APPLICATION**NOTE**

Pharmaceuticals – TG 209 *F1 Libra*®-GC-MS

TG-GC-MS Measurements on Acetylsalicylic Acid (ASA)

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1 TG 209 F1 Libra® with GC-MS coupling

Measurement Parameters

TG 209 F1 Libra® - GC-MS

TGA Parameters:
RT to 500°C
10 K/min
50 ml/min N_2
Sample mass: 1.71 mg
Al_2O_3 crucibles

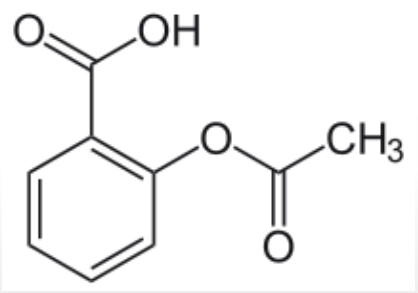
GC Parameters:
Quasi-continuous
GC furnace: 250°C
Split: 10:1
Column: HP 5 ms, 30 m
Valve switching: 30 sec

Introduction

ASA is one of the most widely used non-opioid analgesics. By esterification of the phenolic hydroxyl group of salicylic acid with acetic acid, not only is a better local tolerance achieved, but also a stronger antipyretic, antiphlogistic and especially platelet aggregation inhibiting effect [1] .

The common name is 2-acetoxybenzoic acid. It forms white, needle-shaped crystals which smell faintly of acetic acid. ASA is produced by means of the Kolbe-Schmitt synthesis, an electrophilic aromatic substitution [2].

[1] Mutschler, Arzneimittelwirkungen
[2] Laue, Reaktionsmechanismen



2 Chemical structure of acetylsalicylic acid

NGB · Application Note 008 · E · 0822 · Technical specifications are subject to change.

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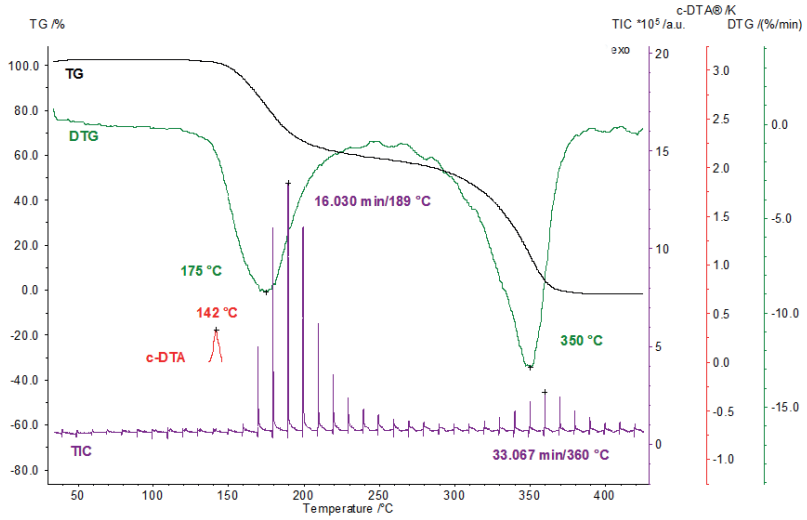


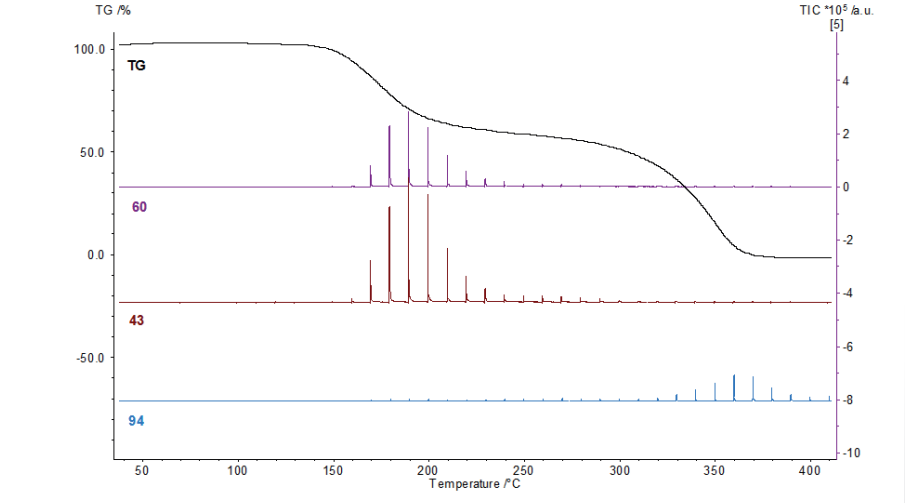
3 TGA results (black), DTA results (green), c-DTA® signal (red) and total ion chromatogram of the ASA measurements (purple)

Measurement Results

Simultaneous coupling of thermogravimetric analysis (TGA) and GC-MS (gas chromatography and mass spectrometry) enables easy analysis of the thermal behavior and decomposition products.

The melting of ASA can be detected by means of c-DTA® at 142°C. ASA decomposes in two steps (DTG minima at 175°C and 350°C). The total ion chromatogram reflects the completely measured spectra as a function of temperature. Corresponding to DTG, two maxima can clearly be seen.

The 1st TG step can be attributed to the formation of acetic acid (mass numbers 43 and 60 figure 4); the 2nd step can be attributed to phenol (mass number 94) as a decomposition product.



4 TGA results (black) and total ion chromatogram of mass numbers 43, 60 and 94

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