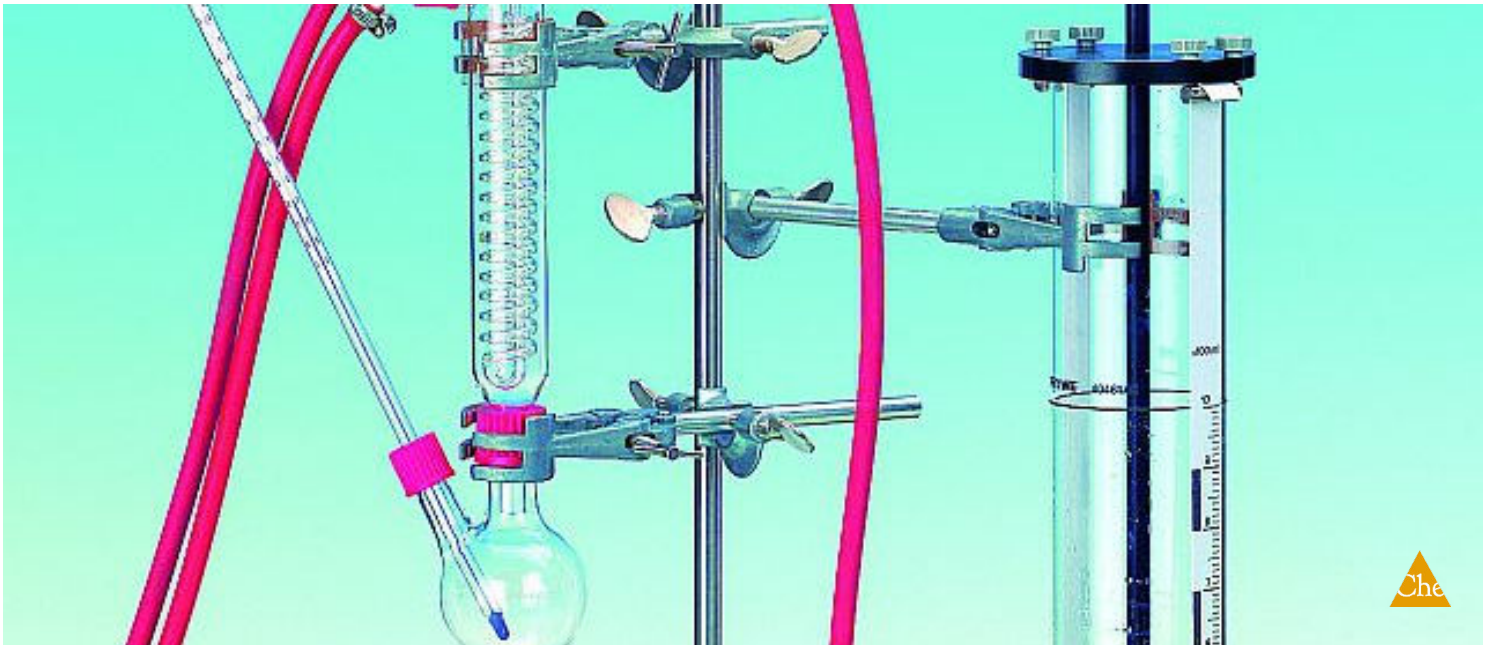
Haloalkanes: Wurtz reaction - lithium organyls



Die Wurtz-Synthese wurde 1854 zur Herstellung höherer Alkane auf Basis von Halogenalkanen entwickelt. Alkyljodide reagieren am einfachsten. Die Reaktion kann am besten mit Lithium gesteuert werden, da die anderen Alkalimetalle viel heftiger reagieren

Chemistry > Organic chemistry > Hydrocarbons

Chemistry > Organic chemistry > Dyestuffs / Household chemistry

Difficulty level	Group size	Preparation time	Execution time
medium	2	10 minutes	10 minutes

Robert-Bosch-Breite 10
37079 Göttingen

Tel.: 0551 604 - 0
Fax: 0551 604 - 107

info@phywe.de
www.phywe.de

General information

Application

Experimental setup

Wurtz's reaction is a coupling reaction. It consists in the reaction of sodium metal with two alkyl halides in the presence of dry ether to form a higher alkane. It also forms a compound that contains sodium and the halogen.

