



Organic Chemistry Experiment

Extract caffeine from tea

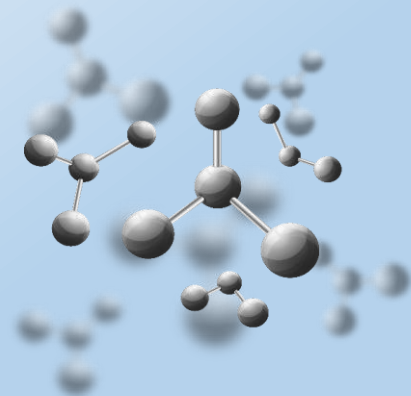
Experimental center for chemical education





Experimental purpose

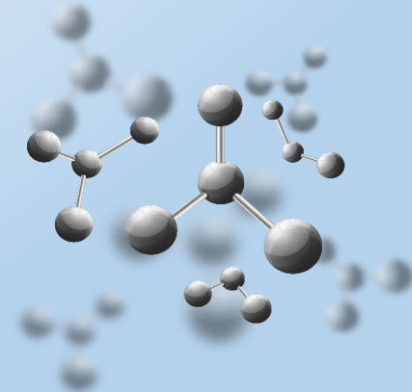
- Learn to extract caffeine from tea by solid-liquid extraction and sublimation
- Master the use of Soxhlet extractor
- Master the operation of distillation and sublimation devices





Background introduction

Tea contains a variety of alkaloids, including caffeine about 1%-5%, tannic acid (tannic acid) about 11%-12%, as well as cellulose, flavonoid pigments, proteins...

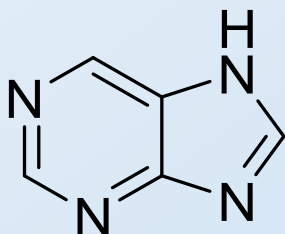




Background introduction

- Caffeine is a derivative of purine, which is **weakly alkaline** and has the effects of stimulating the heart, exciting the brain and diuresis.
- Pure caffeine is **white needle crystal**, tastes bitter, and it is soluble in water, ethanol, dichloromethane.
- The melting point of caffeine is 238°C:

When the caffeine containing crystal water is heated to 100 °C, the crystal water is going to lose and sublimation will begin. The sublimation will be more remarkable at 120 °C, and if the temperature is 178 °C or higher, the sublimation process will be very fast.

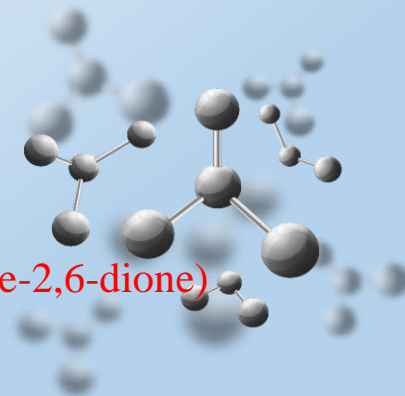


purine



Caffeine

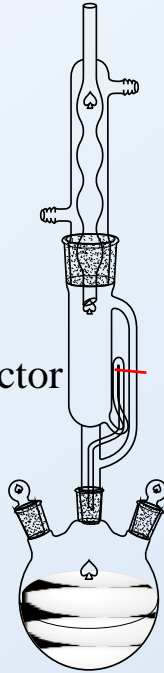
(3,7-Dihydro-1,3,7-trimethyl-1-H-purine-2,6-dione)





Background introduction

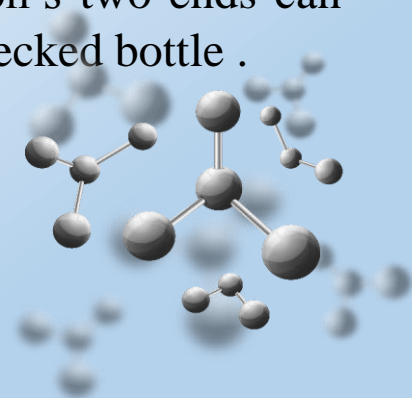
- The extraction of caffeine from tea can be done by soaking method. Soaking method is a liquid-solid extraction. In the laboratory, a Soxhlet extractor can be used for such extraction.
- The Soxhlet extractor for solid-liquid extraction involves three key points: **the solvent reflux, dissolution and siphon**, and these allow the solid tea to be extracted by a pure solvent **each time**.
- The specific process is: heating the three-necked bottle will volatilize the solvent, and the solvent vapor will meet the condensate and drop back to the Soxhlet extractor, which can dissolve the caffeine from the tea. When the liquid level of Soxhlet extractor exceeds the height of the siphon, pressure caused by different liquid levels of the siphon's two ends can make the solvent sucked back into the three-necked bottle .



