHEMISTRY LABORATORY

DISSOLVING

Dissolving is done by adding the substance little by little into the solvent. After each addition, the material is waited to dissolve, while the mixture is mixed with a glass stir rod and large pieces, if any, are crushed. The second part should not be added until the added part is dissolved. If necessary, the mixture is heated for dissolution. During heating, the glass container is not directly in contact with the flame. For this purpose, wire gauze with asbestos is used. When working with organic and flammable solvents, naked flame heating is very dangerous, so electric heaters should be used if possible. If there is no electric heater, heating should be done in low flame and by using wire gauze with asbestos Excessive boiling should be avoided during heating.

CRYSTALLIZATION

Crystallization is one of the purification methods. Crystallization is based on the fact that a substance dissolves in a solvent much in hot and little in cold. For this reason, the choice of solvent is very important in the crystallization process. In some cases, the substance can be separated into oil without crystallization. In such cases, the solution is reheated to dissolve the oily substance and leave to cool very slowly without shaking. The beaker can be wrapped with a clean towel to ensure slow cooling. If crystallization does not occur even though the solution has cooled down, either rubbing the wall of a beaker with a glass stir rod should be applied, or a graft crystal should be thrown into the solution. Also, it should not be forgotten that crystallization is difficult from solutions that are concentrated by evaporation. For this reason, excessive evaporation should be avoided in order to concentrate the solution. After the crystals are obtained, they are filtered off from a funnel, washed with a small amount of solvent used for crystallization which does not dissolve the crystals in cold, and dried.

PROF. DR. SELEN BİLGE KOÇAK CHM356 INORGANIC CHEMISTRY LABORATORY

CRYSTALLIZATION TECHNIQUE

A hot saturated solution of the substance to be crystallized in a suitable solvent is prepared. This solution is hot filtered through a previously heated funnel into a clean flask. The purpose of this filtration is to separate solid particles which are insoluble in hot solvent. The purpose of heating the funnel is to prevent crystallization of the hot saturated solution from the cold funnel walls. For this purpose, the funnel should be heated by either holding it to the vapors of the solution or keeping it in the oven. The saturated solution filtered into the beaker is left to cool by closing the watch glass into the mouth of the beaker. Cooling speed is one of the most important factors affecting the size of the crystals. For this reason, slow cooling is required if large crystals are desired, and fast cooling is required if small crystals are desired. An ice bath can be used for rapid cooling.

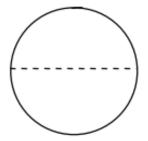
FILTRATION

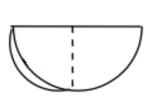
Filtration, the technique used to separate solids from liquids. Two filtration techniques are generally used in chemical separations:

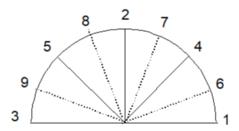
- **1.** *Gravity filtration*
- 2. Vacuum filtration

Gravity filtration

How to Fold Filter Paper?





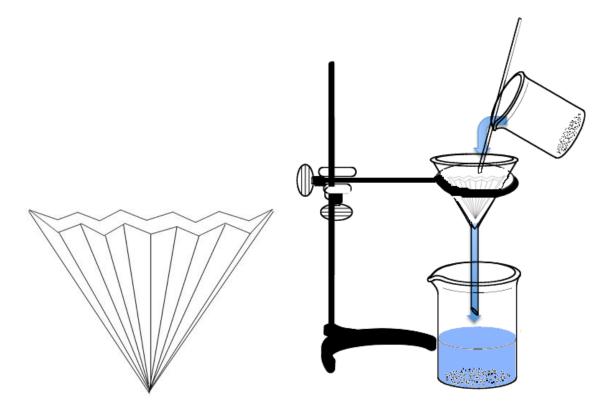


Fold paper in half

Fold paper in quarters

Preparation of crushed filter paper

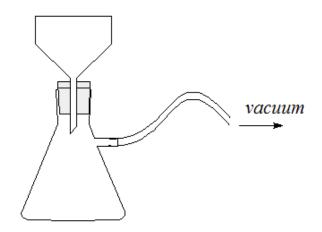
PROF. DR. SELEN BİLGE KOÇAK CHM356 INORGANIC CHEMISTRY LABORATORY



The paper is placed in the funnel in this state. Depending on its diameter, the funnel is placed over an iron ring or a clay triangle, on a support. After a clean beaker is placed under the funnel, the mixture to be filtered is poured with a glass stir rod in the middle of the filter paper. The mixture to be filtered should never be filled up to 1 cm below the upper edge of the filter paper.

Vacuum filtration

The filtering using Bucher funnel



PROF. DR. SELEN BİLGE KOÇAK CHM356 INORGANIC CHEMISTRY LABORATORY

EXPERIMENT NUMBER	1
THE NAME OF THE EXPERIMENT	ALUM (DOUBLE SALT)
FORMULA	$M^{+}M^{3+}(SO_{4})_{2}.12H_{2}O$

EXPERIMENTAL PROCEDURE

An alum is a type of chemical compound, usually a hydrated double salt of metal cation with (+3) charge such as Al^{3+} and Cr^{3+} with the monovalent cation such Na^+ , K^+ and NH_4^+ . Cations with +1 and +3 charges can be crystallized together with sulfate ion and 12 mole water to form a double salt called alum. For this purpose, the following salts are dissolved in approximately 5 mL of water; the solution is set aside crystallization in a large beaker. If a graft crystal is thrown into the solution, it will be seen that the graft crystal grows a day later.

$KAl(SO_4)_2 \ 12H_2O alum \rightarrow$	$0.5 g K_2(SO_4) + 1.9 g Al_2(SO_4)_3.18H_2O$
$NaAl(SO_4)_2 \ 12H_2O alum \rightarrow$	$0.5 g Na_2(SO_4) + 2.4 g Al_2(SO_4)_3.18H_2O$
$NH_4Al(SO_4)_2 \ 12H_2O \ alum \rightarrow$	$0.5 g (NH_4)_2 SO_4 + 2.7 g Al_2(SO_4)_3.18H_2O$
$KCr(SO_4)_2 \ 12H_2O alum \rightarrow$	$0.5 g K_2(SO_4) + 1.4 g Cr_2(SO_4)_3.6H_2O$
$NaCr(SO_4)_2 \ 12H_2O alum \rightarrow$	$0.5 g K_2(SO_4) + 1.76 g Cr_2(SO_4)_3.6H_2O$
$NH_4Cr (SO_4)_2 \ 12H_2O alum \rightarrow$	$0.5 g (NH_4)_2 SO_4 + 2.0 g Cr_2(SO_4)_3.6H_2O$