Apart from sodium, metals like silver, indium, zinc and iron can also be used in the reaction in order to obtain alkanes.

This reaction has its limitations. Only symmetric alkanes can be synthesized via this method. There is a side reaction, which produces an alkene. If the alkyl halides are bulky in nature, there will be a greater amount of alkene formed. Methane cannot be synthesized with the Wurtz reaction since the product of an organic coupling reaction must have at least two carbon atoms. The reaction fails with tertiary alkyl halides.

Robert-Bosch-Breite 10
37079 Göttingen
Tel.: 0551 604 - 0
Fax: 0551 604 - 107
info@phywe.de
www.phywe.de

Other information (1/2)

PHYWE excellence in science

Prior knowledge

Unlike the other alkali- organyls, lithium organyls- with the exception of methyllithium- show a stronger covalent behaviour. They dissolve rather well in organic solvents, such as diethyl ether, tetrahydrofuran, and alkanes, and they are relatively stable in these solvents.

Scientific principle

Wurtz Synthesis was developed in 1854 for the preparation of higher alkanes based on haloalkanes. Alkyl iodides react the easiest. The reaction can be controlled best with lithium, since the other alkali metals react much more violently. Wurtz synthesis is often a side reaction that occurs during organometallic conversions.

Other information (2/2)

PHYWE excellence in science

Learning objective

The objective of this experiment is to understand more about the Wurtz synthesis. The students will learn what the alkali- organyls and the lithium organyls are. Another important point of the experiment ist to see and understand that alky-iodides react the easiest. The students will learn that during organometallic conversions, the Wurtz snythesis is a side reaction.

Tasks

- The students will react ethyl iodide with lithium and ethyllithium with ethyl iodide.
- They will investigate the reactions and write their observations on the Protocol.

PHYWE excellence in science

Robert-Bosch-Breite 10
37079 Göttingen

Tel.: 0551 604 - 0
Fax: 0551 604 - 107

info@phywe.de
www.phywe.de

Safety instructions

- For this experiment gloves and glasses are needed.
- For this experiment the general instructions for safe experimentation in science lessons apply.
- For H- and P-phrases please consult the safety data sheet of the respective chemical.

Theory

The Wurtz reaction is named after Charles Adolphe Wurtz. It is a coupling reaction in chemistry, whereby two alkyl halides react with sodium metal in dry ether solution to form a higher alkane.

This reaction is also used to prepare higher alkanes containing even number of carbon atoms.

$$2R-X+2Na- - > R-R+2Na^{+}X^{-}$$

The Wurtz reaction occurs through a free radical mechanism. Side reactions are possible which produce alkene products.

There is a halogen- metal exchange involving the radical species, with carbon- carbon formation ocurring in a nucleophilic substitution reaction.

There are certain limitations that come with the reaction. For example it is intolerant of a range of functional groups. Also because of the side reactions it is not used very often. The reaction is limited when it comes to the synthesis of symmetric alkanes.

Robert-Bosch-Breite 10
37079 Göttingen
Tel.: 0551 604 - 0
Fax: 0551 604 - 107
info@phywe.de
www.phywe.de

Equipment

Position	Material	Item No.	Quantity
1	Retort stand, h = 750 mm	37694-00	1
2	Right angle boss-head clamp	37697-00	3
3	Universal clamp	37715-01	3
4	Two neck round bottom flask, 100 ml	MAU-27100002	1
5	Dimroth-Condensor, GL 25/12	MAU-27223500	1
6	Gasket for GL25, 8mm hole, 10 pcs	41242-03	1
7	Glass tube,right-angled, 10 pcs.	36701-52	1
8	Lab thermometer,-10..+110 °C	38056-00	1
9	Gasometer 1000 ml	40461-00	1
10	Plastic bag w. hose 10 u.	40469-00	1
11	Stopcock,3-way,t-shaped, glass	36731-00	1
12	Graduated cylinder, Borosilicate, 100 ml	36629-00	1
13	Weather monitor, 6 lines LCD	87997-10	1
14	Pinchcock, width 15 mm	43631-15	2
15	Rubber tubing, i.d. 6 mm	39282-00	3
16	Hose clip, diam. 8-16 mm, 1 pc.	40996-02	2
17	Tweezers,straight,blunt, 200 mm	40955-00	1
18	Knife, stainless	33476-00	1
19	Filter paper,580x580 mm,10 sheets	32976-03	1
20	Beaker, Borosilicate, tall form, 150 ml	46032-00	1
21	Glass rod,boro 3.3,l=300mm, d=7mm	40485-05	1
22	Spoon, special steel	33398-00	1
23	Funnel, glass, top dia. 50 mm	34457-00	1
24	Powder funnel, upper dia. 65mm	34472-00	1
25	Ethyl iodide 100 ml	31008-04	1
26	Tetrahydrofuran 1000 ml	31883-70	1
27	Potassium hydroxide pellets,500 g	30103-50	1
28	Lithium metal, bottle w.can, 25 g	31523-03	1

