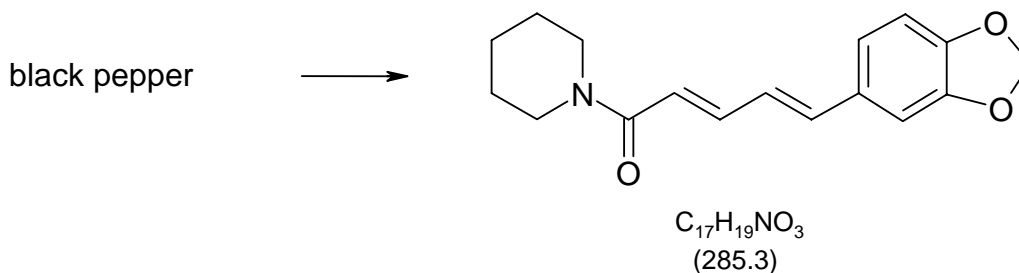


1022 Isolation of piperine from black pepper



Classification

Reaction types and substance classes

isolation of natural product

natural product

Work methods

extracting with Soxhlet extractor, stirring with magnetic stir bar, heating under reflux, filtering, evaporating with rotary evaporator, use of an ice cooling bath, heating with oil bath

Instruction (batch scale 30 g)

Equipment

250 mL round bottom flask, reflux condenser, 100 mL Soxhlet extractor, extraction sleeve, glass wool, heatable magnetic stirrer, magnetic stir bar, rotary evaporator, Buechner funnel, suction flask, ice bath, desiccator, oil bath

Substances

| | |
|--|--------|
| black pepper, powdered | 30 g |
| acetic acid ethyl ester (bp. 77 °C) | 150 mL |
| 10% solution of KOH in ethanol/water 1:1 | 30 mL |

Reaction

150 mL acetic acid ethyl ester is filled in a 250 mL round bottom flask with magnetic stir bar. 50 g powdered black pepper is filled in the extraction sleeve of a Soxhlet extractor and covered with little glass wool. A reflux condenser is fixed on the Soxhlet unit, the mixture is stirred and heated for 5 hours under strong reflux.

Work up

Afterwards the solvent is evaporated at a rotary evaporator. 2.7 g of solid substance remain as residue.

The residue is stirred with 30 mL of a 10% solution of KOH in an ethanol water mixture (1:1). The mixture is filtered and the filtrate cooled in an ice bath on 0-4 °C. A crystalline

sediment precipitates. It is sucked off and washed with little water, again strongly sucked off and dried in the desiccator until it is constant in weight.

Yield: 84 mg, mp 125 –126 °C

Waste management

Recycling

The evaporated acetic acid ethyl ester is collected and redistilled.

Waste disposal

| Waste | Disposal |
|--------------------------------|--------------------------------------|
| ethanolic-aqueous filtrate | solvent water mixtures, halogen free |
| filter residue | solid waste, free from mercury |
| extraction sleeve with content | solid waste, free from mercury |

