Cannizzaro reaction and reaction of benzaldehyde with ethylene glycol

The students learn the cannizzaro reaction and the reaction of benzaldehyde with ethylene glycol.

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/6011a0cfebbd4d000315b7a3

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General information

Application

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Experimental setup

In the first part of the experiment, benzaldehyde disproportionates under the effect of alkalis to alcohol soluble benzyl alcohol and water-soluble benzoic acid that precipitates when the aqueous solution is acidified. In the second part, benzaldehyde reacts with ethylene glycol to form a cyclic acetal. This ethylene acetal is resistant against basic and oxidising reagents.

In an acid medium, it once again splits up into its original products. It is because of these characteristics that cyclic acetals are used for blocking the carbonyl function in preparative, organic chemistry.

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Other information (1/2)

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Prior knowledge

The Students should already be familiar with distillation and microdistillation.

Scientific principle

In the first part of the experiment, benzaldehyde disproportionates under the effect of alkalis to alcohol soluble benzyl alcohol and water-soluble benzoic acid that precipitates when the aqueous solution is acidified. In the second part, benzaldehyde reacts with ethylene glycol to form a cyclic acetal.

Other information (2/2)

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Learning objective

The students learn the cannizzaro reaction and the reaction of benzaldehyde with ethylene glycol.

Tasks

The students show the cannizzaro reaction of benzaldehyde under basic conditions and they prepare benzaldehyde ethylene acetal from benzaldehyde with ethylene glycol

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Safety instructions

- Ethyl alcohol is a highly flammable liquid that can be mixed with water. In combination with air, its vapours may form explosive mixtures.
- Benzaldehyde is a poorly water-soluble, low-flammable liquid with a bitter almond smell. The substance irritates the skin, eyes, and respiratory tract.
- 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

The Cannizzzaro reaction is named after its inventor, the Italian chemist Stanislao Cannizzaro.

Under the effect of alkalis, benzaldehyde disproportionates to alcohol-soluble benzyl alcohol and watersoluble benzoic acid that precipitates when the aqueous solution is acidified.

Due to the reaction with ethylene glycol, benzaldehyde forms a cyclic acetal. This ethylene acetal is resistant against basic and oxidising reagents. In an acid medium, it once again splits up into its original products. It is because of these characteristics that cyclic acetals are used for blocking the carbonyl function in preparative, organic chemistry.