Soxhlet extractor

Siphon

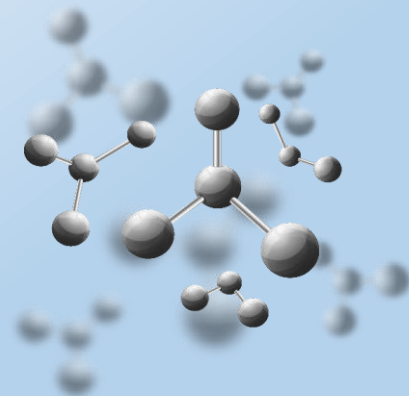
Extraction device





Reagents and physical constants

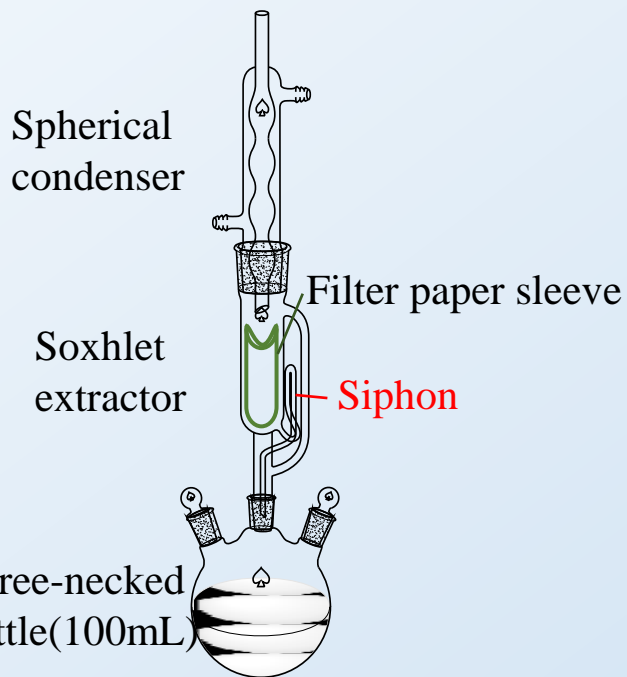
- Tea powder
- 95% ethanol
- caffeine (FW 194.19, mp 234-236.5°C)



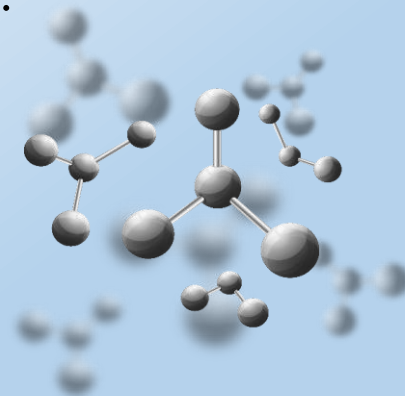


Experimental section

Put 10g tea powder into the **folded filter paper sleeve**, and then put the sleeve into the Soxhlet extractor. Set up the extraction device from bottom to top, weigh 80mL 95% ethanol, add a half to the fat extractor (the liquid level can not exceed the highest point of the siphon), the rest is added to the three-necked bottle. Heat the three-necked bottle to make the ethanol reflux, keep continuous extraction until the color of the extract in the siphon became very shallow. The extraction time was about 1.5 hours.



