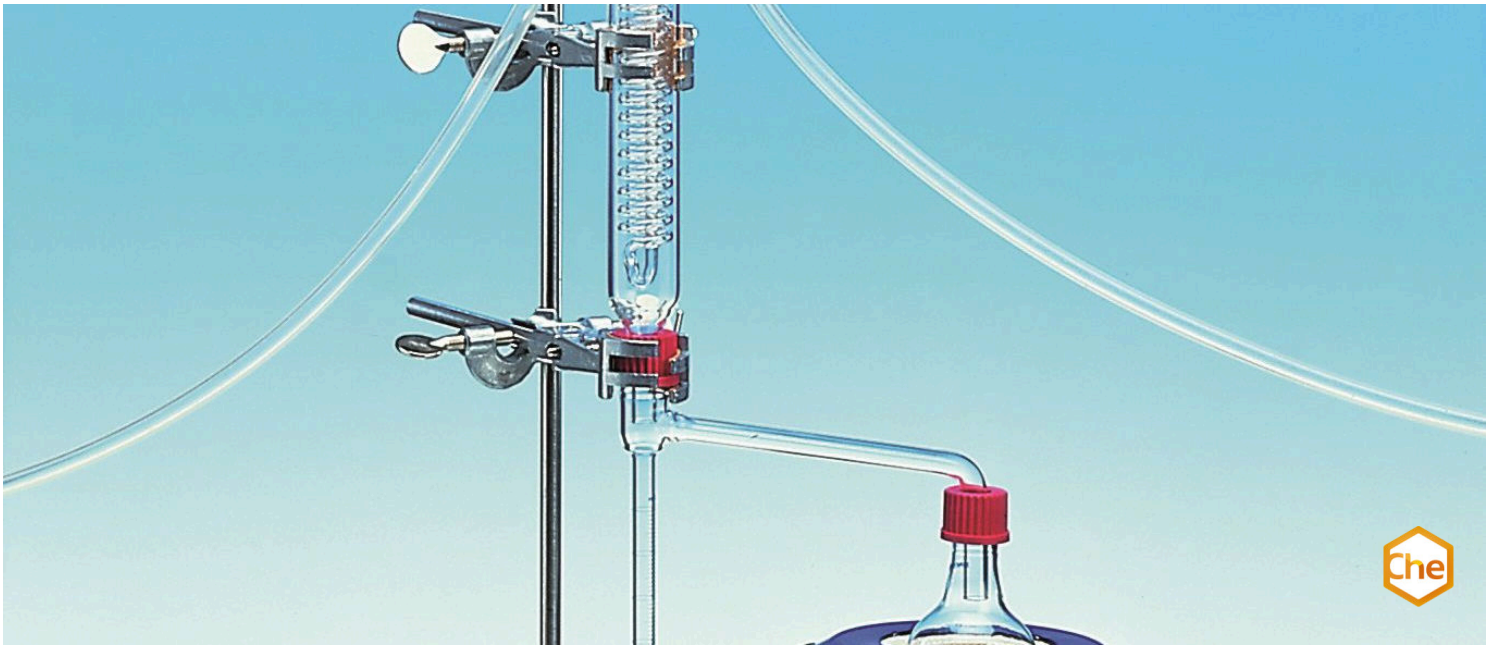
Preparation of p-toluenesulfonic acid



The students to prepare p-Toluenesulphonic acid from toluene and concentrated sulphuric acid.

Chemistry	Organic chemistry	Basics: Organic chemistry
Chemistry	Industrial Chemistry	industrial synthesis

Difficulty level	Group size	Preparation time	Execution time
medium	2	45+ minutes	45+ minutes

This content can also be found online at:

http://localhost:1337/c/6011a165ebbd4d000315b7a9

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General information

Application

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Experimental setup

In this experiment, the students learn how to prepare p-Toluenesulphonic acid from toluene and concentrated sulphuric acid.

p-Toluenesulphonic acid and its salts are used as catalysts for reactions such as esterifications, dehydrations, and polymerisation reactions.
The sulphonation of toluene is an electrophilic substitution at the aromatic compound.

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Other information (1/2)

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Prior knowledge

The Students should already be familiar with sulphonation, aromatic compounds and the azeotropic distillation in theory.

Scientific principle

p-Toluenesulphonic acid and its salts are used as catalysts for reactions such as esterifications, dehydrations, and polymerisation reactions.

Unlike most other electrophilic substitutions, sulphonation is a reversible reaction. If water is present at higher temperatures, hydrolysis is successful.

Other information (2/2)

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Learning objective

The students to prepare p-Toluenesulphonic acid from toluene and concentrated sulphuric acid.

The sulphonation of toluene is an electrophilic substitution at the aromatic compound.

Tasks

The students prepare p-Toluenesulphonic acid from toluene and concentrated sulphuric acid.

