B-Adrenoceptor-blocking drugs

Analysis of esmolol and flumolol in whole blood

Application Note

Clinical Research

Authors

Agilent Technologies, Inc.

Introduction

According to K. Kylberg-Hanssen et al, sodium dodecyl sulfate (SDS) is an effective esterase inhibitor in the determination of labile β-blockers: esmolol and flumolol. The limit of detection (coefficient of variation: 10 - 15%) is 5 nmol/L of whole blood.

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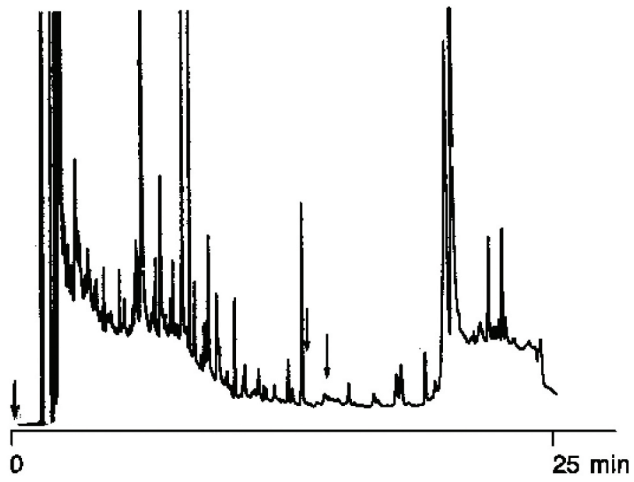
Conditions

Technique	:	GC-capillary
Column	:	Agilent CP-Sil 8 CB, 0.32 mm x 25 m fused silica WCOT CP-Sil 8 CB (0.12 µm) (Part no. CP7741)
Temperature	:	esmolol determination: 100 °C (1 min) ⟶ 185 °C 10 °C/min; 185 °C (15 min) ⟶ 250 °C, 30 °C/min; 250 °C (7 min) flumolol determination: 100 °C (1 min) ⟶ 200 °C, 15 °C/min; 200 °C (10 min) ⟶ 250 °C, 30 °C/min; 250 °C (7 min)
Carrier Gas	:	He, 60 kPa
Injector	:	Splitter, 20 mL/min T = 270 °C
Detector	:	ECD T = 350 °C
Courtesy	:	Reprinted with permission from Clinical Chemistry Vol. 31, No. 6 1985. Copyright American Association for Clinical Chemistry, Inc.

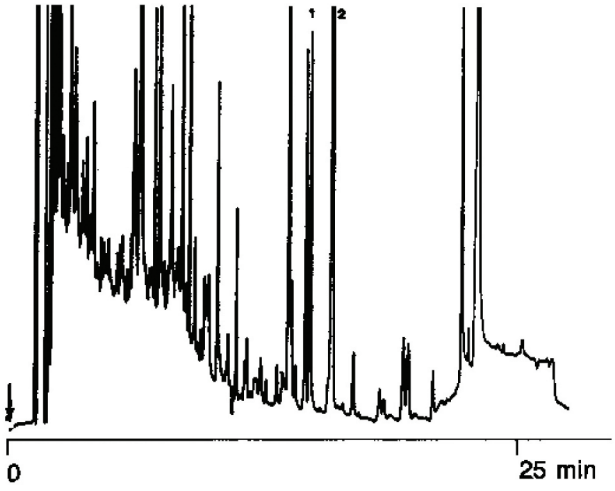
Peak identification

1. flumolol (100 nmol/L)
2. Int. Standard (H 163/37) (500 nmol/L)

Blood sample



Blood sample with added flumolol



Analytical procedure

Collect 5 mL of blood in a tube containing 0.5 mL SDS solution (200 g/L). After mixing transfer 1.0 g of the sample to a 15 mL centrifuge tube. Add 100 µL of the internal standard solution (5 µmol in phosphate buffer, pH = 6.0, 0.1 mol/L). Add 4 mL of toluene and 0.5 mL of phospate buffer (pH = 11.8, 0.2 mol/L). Shake (15 min) and centrifuge. Immerse the tube in an acetone/solid $C0_2$ mixture to freeze the aqueous layer.

Pour the organic layer into a 5 mL centrifuge tube and evaporate under a stream of N_2 at 35 °C. Redissolve the residue in 100 µL of ethylacetate and 25 µL of pentafluoropropionic anhydride. After reacting for 20 min at room temperature, evaporate under nitrogen at 35 °C. Dissolve the residue in toluene (500 µL) and inject 1.5 µL.

Acknowledgement

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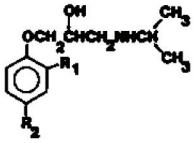
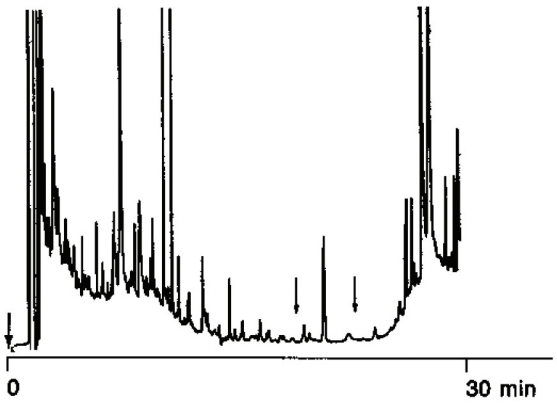
Peak identification

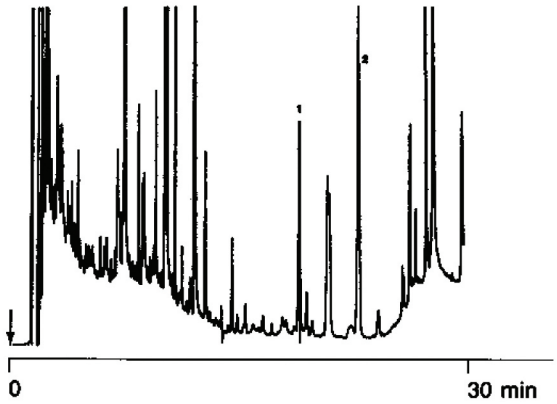
1. esmolol (300 nmol/L)
2. Int. Standard (H 163/37) (500 nmol/L)



Blood sample



Blood sample with added esmolol



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