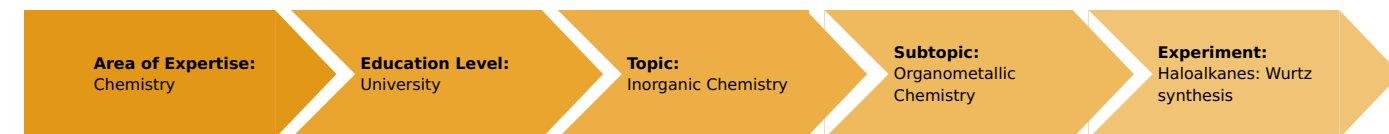


# Haloalkanes: Wurtz synthesis (Item No.: P3101100)

## Curricular Relevance



### Difficulty



Intermediate

### Preparation Time



10 Minutes

### Execution Time



10 Minutes

### Recommended Group Size



2 Students

### Additional Requirements:

- Precision balance, 620 g / 0.001 g

### Experiment Variations:

### Keywords:

Alkali-organyls, Lithium organyls, Wurtz synthesis, Organometallic compounds

## Task and equipment

### Introduction

#### Principle

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.

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.

### Safety instructions



Lithium reaction violently or even in an explosive manner with numerous inorganic and organic compounds, in particular with oxidising agents and halogenated hydrocarbons. Contact with moist skin and mucous membranes leads to severe chemical burns.

**First aid:** Clean the affected skin areas and clothes by first drying and then rinsing with plenty of water. In the event of contact with the eyes, rinse eyes with the lid gap wide open thoroughly with plenty of water.

**Disposal:** Immerse any metal residues (also those on the knife, tweezers, etc.) into isopropyl alcohol, let them react, and add water. In the case of large amounts, work in batches and, if necessary, cool the beaker with the reaction solution from the outside with ice. Remove all sources of ignition (hydrogen formation). Solutions must be neutralised and flushed away.

Water-free potassium hydroxide has a strong irritating effect on the skin, eyes, and mucous membranes. Dusts and mists irritate the respiratory organs. Chemical burns lead to the destruction of the tissue and intense pain.

Tetrahydrofuran is a highly flammable liquid that can be mixed with water. In combination with air, its vapours may form explosive mixtures. Tetrahydrofuran irritates the skin, eyes, and respiratory tract. Cases of systemic poisoning are not known.  
*First aid:* Rinse the affected skin areas and eyes with the lid gap wide open thoroughly with plenty of water.

*Disposal:* Solutions must be diluted with water, neutralised (pH 6-8), and flushed away.

Halogen-containing compounds and solutions must be collected separately from other organic substances in their own collecting vessel that is marked accordingly.

## Potassium hydroxide

H290: May be corrosive to metals.

H302: Harmful if swallowed.

H314: Causes severe skin burns and eye damage.

P280: Wear protective gloves/protective clothing/eye protection/face protection.

## Ethyl iodide

H226: Flammable liquid and vapour

H315: Causes skin irritation.

H335: May cause respiratory irritation.

P210: Keep away from heat, hot surfaces, sparks, open flames and other ignition sources. No smoking.

## n-butane

H220: Extremely flammable gas.

P210: Keep away from heat, hot surfaces, sparks, open flames and other ignition sources. No smoking.

## Tetrahydrofuran

H225: Highly flammable liquid and vapour.

H302: Harmful if swallowed.

H351: Suspected of causing cancer.

EUH019: May form explosive peroxides.

P210: Keep away from heat, hot surfaces, sparks, open flames and other ignition sources. No smoking.

P233: Keep container tightly closed.

## Lithium

H260: In contact with water releases flammable gases which may ignite spontaneously.

H314: Causes severe skin burns and eye damage.

EUH014: Reacts violently with water.

P280: Wear protective gloves/protective clothing/eye protection/face protection.

## Lithium iodide

H315: Causes skin irritation.

H319: Causes serious eye irritation.

P305+351+338: IF IN EYES: Rinse cautiously with water for several minutes. Remove contact lenses if present and easy to do – continue rinsing.

