

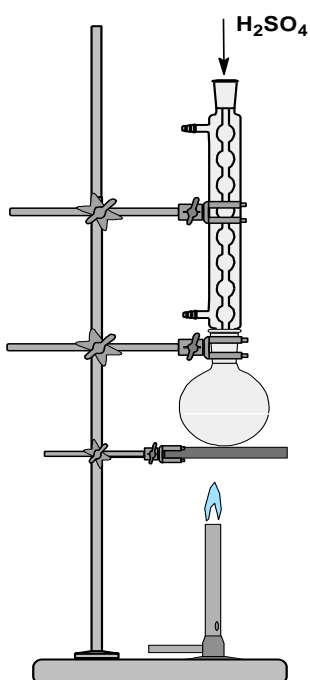
LABORATORY WORK NO.30
PREPARATION OF HALOGENDETRIVATIVES

- **PRINCIPLE :** Halogenderivatives of hydrocarbons can be prepared by substitution reaction from alcohols. The halogenating agent is prepared directly in the reaction mixture from an alkali halogenide and a strong mineral acid .
Butylbromide is prepared (in a laboratory) by reacting hydrogen bromide HBr with butanol under the action of concentrated sulfuric acid to an alkali bromide (e.g. KBr).

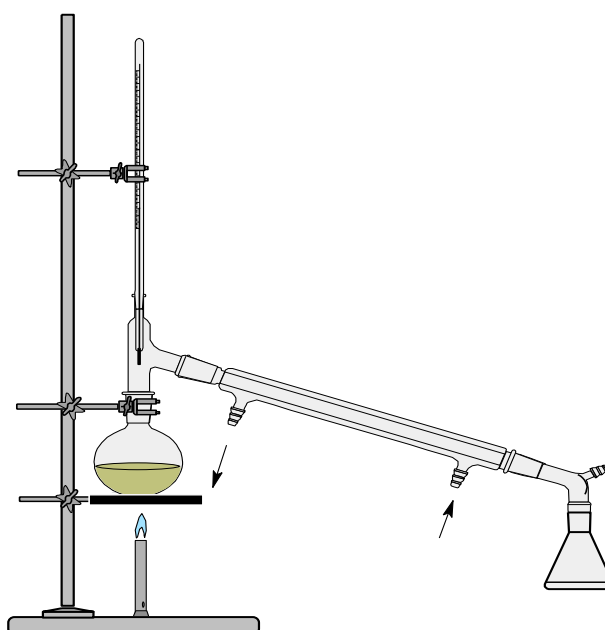
TASK 1. PREPARATION OF BUTYL BROMIDE FROM BUTYL ALCOHOL

- **CHEMICALS:** KBr, butan-1-ol (n-butanol), conc. H_2SO_4 , $CaCl_2$, saturated solution of Na_2CO_3 , phenolphthalein, distilled water
- **AIDS:** distillation apparatus with a ball condenser and a distillation flask 500 ml, distillation apparatus with the Liebig condenser and a fractional flask 500 ml, 250 ml Erlenmeyer flask , graduated cylinder 50 ml (2 pcs), separation funnel 250 ml, 50 ml beaker (2 pcs), 250 ml beaker, funnel, lab spoon, burner or heating mantle, thermometer
- **DRAWING OF THE APPARATUS:**

Preparation of butylbromide



Distillation of the product





■ PROCEDURE :

1. Make up the distillation apparatus with a ball condenser as shown in the picture .
2. Weigh out (on the analytical scales) 38 g of KBr and 0.5 mol of butanol into the 50 ml beakers. Measure out 35 ml of distilled water in the graduated cylinder.
3. Prepare a mixture of KBr, butanol and distilled water into the distillation flask . Pour slowly 35 ml of concentrated H_2SO_4 through the upper end of the cooler and shake gently the flask.
4. Start cooling with water and boil the mixture under the reflux condenser for about 60 min. Observe the reflux of the solvent.
5. After slight cooling, pour the reaction mixture into the side-arm flask, distill and collect it through the Liebig condenser into the receiver with 25 ml of distilled water. Observe the look of the distillate.
6. Finish the distillation after 15 minutes (when only clean fluid is distilled).
7. Separate the distillate in the separation funnel from the upper layer of water. Pour again the bottom layer into the separation funnel and add 12 ml of conc. H_2SO_4 to remove undesired by-products (but-1- en and dibutylether) .
8. Shake the mixture and then separate the bottom layer with H_2SO_4 .
9. Rinse (by small amounts) the upper layer of butylbromide by 30 ml of saturated Na_2CO_3 solution coloured by a drop of phenolphthalein. Discolouration indicates the neutralization of the solution. Be careful – the overflowing H_2SO_4 in the separation funnel reacts violently!
10. Add again 15 ml of distilled water, shake it and separate the water layer (lower) from butyl bromide (upper layer).
11. Pour the layer of butylbromide into a small beaker, add a teaspoon of anhydrous CaCl_2 for drying and leave the product till the next laboratory work.
12. If you have time enough, the second option is possible - distill the product once again at temperature of 90-110 °C and weigh the obtained product, e.g. purified butylbromide.

