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應用手冊

Niflumic Acid/Camphorsulfonic Acid Extracted from Rat Plasma

Waters Corporation

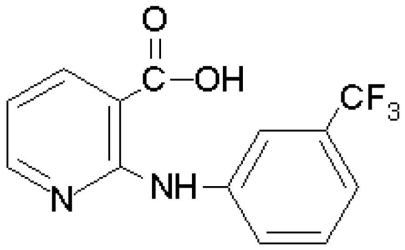
This is an Application Brief and does not contain a detailed Experimental section.

Abstract

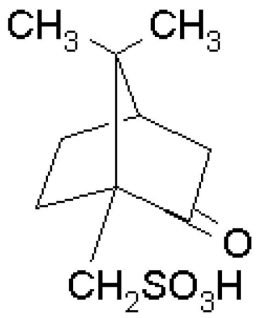
This application brief demonstrates analysis of niflumic acid/camphor sulfonic acid extracted from rat plasma.

Introduction

The compounds used in this study are - Niflumic Acid and (1S)-Camphor-10-sulfonic acid.



Niflumic Acid – 115% recovery
MW 282.2, $C_{13}H_9F_3N_2O_2$



(1S)-Camphor-10-sulfonic acid – 109% recovery
MW 232.3, $C_{10}H_{16}O_4S$

Experimental

Oasis WAX SPE Protocol

Plate:	Oasis WAX µElution Plate

Condition:	200 µL MeOH
Equilibrate:	200 µL H_2O
Load:	100 µL 1:1 diluted rat plasma, 10 pg/µL, w/2% H_3PO_4
Wash 1:	200 µL 25 mM NH_4CH_2COOH buffer in H_2O, pH 5
Wash 2:	200 µL MeOH
Elute:	50 µL (25 µL x 2) MeOH w/2% NH_4OH
Dilute:	50 µL H_2O with 2% HCOOH
Inject:	10 µL

Conditions

Column:	SunFire C_{18} 2.1 x 20 mm *IS*, 3.5 µm
Part number:	186002531
Mobile phase A:	H_2O with 10 mM NH_4CH_3COOH, pH 5.5
Mobile phase B:	MeOH with 10 mM NH_4CH_3COOH, pH 5.5
Flow rate:	0.4 mL/min
Injection volume:	10 µL
Sample concentration:	10 pg/µL each analyte
Temperature:	Ambient

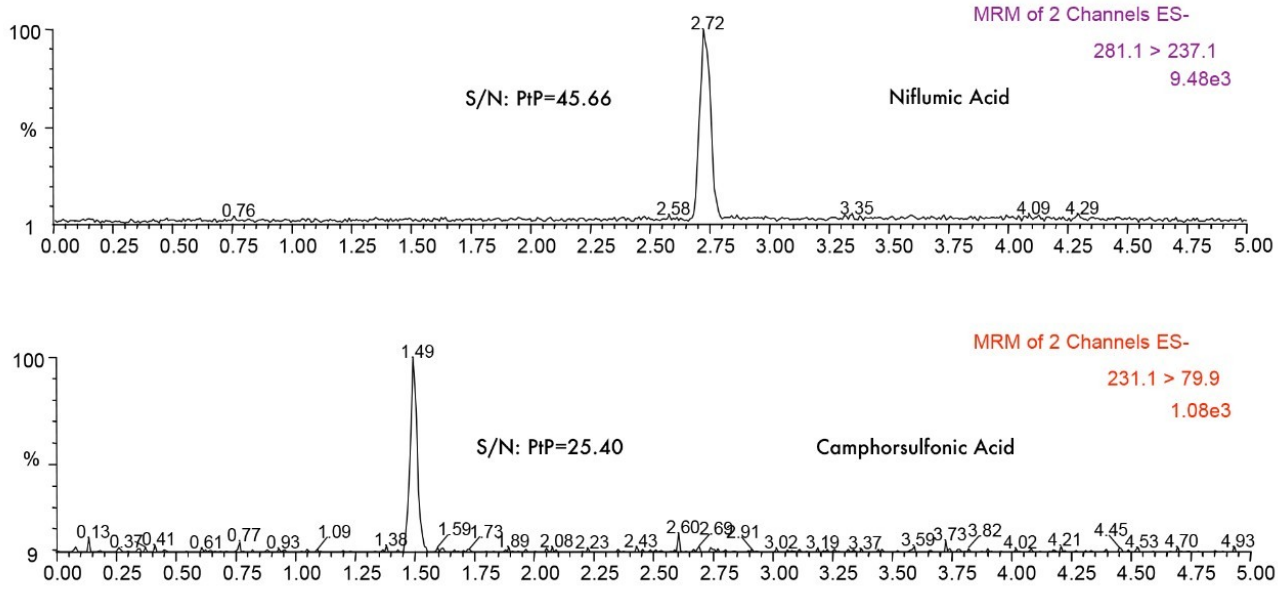
Instrument:	Waters Alliance 2695 Waters Micromass Quattro micro API
ESI source temp.:	150 °C
Desolvation temp.:	350 °C
Cone gas flow:	50 L/Hr
Desolvation gas flow:	600 L/Hr
Collision Cell:	$1.23e^{-3}$ bar (Ar gas)
Compounds	MRM Transition
Niflumic Acid	*m/z* 281.1 → 237.1
Camphorsulfonic Acid	*m/z* 231.1 → 79.9

Gradient

Time (min)	Profile	
	%A	%B
0.0	95	5
3.0	5	95
4.0	5	95
4.1	95	5
5.0	95	5

Results and Discussion

	MRM transition	Cone (V)	CID (eV)
Niflumic acid	*m/z* 281.1 ⟶ 237.1	30	17
Camphorsulfonic acid	*m/z* 231.1 ⟶ 79.9	45	30



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