



Organic Chemistry Experiment

Synthesis of 1-bromobutane

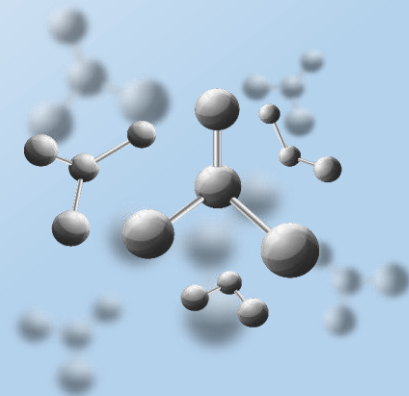
Chemistry teaching experiment
center of Fudan university





Experimental purpose

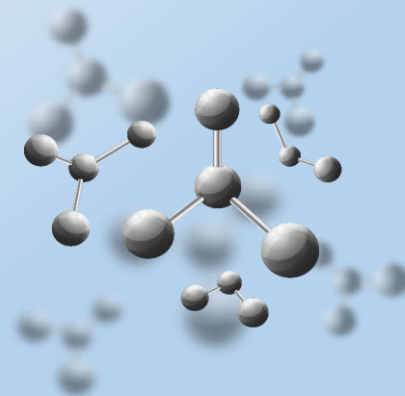
1. Learn how to install gas absorption device
2. Master the method of extracting, washing and separating liquid with a separating funnel
3. Master the method of drying liquid organic compounds with desiccant
4. Synthesis of 1-bromobutane





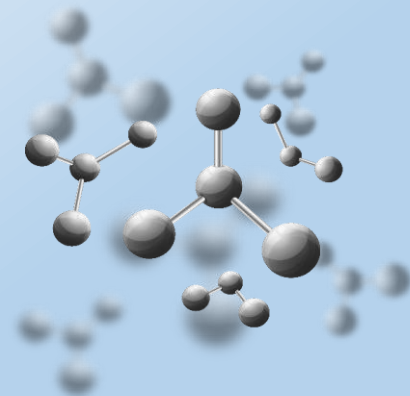
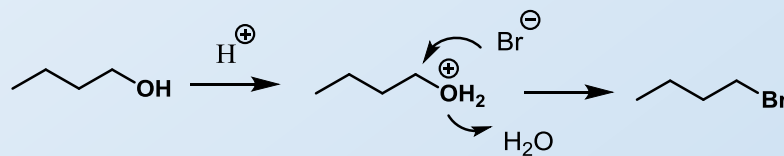
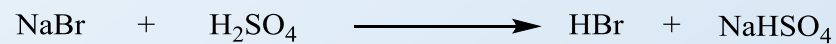
experimental background information

1-Bromobutane is a chemical with the formula C_4H_9Br . Colorless transparent liquid, insoluble in water, slightly soluble in carbon tetrachloride, soluble in chloroform, mixed soluble in ethanol, ether, acetone. It can be used as alkylation agent, solvent, rare element extraction agent and for organic synthesis.





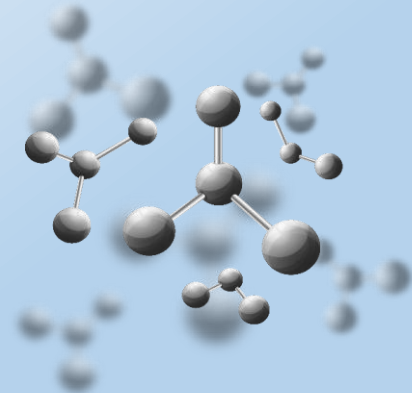
Experiment principle





Experiment noun

1. Reflux Condensing
2. Gas Absorption
3. Extraction and Washing
4. Simple Distillation
5. Fine Distillation





experimental medicine

Sodium Bromide FW 102.89

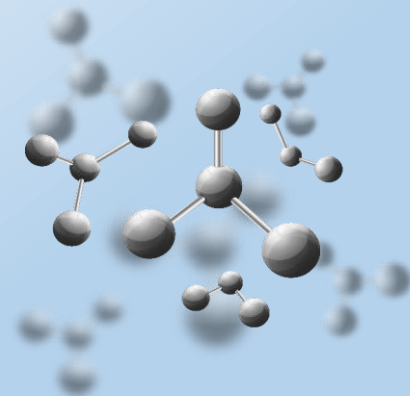
Concentrated Sulfuric Acid FW 98 *d* 1.84

