

Automated Sample Preparation in Analytical Chemistry: Parallel, Multistep Derivatization Protocol on Phyto Biochemicals

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Grit Laue, Syngenta AG, Basel, Switzerland

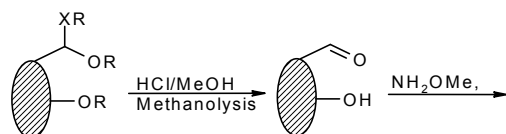
Christelle Lorin, Hai-Ho Nguyen-Ngoc, Josef Schröer, Chemspeed Technologies, Augst, Switzerland

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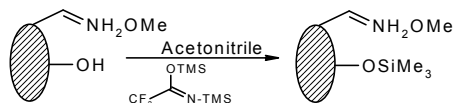
General

- Before running an analytical method, a demanding preparation of the corresponding samples by a chemical derivatization sequence sometimes is necessary.
- The high standards of today's quality management systems need to be fulfilled by automated equipment that is suitable for such chemical applications in analytical chemistry laboratories.

Reaction sketch



R = Alkyl, Acyl,
X = O, N



Objective

- Run a three-step derivatization sequence on different samples originating from plant extracts.
- Achieve a fully unattended execution of this parallel multistep procedure including work-up steps.
- Embark on a demanding chemical protocol using only small sample quantities and solvent volumes (100 to 300 μL per run) with a Chemspeed 2 mL small scale parallel reactor array.
- Operate in challenging conditions for reactions and work-up. These include: extensive reflux of small solvent quantities, establishing and maintaining anhydrous conditions, and evaporation of high boiling solvents.
- Meet the demanding requirements of quality standards in analytical chemistry (e.g. no cross contamination of volatile products between reaction vessels).

Experimental Set-Up

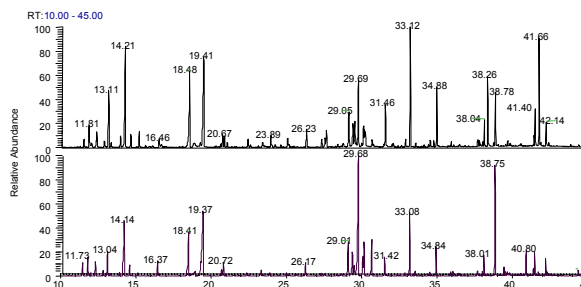
- Set-up for 16 samples on the ASW2000 equipped with an array of 2 mL small scale reaction vessels.
- Each sample was run twice on the Chemspeed equipment in order to verify reproducibility.
- An independent set of samples was prepared by classical methods for use as a reference.

Analytics

- GC/MS of each sample on a Trace GC/TSQ7000 MS (ThermoQuest/ Thermo-Finnigan) machine was accomplished.



Picture of the Chemspeed system used
and the 2 mL small scale reactor



Comparative GC/MS run on analytical sample prepared on the Chemspeed automated synthesizer (upper trace) and the reference sample manually derivatized (lower trace).

Results

- The results obtained from GC/MS analysis of samples prepared on the synthesizer are in alignment to the results of the classical reference experiments.

Summary

- A complete multistep derivatization procedure on samples originating from plant extracts was successfully automated.
- GC/MS analysis was executed on the derivatized compounds.
- Neither cross contamination of the volatile products between reactors, nor other sources of contamination could be detected.
- Based on the above described evaluation, Chemspeed synthesizers were proven to meet the highly demanding quality standards of automated equipment that are a prerequisite for applications in analytical chemistry laboratories.

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