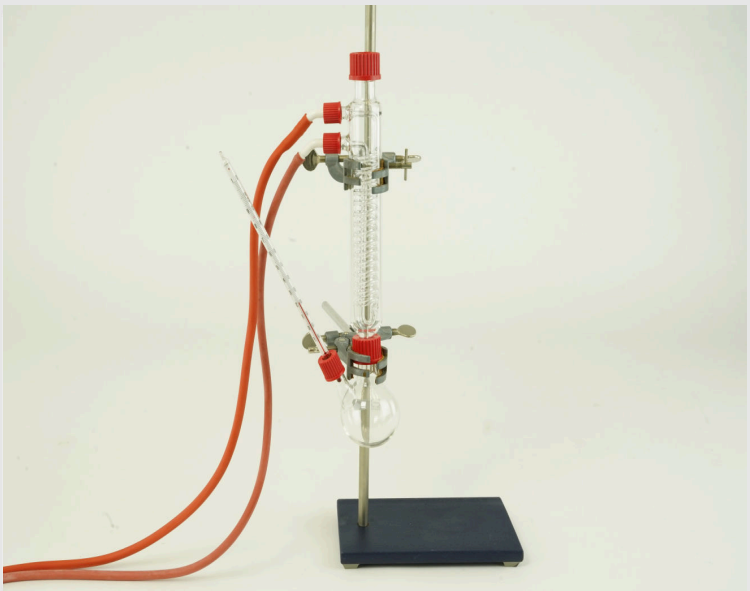
PHYWE excellence in science
Robert-Bosch-Breite 10
37079 Göttingen
Tel.: 0551 604 - 0
Fax: 0551 604 - 107
info@phywe.de
www.phywe.de

PHYWE excellence in science

Setup and procedure

Setup (1/3)

PHYWE excellence in science



Set up the experiment as shown in the figure.

Replace the silicon gasket in the head of the Dimroth condenser with a gasket with an 8 mm hole.

Take the retort stand and fix two clamps on it.

Connect the round bottom flask with the condensor and fix them (with the clamp) on the stand.

Put a lab thermometer in the round bottom flask.

PHYWE excellence in science

Robert-Bosch-Breite 10
37079 Göttingen

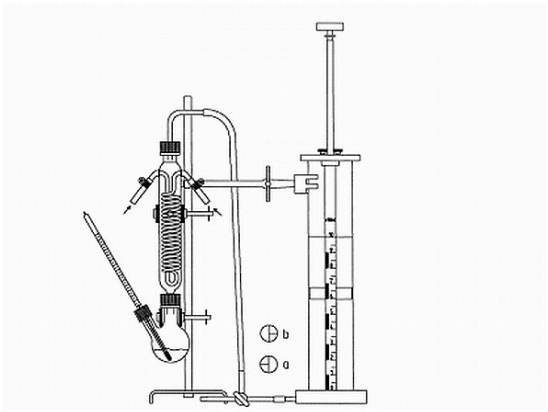
Tel.: 0551 604 - 0
Fax: 0551 604 - 107

info@phywe.de
www.phywe.de

Setup (2/3)

Plase make sure that your experiment setup looks like the picture.

Please make also sure that the position of the stopcock is allwas the same as mentioned in the experiment description.