n-Butyl Alcohol FW 74.12 *bp* 117.7 °C *d* 0.810

Ethanol FW 46.07 *bp* 78.3°C *d* 0.789

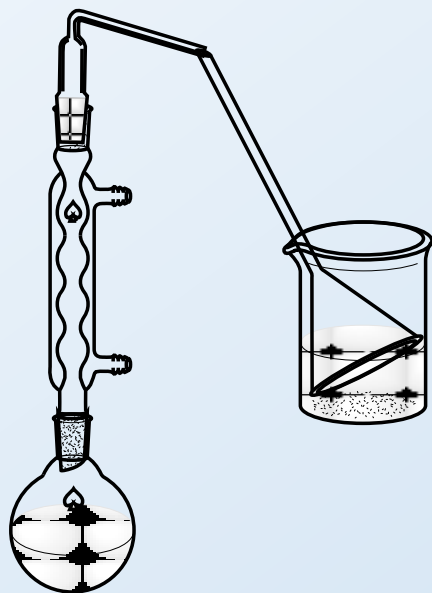
Sodium Bicarbonate FW 84.01

Calcium Chloride Anhydrous FW110.98

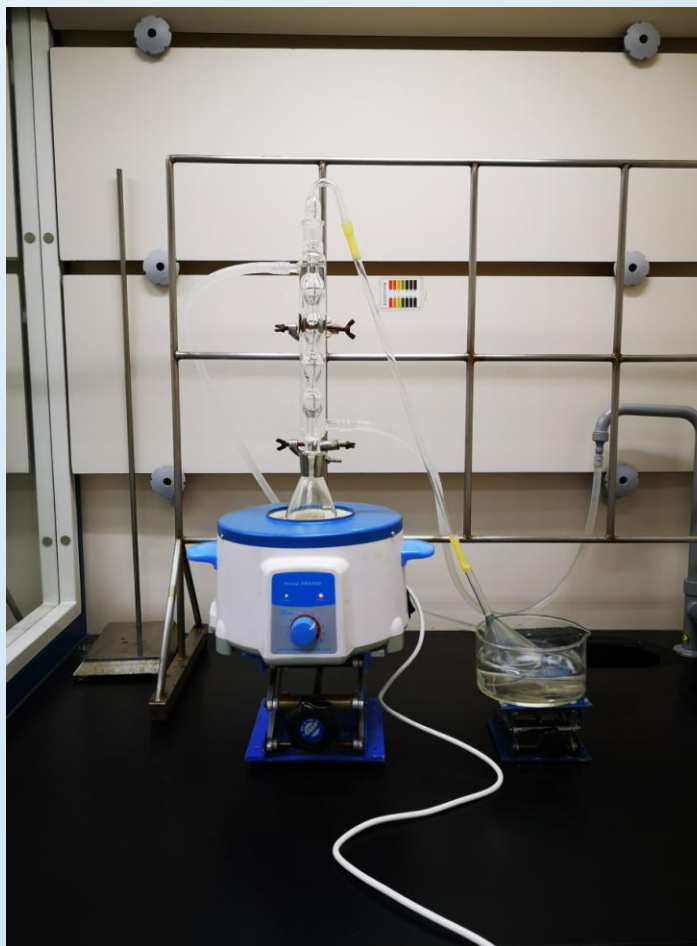




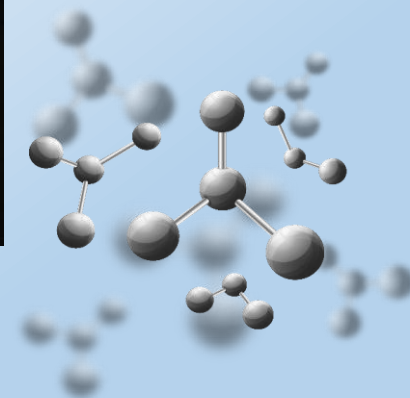
Experimental content. reflux, gas absorption device