Time

6-7 hours, from it 5 hours heating under reflux

Break

Before evaporation

Before dissolving in KOH-solution

Degree of difficulty

Easy

Analytcs

Reaction control with TLC

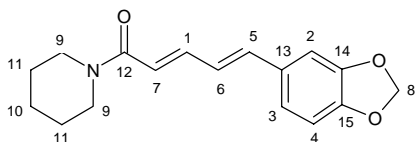
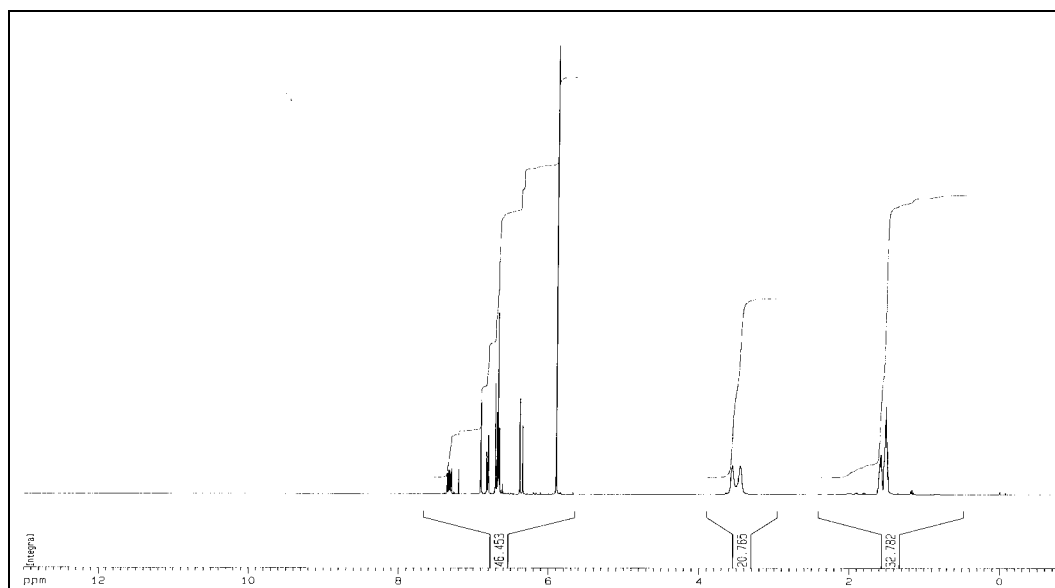
TLC-conditions:

adsorbant: Macherey and Nagel Polygram SilG/UV foils, 0.2 mm

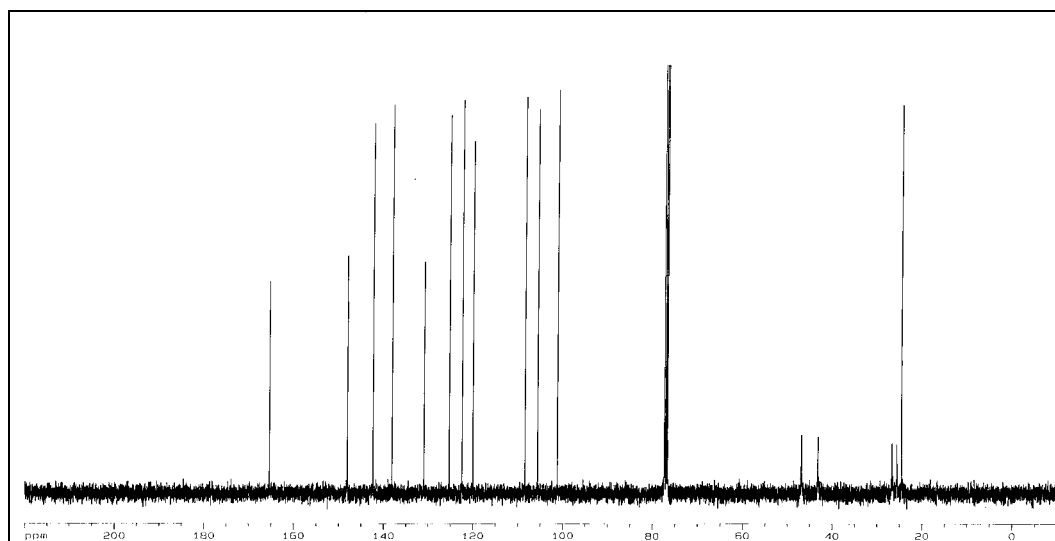
eluent: dichloromethane : acetic acid ethyl ester 3 : 1

visualisation: piperine shows at UV₃₆₅ blue fluorescence, at UV₂₅₄ fluorescence quenching