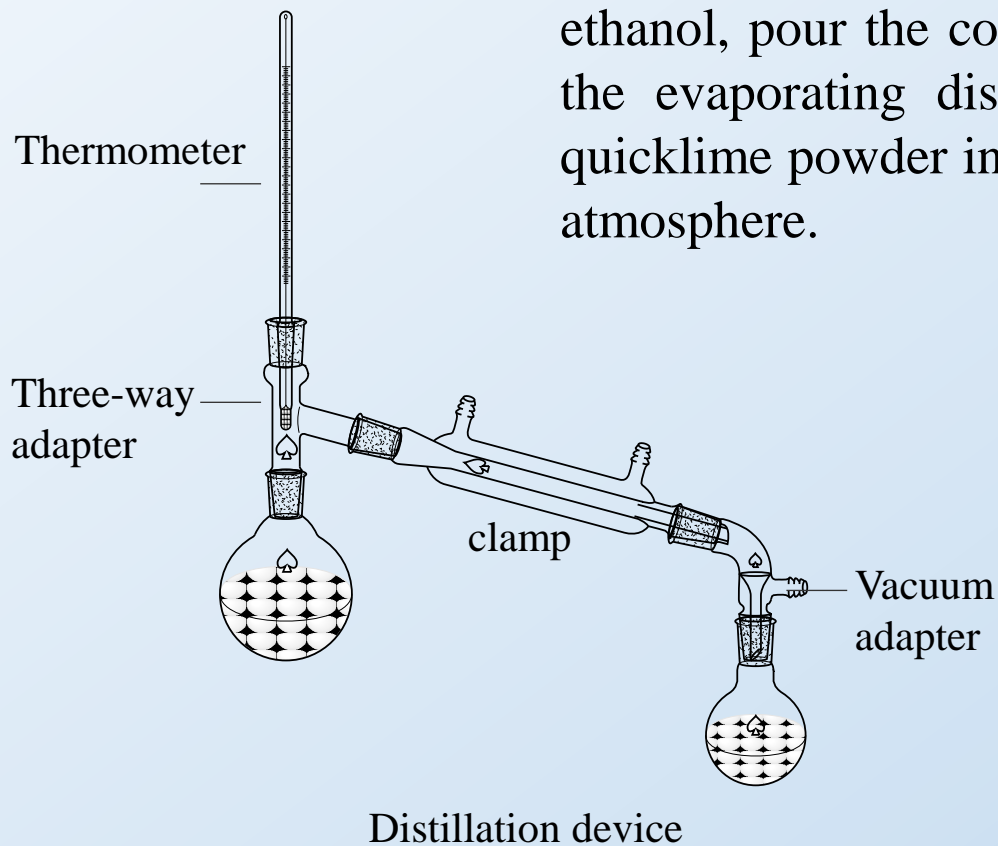
Extraction device





Experimental section

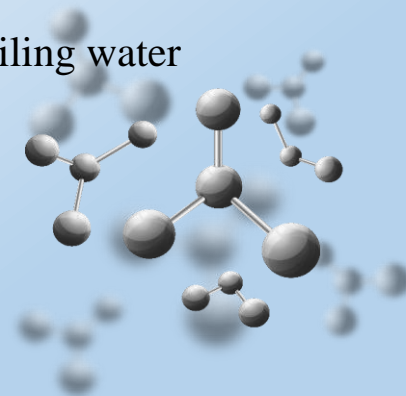
At the end of the extraction, first stop the heating, remove the extraction device after cooling for a while, change it to a distillation device, distill off most of the ethanol, pour the concentrated liquid in the bottle into the evaporating dish immediately, and add 5-6g of quicklime powder into it. Stir-fry in a water steam bath atmosphere.



Water steam bath

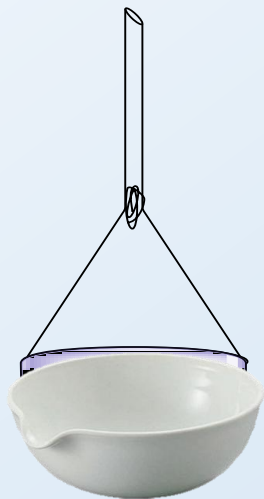
Evaporating dish

Boiling water



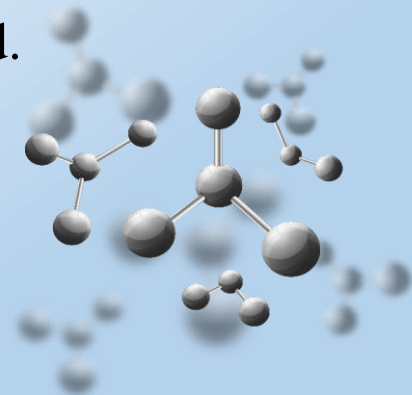


Experimental section



Sublimation device

Use a piece of filter paper with many small holes to cover and seal the evaporating dish, and cover a suitable funnel on it to set up the sublimation device. Heat to sublime. Until white smoke appears in the funnel and there is white needle crystal on the filter paper, stop heating, naturally cool to 100°C . Remove the funnel, gently peel off the filter paper, and use a spatula to gently scrape the crystal on the filter paper. Next mix the residue, use a higher temperature to sublime again. The collected caffeine of two sublimations should be combined and weighed.





Precautions & Tips

- The device should be fixed with clips , kept vertical, and sealed to prevent steam from escaping;
- The size of the filter paper sleeve should be moderate (the bottom size is 2.3cm*2.3cm), when put it into Soxhlet extractor, be careful not to block the edge of the siphon tube, and the height of the tea leaves in the sleeve should not exceed the siphon;
- The ethanol in the bottle can't be distilled too dry, otherwise the residual liquid is too sticky to be poured out;
- Stir fry as much as possible, otherwise it will affect the quality of the product;
- The temperature of sublimation is the key point. If the temperature is too low, no caffeine will sublime; If the temperature is too high, the caffeine will be coked, which directly affects the yield and quality of the caffeine crystal.