- **CONCLUSION:** Evaluate the result of the work and describe the characteristics of the product.



STUDENT'S SHEET No. 30 PREPARATION OF HALOGENDETIVATIVES

1. Vocabulary:

Translate the vocabulary and learn it:

- | | |
|----------------------|-----|
| a) alkalický bromid | 1. |
| b) kuličkový chladič | 2. |
| c) odbarvení | 3. |
| d) nadbytek | 4. |
| e) vedlejší produkt | 5. |
| f) nežádoucí | 6. |
| g) přečištěný | 7. |
| h) protřepat | 8. |
| i) získaný | 9. |
| j) oddělit | 10. |

2. Complete the crossword.

Find the following words: BUTYLBROMID, HORNÍ VRSTVA, DOLNÍ VRSTVA, DĚLICÍ NÁLEVKA, NALIJ, DESTILUJ

O	T	J	B	W	R	U	R	G	U	J	P	O
D	A	S	B	A	G	M	G	A	P	I	O	L
I	L	R	U	P	P	E	R	L	A	Y	E	R
S	U	L	T	C	X	L	L	U	L	N	K	I
T	M	P	Y	S	E	D	I	M	E	N	T	J
I	I	H	L	G	S	P	P	O	U	R	A	R
L	N	U	B	M	E	O	H	I	J		T	H
L	I	R	R	E	T	H	A	N	E	P	L	G
H	U	L	O	W	E	R	T	C	M	I	E	F
N	M	H	M	H	I	L	I	E	T	P	N	D
I	O	O	I	A	P	A	C	R	N	E	N	S
K	X	L	D	N	L	Y	E	L	R	T	U	A
O	I	D	E	E	O	E	D	T	F	T	F	Y
L	D	S	E	P	A	R	A	T	I	O	N	Q
P	E	R	E	M	J	T	X	E	E	U	E	W
Z	W	A	T	E	R	B	O	N	D	U	G	E

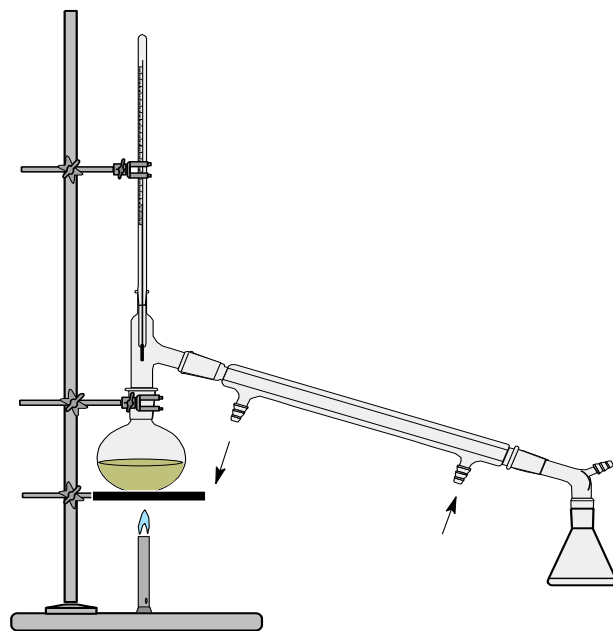
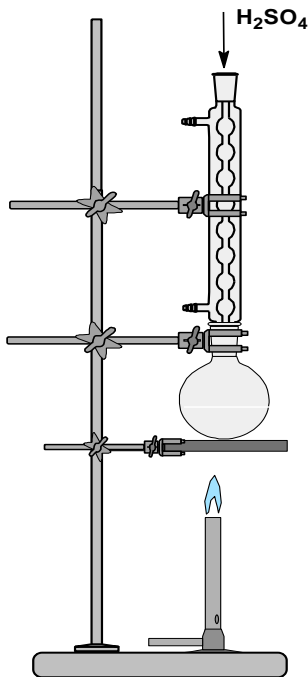
3. Fill the words in gaps:

cooling, receiver, Evaluate, water, flask, look, end

After slight, pour the reaction mixture into the side-arm,
distill and collect it through the Liebig condenser into the (at the
.....of the apparatus) with 25 ml of distilled Observe theof
the distillate.

..... the result of the work and describe the characteristics of the product.

4. Describe as many laboratory items as you know:



5. Underline the obtained product and write the structure and summary formulas for all derivatives:

1. Methylbromide

2. Ethyl bromide

3. Propylbromide



INVESTICE DO ROZVOJE VZDĚLÁVÁNÍ

4. Butylbromide