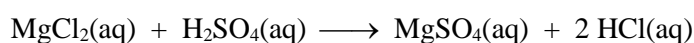
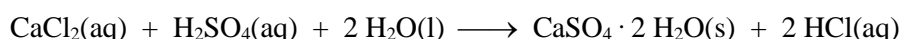
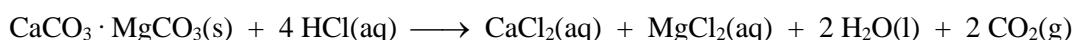


Preparation of calcium sulphate hemihydrate and magnesium sulphate heptahydrate

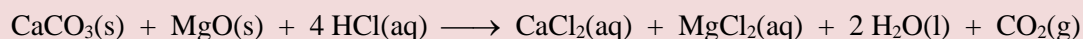
Calcium sulphate hemihydrate $\text{CaSO}_4 \cdot 0,5\text{H}_2\text{O}$ can be obtained by heating of calcium sulphate dihydrate $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ (gypsum) over $150\text{ }^\circ\text{C}$. Gypsum has layer-like structure. Layers of cations and anions are connected by the molecules of water with relatively weak hydrogen bonds. Therefore the crystals of gypsum are easily fissile. Mohs scale of mineral hardness defines hardness value 2 as gypsum. After the reaction with water calcium sulphate hemihydrate hardens to solid substance formed of fibrous crystals of dihydrate.

Magnesium sulphate heptahydrate $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$ is white crystalline well soluble in water. It is usually used in medicine as a laxative, as a source of magnesium, or as a bath salt. Commonly it is a component of mineral waters. Heating $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$ the lower hydrates are stepwise obtained. Over $238\text{ }^\circ\text{C}$ anhydrous magnesium sulphate is formed. Depending of temperature various hydrates $\text{MgSO}_4 \cdot x\text{H}_2\text{O}$, ($x = 1, 2, 4, 5, 6, 7$ and 10) crystallize from the aquatic solution.

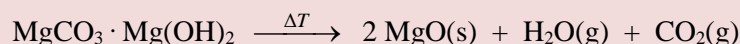
Both calcium and magnesium sulphates are commonly prepared from calcium-magnesium carbonate $\text{CaCO}_3 \cdot \text{MgCO}_3$ (dolomite) by the reaction with HCl a H_2SO_4 .



Attention! Because of unavailability of chemically pure dolomite, a mixture of calcium carbonate and magnesium oxide must be used instead. Mix both chemicals in molar ratio $\text{Ca} : \text{Mg} = 1 : 1$ like in dolomite.



Magnesium oxide should be prepared by thermal decomposition of basic magnesium carbonate $\text{MgCO}_3 \cdot \text{Mg}(\text{OH})_2$ at higher temperature (annealing), according to the reaction



Work

Prepare calcium sulphate hemihydrate and magnesium sulphate heptahydrate from 4,0 g of mixture of calcium carbonate and magnesium oxide with molar ratio $\text{Ca} : \text{Mg} = 1 : 1$.

Chemicals

- calcium carbonate, CaCO_3 , white crystalline
- magnesium carbonate basic, $\text{MgCO}_3 \cdot \text{Mg}(\text{OH})_2$, white crystalline
- hydrochloric acid, HCl , concentrated, $w(\text{HCl}) = 0,36$
- sulphuric acid, H_2SO_4 , concentrated water solution, $w = 0,96$

Procedure

In a clean iron crucible anneal enough basic magnesium carbonate. Let cool down the obtained magnesium oxide powder. Mix the calculated amount of magnesium oxide with the calculated amount of calcium carbonate. If necessary, spread the mixture in a mortar. Put the powdered mixture to a beaker and add stoichiometric volume of 20,0 % hydrochloric acid. If some unreacted solid is still present in the beaker, add another portion of hydrochloric acid remove all traces of a white solid. After the reaction heat the solution and filter it through a fluted filter paper.

Add calculated volume of 20,0 % solution of sulphuric acid in small excess to the filtrate containing CaCl_2 and MgCl_2 . White $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ precipitates while MgSO_4 remains dissolved in the solution. Filter out the precipitated $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ on a Büchner funnel and wash the filter cake with water. After drying over $120\text{ }^\circ\text{C}$ in an oven we obtain $\text{CaSO}_4 \cdot 0,5\text{H}_2\text{O}$.

Evaporate the filtrate containing magnesium sulphate in an evaporation dish until dry to eliminate the unreacted excess of hydrogen chloride. Weigh the dried $\text{MgSO}_4 \cdot \text{H}_2\text{O}$ and dissolve it in the calculated amount of water to obtain the solution saturated at $45\text{ }^\circ\text{C}$. Cool down the mixture in an ice bath. Under $48\text{ }^\circ\text{C}$ heptahydrate $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$ crystallizes. Over this temperature hexahydrate is formed.

Safety instructions

Calcium carbonate – CaCO₃

- R37/38** Irritating to respiratory system and skin.
- S26** In case of contact with eyes, rinse immediately with plenty of water and seek medical advice
- S36** Wear suitable protective clothing

Magnesium carbonate – MgCO₃

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Magnesium hydroxide – Mg(OH)₂

- R36/37/38** Irritating to eyes, respiratory system and skin.
- S26** In case of contact with eyes, rinse immediately with plenty of water and seek medical advice
- S36** Wear suitable protective clothing

Magnesium oxide – MgO

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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

Sulfuric acid – H₂SO₄

- R23** Toxic by inhalation.
- R34** Causes burns.
- R49** May cause cancer by inhalation.
- S23** Do not breathe gas/fumes/vapour/spray (appropriate wording to be specified by the manufacturer)
- S45** In case of accident or if you feel unwell seek medical advice immediately (show the label where possible)
- S36/37/39** Wear suitable protective clothing, gloves and eye/face protection.

Calcium sulfate hemihydrate – CaSO₄ · 0,5H₂O

- R36/37/38** Irritating to eyes, respiratory system and skin.
- S26** In case of contact with eyes, rinse immediately with plenty of water and seek medical advice
- S37/39** Wear suitable gloves and eye/face protection.

Magnesium sulfate heptahydrate – MgSO₄ · 7H₂O

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