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Safety instructions

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- Toluene is a volatile, water-insoluble, highly flammable liquid. Its vapours are heavier than air but they can form explosive mixtures in combination with air. Toluene is harmful (low level of toxicity) and it has a strong irritating effect on the mucous membranes. A narcotic effect is possible.
- Concentrated acids are highly caustic. They burn the skin and destroy textile fabrics. For diluting, first add the water, then the acid (protective glasses, laboratory coat, gloves).
- For the H- and P-phrases please refer to the corresponding safety data sheets.
- The general instructions for safe experimentation in science education apply to this experiment.

Theory

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p-Toluenesulphonic acid and its salts are used as catalysts for reactions such as esterifications, dehydrations, and polymerisation reactions.
They are more gentle reagents than sulphuric acid and, as powders, they can be dosed better. The sulphonation of toluene is an electrophilic substitution at the aromatic compound. The sulphonating reagents can be the free sulphur trioxide or the HSO3+ cation, which is present in the concentrated sulphuric acid in equilibrium.

Unlike most other electrophilic substitutions, sulphonation is a reversible reaction. If water is present at higher temperatures, hydrolysis is mostly successful. In order to displace the equilibrium towards the desired product, the generated water must be bound or removed from the reactionmixture by way of azeotropic distillation.

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Equipment

Position	Material	Item No.	Quantity
1	Support base DEMO	02007-55	1
2	Support rod, stainless steel, l = 600 mm, d = 10 mm	02037-00	2
3	Retort stand, h = 750 mm	37694-00	1
4	Right angle boss-head clamp	37697-00	3
5	Universal clamp	37715-01	3
6	Round bottom flask, 100ml, GL 25/12	35841-15	1
7	Water separator GL25/12	35790-15	1
8	Dimroth-Condensor, GL 25/12	MAU-27223500	1
9	Graduated cylinder, Borosilicate, 100 ml	36629-00	1
10	Beaker, Borosilicate, tall form, 150 ml	46032-00	1
11	Beaker, Borosilicate, tall form, 250 ml	46027-00	1
12	Buchner funnel, i.d. = 70, porcelain	32707-00	1
13	Circular filter,d 70 mm,100 pcs	32977-02	1
14	Filter flask, 500ml, PN 34.5	34419-01	1
15	Rubber gaskets,conical,set of 8	39265-00	1
16	Water jet pump, plastic	02728-00	1
17	Porcelain dish, 75ml, d = 80 mm	32516-00	1
18	Funnel, glass, top dia. 50 mm	34457-00	1
19	Heating mantle f. roundbottom flask, 100 ml	49541-93	1
20	Clamp for heating mantle	49557-01	1
21	Power regulator	32288-93	1
22	Desiccator, vacuum, diam. 150 mm	34126-00	1
23	Porcelain plate f.desiccator150mm	32474-00	1
24	Rubber tubing, i.d. 6 mm	39282-00	2
25	Rubber tubing,vacuum,i.d.6mm	39286-00	1
26	Hose clip, diam. 8-16 mm, 1 pc.	40996-02	2
27	Glass rod,boro 3.3,l=300mm, d=7mm	40485-05	1
28	Wash bottle, plastic, 500 ml	33931-00	1
29	Spoon, special steel	33398-00	1
30	Crucible tongs, 200 mm, stainless steel	33600-00	1
31	Boiling beads, 200 g	36937-20	1
32	Silica gel, orange, granular, 500 g	30224-50	1
33	Silicon grease Molykote, 50 g	31863-05	1
34	Toluene 250 ml	30236-25	1
35	Sulphuric acid, 95-97%, 500 ml	30219-50	1
36	Sodium chloride, 500 g	30155-50	1
37	Water, distilled 5 l	31246-81	1

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Setup and procedure

Setup (1/2)

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Setup of the base

Set up the experiment as shown in the figure on the left and right side

Take a retort base and put two rods in the base (left figure)

Fix the heating mantle on the rod on the right side. The clamp to fix the heating mantle is in the scope of supply ot the heating mantle.

Setup heating mantle

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Setup (2/2)

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Set up the water separtor as shown in the figure on the right.

Fill the 100 ml round bottom flask with 50 ml of toluene and 10 ml of concentrated sulphuric acid.

Connect the flask and the water seperator and fix them with a clamp as shown in the figure on the right.

The flask with the toluene is positioned in the middle ot the heating mantle.

Take care that the seperator is closed, the tap should be positioned horizontal (as shown)

Procedure

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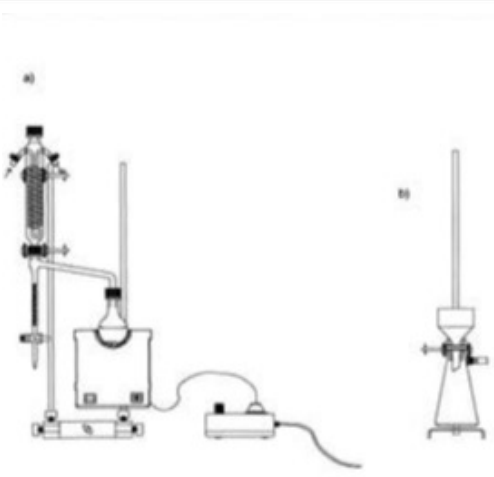
Boil the reaction mixture for 15 minutes with a return flow at the water separator. Then, let it cool. In the meantime, prepare a saturated sodium chloride solution and pour it into the cool reaction mixture (if necessary, dilute it carefully beforehand with a little water).

