

Isolation of caffeine from tea leaves

Materials:

black tea
calcium carbonate
methylene chloride
isopropanol
hexane
magnesium sulphate

Methods:

extraction
melting point analysis

Theory:

1. Caffeine. Structure and properties.
2. Isolation of natural products.
3. Theory and practice of extraction. Continuous extraction.
4. Identification of chemical compounds: melting point determination, chromatography, specific reactions, derivative synthesis.

Introduction:

1. Draw the structure of caffeine. Write the systematic name for caffeine.
2. Which methods could be used for caffeine isolation?
3. Select solvents which could be used for extraction of aqueous solution: benzene, diethyl ether, acetic acid, THF, methylene chloride, chloroform.
4. Find the parameters which characterize pure caffeine. How to measure/check these parameters?

Experiment:

A sample of crushed black tea leaves (50g) in Erlenmeyer flask is soaked in water (500 ml). Powdered calcium carbonate is added to the flask and the mixture is brought to boiling (heating mantle). The gentle boiling is continued for 20 minutes and the hot solution is filtered under reduced pressure on Buchner funnel (two discs of filtering paper and 1 cm Celite layer). The residue is rinsed with hot water (twice, 50 ml).

The filtrate is cooled to room temperature and extracted with methylene chloride (four times, 30 ml). Vigorous shaking should be avoided to prevent emulsion formation.

Combined organic extracts are dried over magnesium sulfate, filtered through filtering funnel (fluted filter paper) in into round bottom flask (250 ml). Rinse the drying agent with methylene chloride (20 ml). Concentrate the combined filtrates using vacuum rotary evaporator, transfer the residue to small (50 ml) round bottom flask and evaporate to dryness using vacuum rotary evaporator.

The residue is dissolved in isopropanol (reflux condenser required) and hexane is added to the boiling mixture. Caffeine crystals form during cooling of the mixture. The collected crystals are dried and characterized. The melting point of caffeine is 236°.

Report:

1. Briefly describe the experiment.
2. Calculate the isolation yield of caffeine.
3. Compare the melting point of caffeine with literature data.
4. Discuss other methods for purification and identification of caffeine.

The proper safety measures have to be observed (personal protection, MSDS)

All experiments have to be carried out in fume hood.

All organic solvent waste has to be disposed into special container.