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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	Right angle boss-head clamp	37697-00	3
4	Universal clamp	37715-01	3
5	Support ring, i.d. 70mm,	37721-01	1
6	Test tube rack, wood, for 6 tubes d= 30 mm	40569-10	1
7	Test tube,200x30 mm	36304-01	2
8	Round-bottom flask, 250 ml, GL 25	MAU-27220002	2
9	Water separator GL25/12	35790-15	1
10	Dimroth-Condensor, GL 25/12	MAU-27223500	1
11	Short distillation head, GL 18/8	MAU-27224500	1
12	Adapter for 4 flasks, GL25	MAU-27227500	1
13	Round bottom flask, 100ml, GL 25/12	35841-15	1
14	Round-bottom flask, 50 ml	MAU-27220001	4
15	Lab thermometer,-10..+250C	38065-00	2
16	Secure bottle, 500 ml, 2 x GI 18/8, 1 x 25/12	34170-01	1
17	Spring manometer, 0...-1000 mbar	34170-02	1
18	Glass tubes, right-angled, 10	36701-57	1
19	Stopcock,3-way,t-shaped, glass	36731-00	1
20	Water jet pump, plastic	02728-00	1
21	Heating mantle f. roundbottom flask, 100 ml	49541-93	1
22	Heating mantle f. roundbottom flask, 250ml	49542-93	1
23	Clamp for heating mantle	49557-01	2
24	Power regulator	32288-93	1
25	Graduated cylinder, borosilicate, 25 ml	36627-00	1
26	Beaker, Borosilicate, tall form, 150 ml	46032-00	1
27	Separatory funnel 250 ml pear-sh.	36884-00	1
28	Erlenmeyer flask, borosilicate, wide neck, 250 ml	46152-00	1
29	Funnel, glass, top dia. 80 mm	34459-00	1
30	folded filter,qual.,185 mm,100pcs	47580-05	1
31	Abbe refractometer	35912-00	1
32	Immersion thermostat Alpha A, 230 V	08493-93	1
33	External circulation set for thermostat Alpha A	08493-02	1
34	Bath for thermostat, makrolon	08487-02	1
35	Weighing dishes, square shape, 84 x 84 x 24 mm, 500 pcs.	45019-50	1
36	Hose clip, diam. 8-16 mm, 1 pc.	40996-02	2
37	Rubber tubing, i.d. 6 mm	39282-00	6
38	Rubber tubing,vacuum,i.d.6mm	39286-00	2
39	Spoon, special steel	33398-00	1
40	Wash bottle, plastic, 500 ml	33931-00	1
41	Glass rod,boro 3.3,l=300mm, d=7mm	40485-05	1
42	Pasteur pipettes, 250 pcs	36590-00	1
43	Rubber caps, 10 pcs	39275-03	1
44	Boiling beads, 200 g	36937-20	1
45	Benzaldehyde 500 ml	30036-50	1
46	Ethyl alcohol, absolute 500 ml	30008-50	1
47	Ethylene glycol 250 ml	30085-25	1
48	Toluene 250 ml	30236-25	1
49	Ortho-phosphoric acid 85% 250 ml	30190-25	1
50	Hydrochloric acid 37 %, 1000 ml	30214-70	1
51	Potassium carbonate,98-100% 250 g	30096-25	1
52	Potassium hydroxide pellets,500 g	30103-50	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.

Take the support base and put the two support rods into the base.

Fix the heating mantle on the support rod.

Place the round bottom flask of the destillation apparatus in the heating mantle.

Be careful and wear gloves, goggles and labcoat.

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Procedure (1/2)

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1. CANNIZZARO reaction

- Mix approximately 5 ml of benzaldehyde with 25 ml of a 10% ethanolic potassium hydroxide solution in a test tube. Add a little alcohol, filter the solid substance out, and wash it with a little alcohol. Then, dissolve the solid substance in approximately 25 ml of water and add some hydrochloric acid.

2. Benzaldehyde ethylene acetal

- Dissolve approximately 120 ml of toluene, 10.6 g of benzaldehyde and 7.5 g of ethylene glycol in a 250 ml round bottom flask.
- Add 10 drops of ortho-phosphoric acid and some boiling beads to this mixture.

Procedure (2/2)

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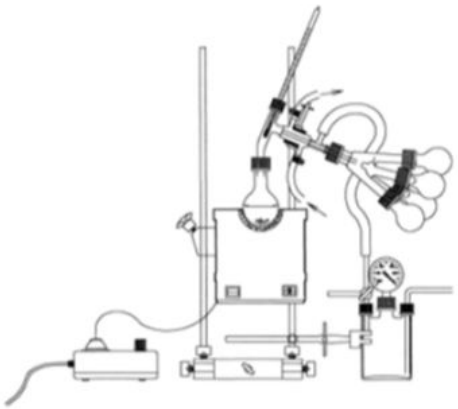
Boil the mixture with a return flow at the water separator until no more water is separated (Fig. right).

When the solution has cooled, wash it with diluted lye and water, separate the organic phase, dry it by way of potassium carbonate and distil it. Under normal pressure, the solvent toluene distils into a 250 ml flask.

Pour the remaining liquid into a 100 ml flask and equip the micro-distillation apparatus with an adapter with four flasks.

Then, distil the product under the full vacuum of the vacuum pump and determine the refractive index of the purified product.