reflux, gas
absorption device

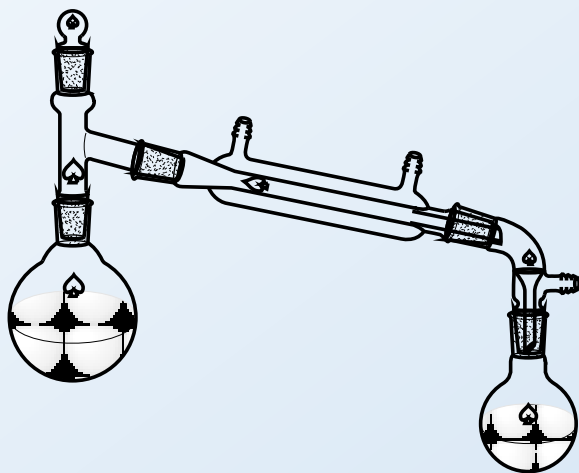


reflux, gas
absorption device

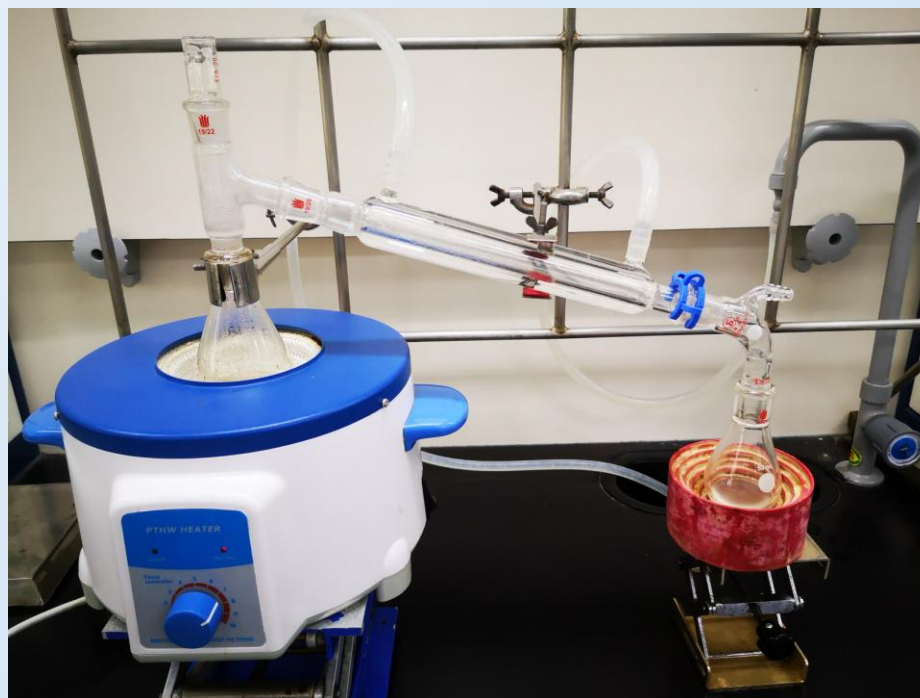




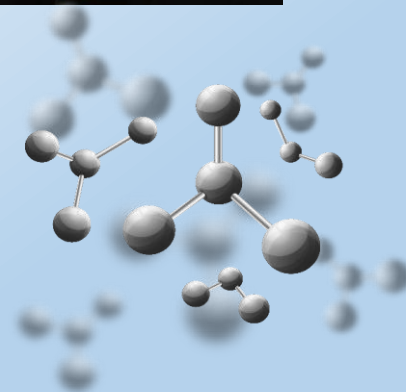
Experiment content. crude distillation



crude distillation



crude
distillation

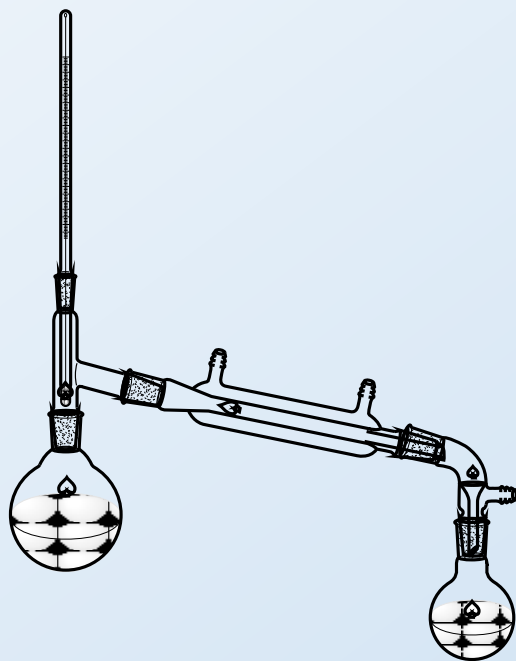




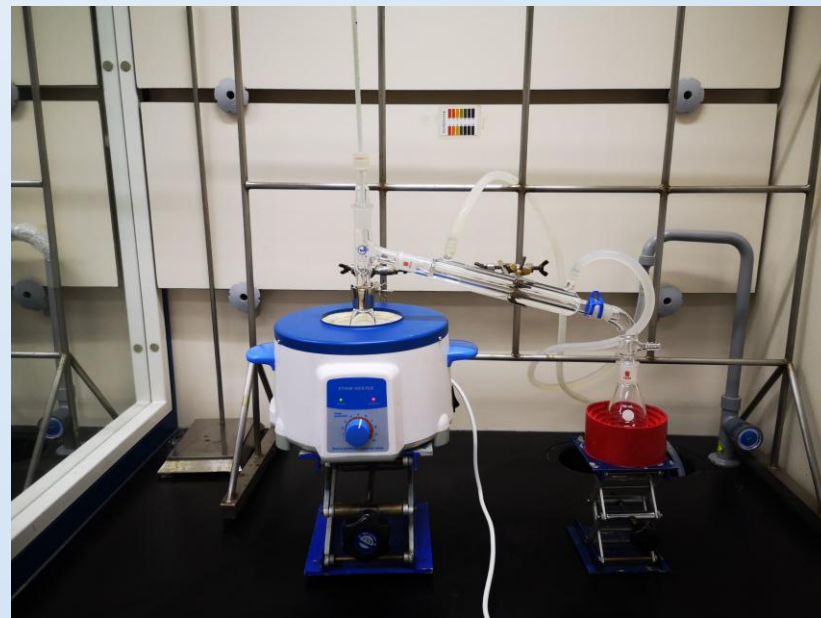
Experimental content. extraction, fine distillation



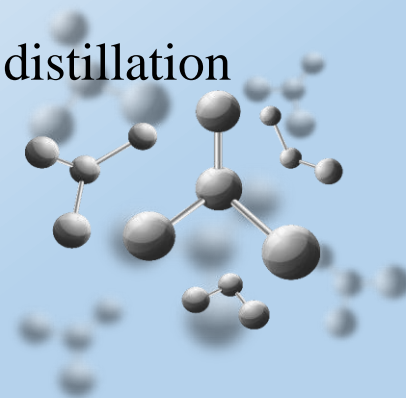
extraction



fine distillation



fine distillation





Experimental procedure

1. Reflux reaction

10 mL of water, 10 mL of concentrated sulfuric acid, 8 mL of n-butanol and 11.5 g of sodium bromide, two zeolites were added to a 100 mL round-bottom flask and reflux for 0.5 h.

2. Simple Distillation

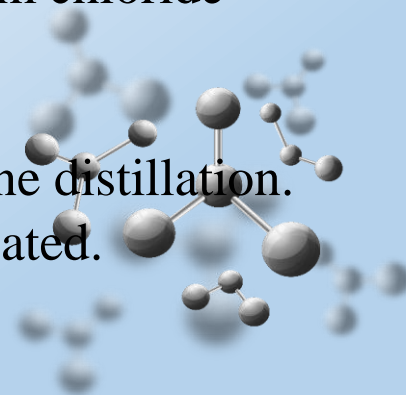
Replace it with a distillation unit. Until the distillate is free of oil droplets or clarified.

3. Extraction and drying

The crude distillation products were washed with 10 mL of water and 10 mL of concentrated sulfuric acid, and then washed with water, sodium dicarbonate aqueous solution and water sequentially. The calcium chloride anhydrous was added.

4. Fine Distillation

The dried product is filtered into a dry round bottom flask for fine distillation. Fractions of 99~103°C were collected, weighed and yield calculated.





Notes

1. The gap between the funnel and the water in the gas absorption device should be well controlled.
2. The adding sequence of concentrated sulfuric acid and water cannot be changed, concentrated sulfuric acid must be slowly dropped into the water.
3. After adding sodium bromide, use paper towel to wipe the bottle mouth powder to ensure air tightness.
4. Control the reaction rate in the reflux reaction。
5. Judge whether or not the product is completely steamed in simple distillation, two or three drops of distillate to the beaker containing water, and observe whether it is stratified
6. The distillation unit shall be built and dismantled in order.
7. During the extraction process, it is necessary to check the leakage of the separation funnel. During the extraction process, open the cock and let off air in time according to the standard operating posture.
8. The distillation before judging whether the thermometer is constant, you can catch the product。

