

## Application Note AN # 66

# ASPIRIN® Decomposition studies by TG-FT-IR

Monitoring of decomposition steps and identification of evolved gases by combined TG-FT-IR technique

### Introduction

The supply of pharmaceutical products depends on the storage stability of the active ingredient in the formulation. Therefore, the decomposition behavior of the active ingredient is very important in determining the shelf life of a pharmaceutical product. Knowledge of the decomposition products from the active ingredient is useful in helping to avoid the occurrence of undesired by-products, which form during storage.

### Application

The analysis objective is to monitor and identify decomposition behavior and thermal stability in pharmaceutical formulations. TG-FT-IR method was used under the following circumstances and samples:

- Sample: 9.89mg Acetylsalicylic Acid (ASPIRIN® pulverized tablet)
- Sample: crucible; Al<sub>2</sub>O<sub>3</sub> type
- Flowrate: 45ml/min; N<sub>2</sub>

- For simultaneous FT-IR measurement, a spectral resolution of 4cm<sup>-1</sup>, in the spectral range from 650 - 5000cm<sup>-1</sup> are used. Spectra were recorded approximately every 15 seconds.

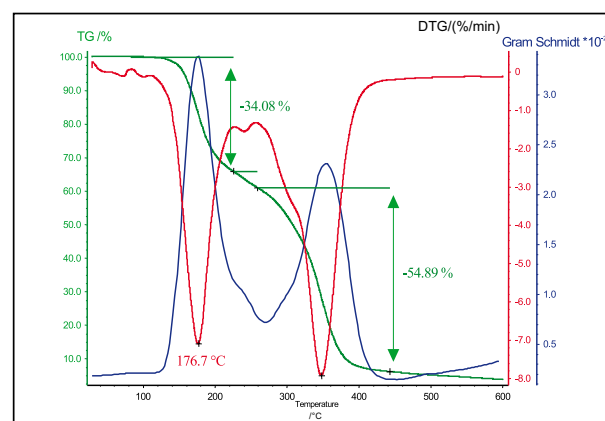
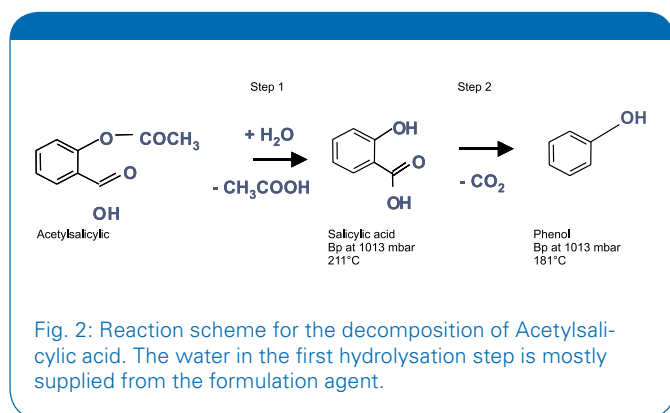


Fig. 1: TG, 1<sup>st</sup> derivative and Total-IR-reconstruction (Gram Schmidt) of ASPIRIN® decomposition

The total IR chromatogram along with the thermal decomposition behavior as a function of temperature and time (Fig. 1),

shows first the hydrolyses of the ester group of the acetylsalicylic acid into acetic acid and salicylic acid during the first thermal decomposition step. With the help of the TG-FT-IR coupling, it is easy to follow this step and to obtain the IR spectrum of the more readily volatile acetic acid. Comparison with the spectral database simplifies the interpretation (Fig. 3). At higher temperatures, in a further decomposition step, the decarboxylation of the salicylic acid is recorded with a strong release of phenol and CO<sub>2</sub>. Salicylic acid produced by the ester hydrolysis in the first step can also be monitored by the FT-IR. The quantification of the IR-signal (as easily done by the Pulse TA® method) will allow to even quantify the efficiency of the decarboxylation in step two.



## Results

This technique allows monitoring the complete decomposition steps of the acetylsalicylic acid in an Aspirin tablet. TG-FT-IR is excellently suited to follow the decomposition steps of the acetylsalicylic acid and to set up the decomposition reaction scheme as shown in Fig. 2.

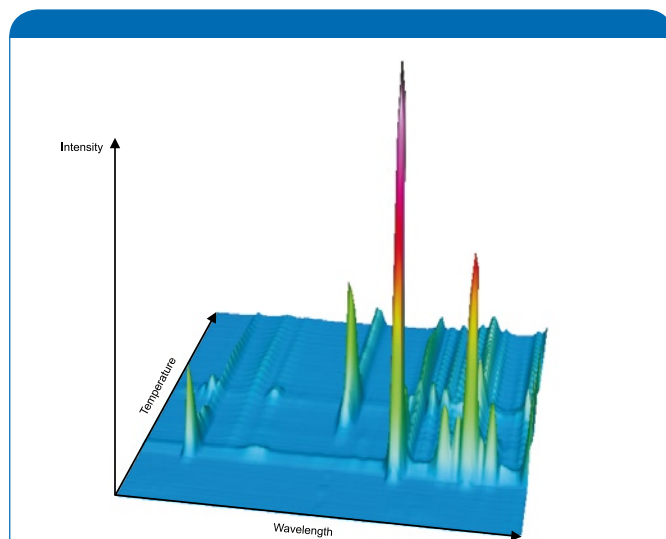


Fig. 3: 3D-IR representation of the ASPIRIN® decomposition. The two decomposition steps can be clearly seen by producing different eluents.

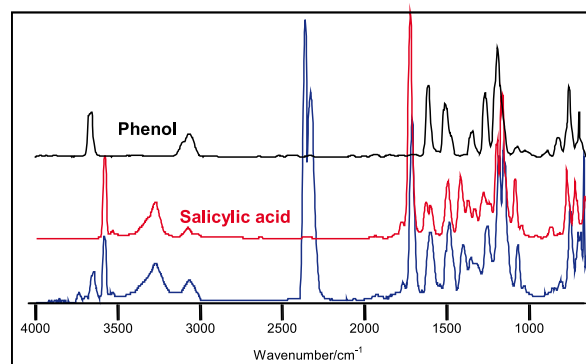


Fig. 4: Spectrum at a decomposition temperature of 345°C versus reference spectra of salicylic acid and phenol out of a digital spectral reference library.

TG-FT-IR is based on a cooperation with [www.ngb.netzsch.com](http://www.ngb.netzsch.com)

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