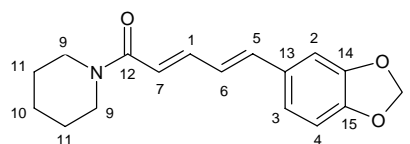
R_f (Piperine) 0.5

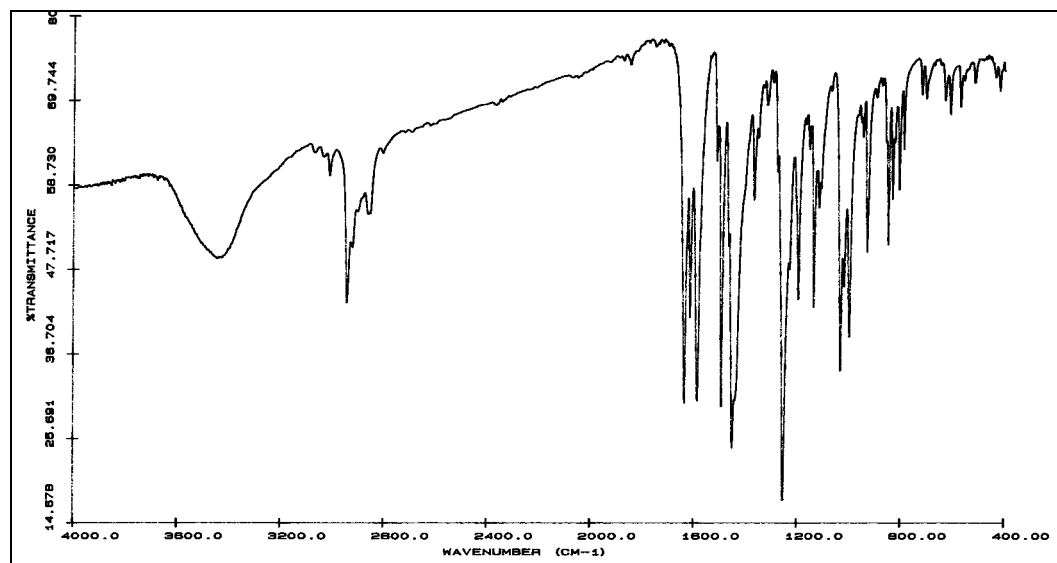
^1H NMR spectrum of piperine (400 MHz, CDCl_3)

| δ (ppm) | Multiplicity | Number of H | Assignment |
|----------------|--------------|-------------|------------|
| 1.49–1.54 | m | 4 | 11-H |
| 1.56–1.59 | m | 2 | 10-H |
| 3.45–3.56 | m | 4 | 9-H |
| 5.90 | s | 2 | 8-H |
| 6.36 | d | 1 | 7-H |
| 6.66 | m | 1 | 6-H |
| 6.67 | m | 1 | 5-H |
| 6.70 | d | 1 | 4-H |
| 6.80–6.82 | dd | 1 | 3-H |
| 6.90–6.91 | m | 1 | 2-H |
| 7.30–7.36 | ddd | 1 | 1-H |
| 7.26 | | | Solvent |

^{13}C NMR-spectrum of piperine (100 MHz, CDCl_3)

| δ (ppm) | Assignment |
|----------------|------------|
| 24.60 | C-10 |
| 25.55 | C-11 |
| 26.66 | C-11 |
| 43.17 | C-9 |
| 46.84 | C-9 |
| 101.21 | C-8 |
| 105.60 | C-2 |
| 108.41 | C-4 |
| 120.02 | C-7 |
| 122.42 | C-3 |
| 125.30 | C-5 |
| 130.95 | C-13 |
| 138.13 | C-6 |
| 142.39 | C-1 |
| 148.05 | C-14 |
| 148.13 | C-15 |
| 165.35 | C-12 |
| 76.5-77.5 | Solvent |



IR spectrum of piperine (KBr)

| (cm ⁻¹) | Assignment |
|---------------------|------------------------------------|
| 3065-3010 | C-H-valence, arene and alkene |
| 2940-2850 | C-H-valence, alkane |
| 1635 | C=O-valence, carboxylic acid amide |
| 1610 | C=C-valence, alkene, arene |
| 1580, 1490 | C=C-valence, arene |