Toluene: Bromination in the nucleus

Bromine is polarised and, thereby, activated by zinc chloride as a Lewis acid. It can attach itself in an ionic manner to the toluene nucleus via several complex intermediate stages. Following a dehydrobromination, bromotoluene is formed, i.e. the product of bromination in the nucleus.

Chemistry > Organic chemistry > Hydrocarbons

Chemistry > Organic chemistry > Dyestuffs / Household chemistry

Difficulty level	Group size	Preparation time	Execution time
hard	2	10 minutes	20 minutes

This content can also be found online at:

http://localhost:1337/c/600e967ee9356e0003e697e9

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General information

Application

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Experimental setup

Bromine is polarized and, thereby, activated by zinc chloride as a Lewis acid. It can attach itself in an ionic manner to the toluene nucleus via several complex intermediate stages.

Following a dehydrobromination, bromotoluene is formed, i.e. the product of bromination in the nucleus. In the absence of a catalyst and under the influence of light, however, side chain bromination takes place via radical intermediate stages. The reaction can be controlled in a targeted manner by varying the reaction conditions.

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Other information (1/2)

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Prior knowledge

The Students should already be familiar with the bromination in the nucleus of toluene in theory.

Scientific principle

Bromine is polarized and, thereby, activated by zinc chloride as a Lewis acid.

Other information (2/2)

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Learning objective

The students learn the bromination in the nucleus of toluene.

Tasks

The students brominate toluene using bromine, change the reaction conditions to optimize their results and distillate the resulting mixture.

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Safety instructions (1/3)

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- Experiments involving bromine must be performed under an exhaust hood.
- Bromine vapors irritate the mucous membranes and cause inflammation. Liquid bromine burns the skin.
- Bromine must be kept under lock and key. Wear protective gloves when transferring bromine into another vessel.
- 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.

Safety instructions (2/3)

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- Dichloromethane (methylene chloride) is a colourless, sparingly water-soluble liquid that decomposes in naked flames and hot surfaces (≥ 120°C) while forming irritant, corrosive substances. Degreasing of the skin. Increases the carbon monoxide level in the blood if ingested in small concentrations.
- 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.

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Safety instructions (3/3)

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- Solid residues that contain heavy metals or their ions must also be collected in this vessel.
- Silver-containing solutions and solids must be collected in their own collecting vessels (treatment of the silver to obtain silver nitrate).
- 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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Bromine is polarized and, thereby, activated by zinc chloride as a Lewis acid. It can attach itself in an ionic manner to the toluene nucleus via several complex intermediate stages. Following a dehydrobromination, bromotoluene is formed, i.e. the product of bromination in the nucleus.

In the absence of a catalyst and under the influence of light, however, side chain bromination takes place via radical intermediate stages. The reaction can be controlled in a targeted manner by varying the reaction conditions.

Remember:
Cold, catalyst ~ nucleus
Boiling heat, sunlight ~ side chain

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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	Lab jack, 200 x 200 mm	02074-01	1
7	Three neck round bottom flask, 100 ml	MAU-27220501	1
8	Closure caps,10, GL25	41221-03	1
9	Liebig Condenser, with head, GL18/8	MAU-27223000	1
10	Dropping funnel, 50 ml, Gl 18	MAU-27222000	1
11	Round-bottom flask, 50 ml	MAU-27220001	5
12	Adapter for 4 flasks, GL25	MAU-27227500	1
13	Cristallizing dish, boro3.3, d = 125 mm	46244-00	1
14	Lab thermometer,w.stem,-10..+250C	38061-01	1
15	Magnetic stirrer with heater MR Hei-Standard	35751-93	1
16	Magnetic stirring bar 15 mm, cylindrical	46299-01	1
17	Magnetic stirring bar 30 mm, cylindrical	46299-02	1
18	Magnetic stirring bar, 50 mm, cylindrical	46299-03	1
19	Separator for magnetic bars	35680-03	1
20	Graduated cylinder, borosilicate, 25 ml	36627-00	2
21	Beaker, Borosilicate, low-form, 400 ml	46055-00	1
22	Secure bottle, 500 ml, 2 x Gl 18/8, 1 x 25/12	34170-01	1
23	Spring manometer, 0...-1000 mbar	34170-02	1
24	Glass tube,right-angled, 10 pcs.	36701-52	1
25	Glass tubes, right-angled, 10	36701-57	1
26	Stopcock,3-way,t-shaped, glass	36731-00	1
27	Water jet pump, plastic	02728-00	1
28	Funnel, glass, top dia. 50 mm	34457-00	1
29	Test tube, 160 x 16 mm, 100 pcs	37656-10	1
30	Test tube rack for 12 tubes, holes d= 22 mm, wood	37686-10	1
31	Pasteur pipettes, 250 pcs	36590-00	1
32	Rubber caps, 10 pcs	39275-03	1
33	Bromine filler w. rubber cap	45100-00	1
34	Rubber gloves, size L (9), one pair	39324-00	1
35	Teclu burner, DIN, natural gas	32171-05	1
36	Safety gas tubing, DVGW, sold by metre	39281-10	1
37	Lighter f.natural/liquified gases	38874-00	1
38	Hose clip f.12-20 diameter tube	40995-00	2
39	Hose clip, diam. 8-16 mm, 1 pc.	40996-02	2
40	Spoon, special steel	33398-00	1
41	Glass rod,boro 3.3,l=300mm, d=7mm	40485-05	1
42	Wash bottle, plastic, 500 ml	33931-00	1
43	Rubber tubing, i.d. 6 mm	39282-00	3
44	Rubber tubing,vacuum,i.d.6mm	39286-00	2
45	Silicone oil 500 ml	31849-50	1
46	Dichloromethane 250 ml	31255-25	1
47	Toluene 250 ml	30236-25	1
48	Bromine-Solution, 250 ml	30046-25	1
49	Zinc chloride, dry, 250 g	31983-25	1
50	Silver nitrate, cryst. 15 g	30222-00	1
51	Nitric acid , 65% 1000 ml	30213-70	1
52	Copper foil, 0.1 mm, 100 g	30117-10	1
53	Water, distilled 5 l	31246-81	1

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Setup and procedure

Setup

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Set up the experiment as shown in the figure on the right.