If the sodium salt of the p-toluenesulphonic acid does not crystallise immediately, prepare some initial crystallisation nuclei by rubbing with a glass rod.

Filter the precipitated salt by suction and fill it into an evaporating dish. Dry it at 105°C to 110°C in a drying cabinet and let it cool in a desiccator.



Experimental setup

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Evaluation

Evaluation (1/6)

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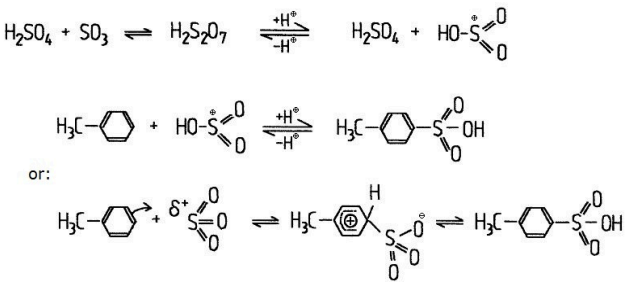
Result

During boiling, the water is separated at the water separator. When the mixture cools, a thick paste may precipitate. This paste dissolves again when water is added carefully. From the saturated sodium chloride solution, which was poured into the product, p-toluenesulphonic acid sodium salt crystallises out in the form of white, glossy crystals.



The sulphonation of toluene is an electrophilic substitution at the aromatic compound. The sulphonating reagents can be the free sulphur trioxide or the HSO3+ cation, which is present in the concentrated sulphuric acid in equilibrium.

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Evaluation (2/6)

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Unlike most other electrophilic substitutions, sulphonation is a reversible reaction. If water is present at higher temperatures, hydrolysis is mostly successful.

$$Ar + H_2SO_4 \rightleftharpoons ArSO_3 + H_2O$$

In order to displace the equilibrium towards the desired product, the generated water must be bound or removed from the reaction mixture by way of azeotropic distillation. Pure p-toluenesulphonic acid is highly hygroscopic. The stoichiometric addition of water to the cool mixture or any water residues that are still present enable the targeted isolation of the p-toluenesulphonic acid monohydrate. Normally, however, the free sulphonic acid is not isolated. Instead, it is converted directly into sodium salt.
When the cool reaction mixture is added to a saturated sodium chloride solution, the sodium salt of the p-toluenesulphonic acid precipitates out. Arylsulphonic acids are strong acids. Their acidic strength can be compared to that of hydrochloric acid. This is why the acid is dissociated to a large extent. Due to the surplus of sodium ions in the saturated sodium chloride solution, the solution equilibrium is shifted to such an extent that sodium sulphonate precipitates out.

Evaluation (3/6)

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$$ArSO_3^- + Na^+ \rightleftharpoons ArSO_3Na$$

p-toluenesulphonic acid and its salts are used as catalysts for reactions such as esterifications, dehydrations, and polymerisation reactions. They are more gentle reagents than sulphuric acid and, as powders, they can be dosed better.
· **p-toluenesulphonic acid:**
Molar mass: 172.20 g/mol
Melting point: 38 °C
· **p-toluenesulphonic acid monohydrate:**
Molar mass: 190.22 g/mol
Melting range: 103-104 °C
Solubility in H2O/20 °C: 670 g/l
· **p-toluenesulphonic acid sodium salt:**
Molar mass: 194.19 g/mol

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Evaluation (4/6)

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What for are p-toluenesulphonic acid and its salts used?

- ○ p-toluenesulphonic acid and its salts are used as catalysts for reactions such as esterifications, dehydrations, and polymerisation reactions.
- ○ p-toluenesulphonic acid and its salts are used as binding agents in the Kjeldahl-analysis.
- ○ None of the answers is correct.
- ○ p-toluenesulphonic acid and its salts are used as rinsing agents.

Überprüfen

Evaluation (5/6)

PHYWE

What is the sulphonation of toluene exactly?

- ☐ The sulphonation of toluene is an electrophilic substitution at the aromatic compound.
- ☐ None of the answers is correct.
- ☐ The sulphonation of toluene is an electrophilic substitution at the H-O group.
- ☐ The sulphonating reagents can be the free sulphur trioxide or the HSO3+ cation, which is present in the concentrated sulphuric acid in equilibrium.

Überprüfen

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Evaluation (6/6)

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Summary of the experiment

During boiling, the water is _____ at the water separator. When the mixture _____, a thick paste may precipitate.
This paste dissolves again when water is added carefully. From the saturated sodium chloride solution, which was poured into the product, _____ acid sodium salt crystallises out in the form of _____, glossy crystals.

white
p-toluenesulphonic
cools
separated

Überprüfen

Folie	Punktzahl / Summe
Folie 16: p-toluenesulphonic acid and its salts	0/1
Folie 17: Sulphonation of toluene	0/2
Folie 18: Summary of the experiment	0/4

Gesamtsumme 0 / 7

Lösungen Wiederholen

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