Boil the mixture with a return flow at the water separator until no more water is separated

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Evaluation

Evaluation (1/5)

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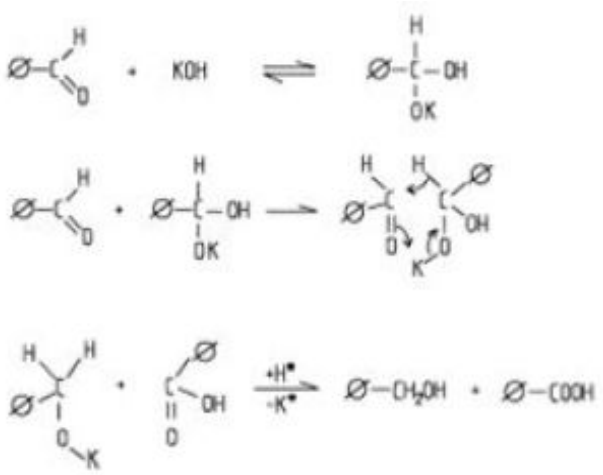
1. CANNIZZARO reaction

Result
The reaction mixture heats before it solidifies. During the acidification, a white substance precipitates from the aqueous solution.

Interpretation
Under the effect of alkalis, benzaldehyde disproportionates to alcohol-soluble benzyl alcohol and watersoluble benzoic acid that precipitates when the aqueous solution is acidified.



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Evaluation (2/5)

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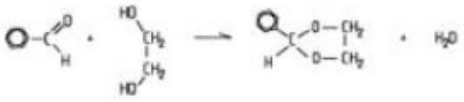
2. Benzaldehyde ethylene acetal

Result
When the solution is boiled with a return flow, approximately 2 ml of water are separated. At a pressure of approximately 20 hPa, the product starts to boil at 110-112°C. The refractive index of the colourless liquid is n = 1.5267 (literature value).

Interpretation
Due to the reaction with ethylene glycol, benzaldehyde forms a cyclic acetal. This ethylene acetal is resistant against basic and oxidising reagents. In an acid medium, it once again splits up into its original products. It is because of these characteristics that cyclic acetals are used for blocking the carbonyl function in preparative, organic chemistry.

Data
The refractive indexes n of the compounds are as follows:
Toluol (toluene): 1.494
Benzaldehyde: 1.546
Ethylene glycol (ethane diol): 1.427
Benzaldehyde ethylene acetate: 1.5267
Molar masses:
Benzaldehyde: 106.13 g/mol
Ethylene glycol: 62.07 g/mol
Boiling point of toluene: 111 °C



Evaluation (3/5)

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What happens during the acidification in the cannizzaro reaction?

- ○ None of the answers is correct.
- ○ During the acidification, a black substance precipitates from the aqueous solution.
- ○ During the acidification, a white substance precipitates from the aqueous solution.
- ○ During the acidification, a highly explosive gas is formed.

Überprüfen

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Evaluation (4/5)

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When does the product of the second part (benzaldehyde ethylene acetal) start to boil?

- ○ At a pressure of approximately 20 hPa, the product starts to boil at 50-60°C because of the ethylene.
- ○ At a pressure of approximately 20 hPa, the product starts to boil at 100°C, like every other liquid substance.
- ○ None of the answers is correct.
- ○ At a pressure of approximately 20 hPa, the product starts to boil at 110-112°C.

Überprüfen

Evaluation (5/5)

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Summary of the experiment

Due to the reaction with ethylene glycol, benzaldehyde forms a _____. This ethylene acetal is resistant against basic and oxidising reagents. In an acid medium, it once again _____ into its original products.
It is because of these characteristics that cyclic acetals are used for blocking the _____ function in preparative, organic chemistry.

carbonyl
splits up
cyclic acetal

Überprüfen

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Folie	Punktzahl / Summe
Folie 15: Acidification	0/1
Folie 16: Boiling	0/1
Folie 17: Summary of the experiment	0/3

Gesamtsumme 0 / 5

Lösungen Wiederholen

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