## Equipment

Position No.	Material	Order No.	Quantity
1	Retort stand, h = 750 mm	37694-00	1
2	Right angle boss-head clamp	37697-00	3
3	Universal clamp	37715-00	3
4	Round bottom flask, 100ml, GL25/12, GL18/8	35842-15	1
5	Condenser, Dimroth type GL25/12	35815-15	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..+100 °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 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, high, BORO 3.3, 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. 55 mm	34457-00	1
24	Powder funnel, upper dia. 65mm	34472-00	1
25	Ethyl iodide 25 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

## Task

Investigate the reaction of ethyl iodide with lithium and the following reaction of ethyllithium with ethyl iodide.



## Set-up and procedure



The experiment is set up as per Fig. 1. Replace the silicon gasket in the head of the Dimroth condenser with a gasket with an 8 mm hole.

In order to dry the tetrahydrofuran, fill it into a bottle and add some potassium hydroxide flakes so that the bottom is covered approximately 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 (You can also fill the potassium hydroxide flakes into the original bottle. It is in this way that you will always have dried tetrahydrofuran.). Fill the round bottom flask with 10 g of ethyl iodide and 50 ml of dried tetrahydrofuran.

Add 3 to 4 pea-sized, decrusted lithium chunks to the tetrahydrofuran and ethyl iodide 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. 2) before the three-way 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).



Fig. 1

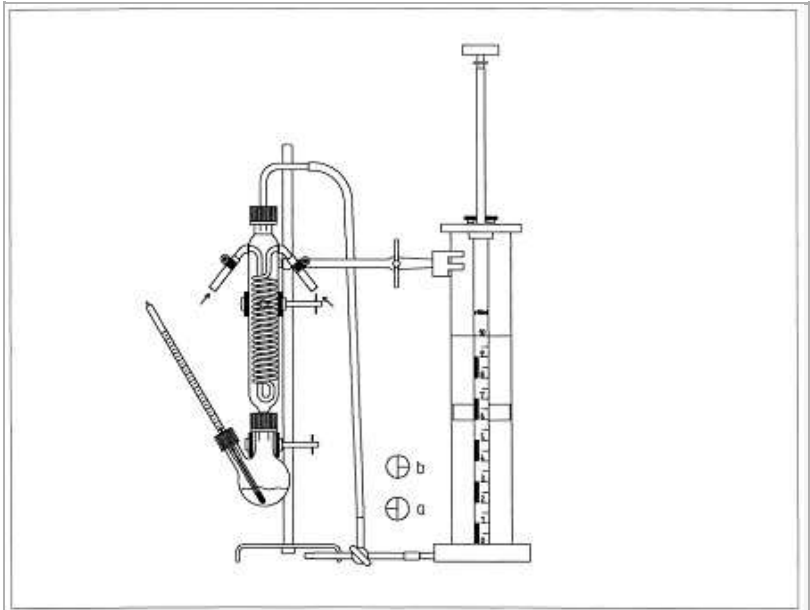


Fig. 2

Close the connection to the gasometer (stopcock in position a, Fig. 2) and disconnect the hose that leads to the apparatus. Then, push the hose of the plastic bag (shorten the hose beforehand to 10 cm), whose empty weight is known, over the hose connector of the three-way stopcock (Fig. 3). 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.

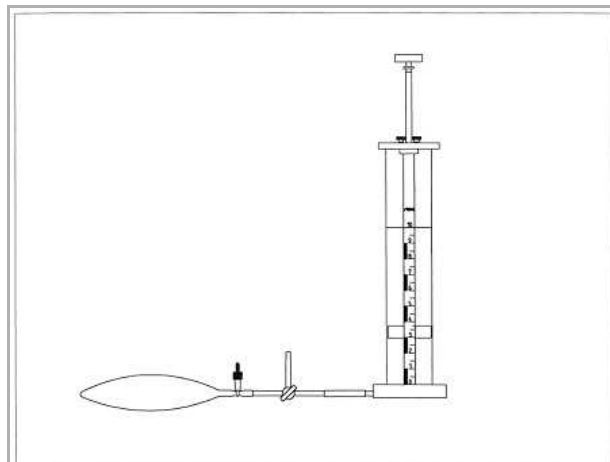
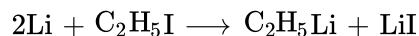


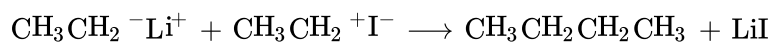
Fig. 3

## Results and evaluation

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.



### Note

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.

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.