Experiment setup (dimensional sketch)

Experiment setup (picture)

Setup (3/3)

Drying of tetrahydrofuran

In order to dry the tetrahydrofuran, fill into a bottle and add some potassium hydroxide flakes so that the bottom is covered appproximately 1 to 2 cm high.

Seal the bottle and shake it.

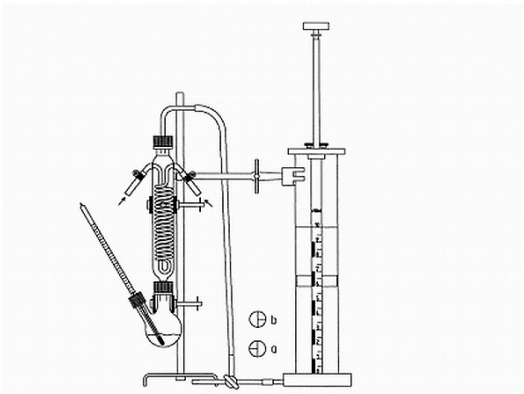
During shaking, open the seal from time to time in order to avoid any excessive pressure inside the bottle.

The potassium hydroxide flakes can remain in the bottle.

Fill the round bottom flask with 10 g of ethyl iodide and 50 ml of dried tetrahydrofuran.

Robert-Bosch-Breite 10
37079 Göttingen
Tel.: 0551 604 - 0
Fax: 0551 604 - 107
info@phywe.de
www.phywe.de

Procedure (1/2)



Positioning of the stopcock

Add 3 to 4 pea-sized, decrusted lithium chunks to the tetrahydrofuran and ethyl idodide solution and switch the water cooling system on.

The gas that is produced must now push the air out of the apparatus (stopcock in position a, Fig. left) before the threeway stopcock towards the gasometer can be opened (position b).

In the gasometer and at room temperature, collect a gas quantity that corresponds to one litre of gas at 1013 hPa and 0°C (see the operating instructions of the gasometer).

Close the connection to the gasometer (stopcock in position a, Fig. left) and disconnect the hose that leads to the apparatus.

Procedure (2/2)

Setup of the gasometer

Make shure that the connection to the gasometer is closed.

Push the hose of the plastic bag, whose empty weight is known, over the hose connector of the three-way stopcock.

Push the gas out of the gasometer and into the plastic bag.

Seal the supply hose by way of a pinchcock and weigh the filled bag.

Robert-Bosch-Breite 10
37079 Göttingen
Tel.: 0551 604 - 0
Fax: 0551 604 - 107
info@phywe.de
www.phywe.de

PHYWE excellence in science

Evaluation

Evaluation (1/3)

PHYWE excellence in science

Lithium reacts with ethyl iodide to ethyllithium and lithium iodide.

As an anion, the lithium- organic compound engages in a nucleophilic manner with the positively charged carbon atom of the ethyl iodide.

This leads to the formation of n- butane and lithium iodide.

PHYWE excellence in science

Robert-Bosch-Breite 10
37079 Göttingen

Tel.: 0551 604 - 0
Fax: 0551 604 - 107

info@phywe.de
www.phywe.de

Evaluation (2/3)

PHYWE excellence in science

Complete the reactions

$2Li+$ [ ] $->C_2H_5Li+$ [ ]

$CH_3CH_2^-Li^+ +$ [ ] $->$ [ ]
$+LiI$

Check

LiI

C_2H_5I

$CH_3CH_2CH_2CH_3$

$CH_3CH_2^+I^-$

Evaluation (3/3)

PHYWE excellence in science

Lithium reacts with ethyl iodide to...

ethyllithium and elementary lithium

ethyllithium and lithium iodide

PHYWE excellence in science

Robert-Bosch-Breite 10
37079 Göttingen

Tel.: 0551 604 - 0
Fax: 0551 604 - 107

info@phywe.de
www.phywe.de

Slide	Score/Total
Slide 16: Reaction scheme	0/4
Slide 17: Summary of the experiment	0/3

Total Score 0/7

Show solutions Retry

PHYWE excellence in science
Robert-Bosch-Breite 10
37079 Göttingen
Tel.: 0551 604 - 0
Fax: 0551 604 - 107
info@phywe.de
www.phywe.de