In order to perform the reaction without light, cover the distillation flask, i.e. the part that is not immersed in the heating bath, and the dropping funnel with household aluminum foil.

Fill the distillate flask to two thirds with dichloromethane. A piece of rubber tubing runs from the hose connector of the vacuum adapter to the rim of the beaker that is three quarters full with water.

Experimental setup

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Procedure (1/2)

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Experimental setup

Fill approximately 40 ml of toluene and a small quantity of waterfree zinc chloride (covering the tip of a spatula) into the threeneck flask.

Fill the dropping funnel with a solution of 10 ml of bromine and 10 ml of toluene and seal the third neck of the flask with a GL 25 sealing cap.

Let the bromine solution drip slowly into the content of the flask while stirring.

After the start of the reaction, gas flows through the apparatus.

This gas is washed by the dichloromethane in the adapter and absorbed by the water in the beaker.

Procedure (2/2)

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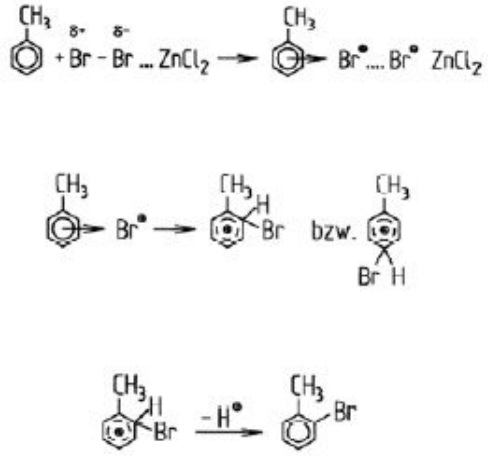
When the reaction is complete, remove the dropping funnel from the three-neck flask and seal this neck also with a sealing cap. Replace the 50 ml round bottom flask with an adapter with four 40 ml round bottom flasks and distil the reaction mixture in the water jet pump vacuum.

Put several drops of the distillate onto a thoroughly heated piece of copper foil and heat it over the nonluminous burner flame.

Acidify part of the aqueous solution from the beaker in a test tube with diluted nitric acid and add an aqueous silver nitrate solution. As a reference, add some bromine to a toluene sample in a test tube and shake it.



Reaction chain

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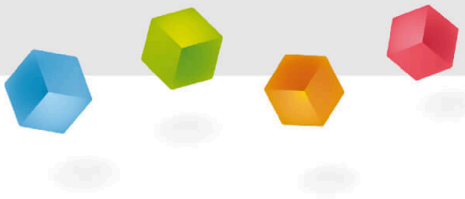

Evaluation

Evaluation (1/4)

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Result

After a short period, a reaction commences that becomes increasingly stronger over time. The gas that is formed dissolves in the water in the beaker while displaying streaks. Caused by the silver nitrate solution, a yellow substance precipitates from the acidified sample.

The bromine vapours that are dragged along during the gas development are dissolved in the dichloromethane. In the water jet pump vacuum (approximately 20 hPa), a liquid passes over at 60°C. A Beilstein test of this liquid leads to a positive result.

The toluol, to which bromine was added, also decolours over time. This reaction, however, is comparatively slow.

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Evaluation (2/4)

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What happens to the toluol, to which bromine was added?

- ○ The toluol, to which bromine was added, decolours over time. This reaction is comparatively slow.
- ○ The toluol, to which bromine was added, becomes yellow over time.
- ○ None of the answers is correct.
- ○ The toluol, to which bromine was added, decolours. This reaction happens immediately.

Überprüfen

Evaluation (3/4)

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What are the bromine vapors that are dragged along during the gas development dissolved in?

- ○ None of the answers is correct.
- ○ The bromine vapours that are dragged along during the gas development are dissolved in the water.
- ○ The bromine vapours that are dragged along during the gas development are dissolved in the hydrochloric acid.
- ○ The bromine vapours that are dragged along during the gas development are dissolved in the dichloromethane.

Überprüfen

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Evaluation (4/4)

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Summary of the experiment!

Bromine is polarized and, thereby, _____ by zinc chloride as a _____. It can attach itself in an ionic manner to the toluene nucleus via several complex intermediate stages. Following a dehydrobromination, _____ is formed, i.e. the product of bromination in the _____. In the absence of a catalyst and under the influence of light, however, side chain bromination takes place via radical intermediate stages. The reaction can be controlled in a targeted manner by varying the reaction conditions.

activated
nucleus
bromotoluene
Lewis acid

Überprüfen

Folie	Punktzahl / Summe
Folie 16: Reactifity of Toluol	0/1
Folie 17: Bromine vapors	0/1
Folie 18: Summery of the experiment	0/4

Gesamtsumme 0 / 6

Lösungen Wiederholen

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