FLASH PYROLYSIS PLATFORM

New chemistry in an instant

Fast, green, wider chemistry

- New chemical space to perform a wide range of intramolecular reactions
- Extremely high temperature up to 1000 °C
- Capable of ring closure, opening reactions, monomerization reactions and pyrolysis
- Fast and clean reactions in milliseconds

While a number of avenues are available to organic chemists for the synthesis of novel structures, it has also been shown that chemists employ a relatively small chemical technology toolbox both in terms of the set of chemical reactions used and the applied physical properties (temperature, pressure, time) of the chemical space. In response to these limitations and needs we have developed the Flash Pyrolysis Platform that reaches beyond the already known capabilities of the usual flash vacuum pyrolysis instruments by enabling one to apply non-volatile starting materials as well, via our own interchangeable vaporizer system.

The extremely high temperature, up to 1000°C, offers chemists new chemical space where they can perform a wide range of intramolecular reactions like rearrangements, ring closure or opening reactions, monomerization reactions and pyrolysis. The Flash Pyrolysis Platform gives a quick and clean entry to reactions enabling the synthesis of compounds that are otherwise complex or unsuccessful to obtain.

THE FLASH PYROLYSIS PLATFORM IS CAPABLE OF PROVIDING TWO INTERCHANGEABLE MODES:

- Flash Vacuum Pyrolysis (FVP) and,
- Continuous Liquid Spray Vaporization (CLSV)

"*Flash vacuum pyrolysis and continuous liquid spray vaporization are acknowledged methods of achieving fast clean reactions yet are largely overlooked or unexplored due to the lack of user friendly and reliable instruments. Drawing on its over 14 years of synthesis innovation ThalesNano is proud to take a further step with the implementation of the FVP, to support its customers and chemists in general to push the boundaries of what is possible in their search for new molecules*." says Dr. Ferenc Darvas, President of ThalesNano Inc.

ADVANTAGES:

- Simple: Easy to use
- Interchangeable: smooth change between flash vacuum and spray pyrolysis modes
- Powerful: Volatile and non-volatile materials can be reacted
- Efficient: The extremely high temperature translates into short reaction times (less than 1 second)
- Green chemistry: solvent free reactions (FVP)

FLASH VACUUM PYROLYSIS

During the course of an FVP reaction the substrate is sublimed or distilled in vacuum through the hot reactor tube. Due to the applied conditions (high temperature and vacuum) the FVP process typically facilitates the occurrence of unimolecular reactions where the material (in gas phase) is passed through the reaction zone in the range of milliseconds. Product then is collected in a cold trap.

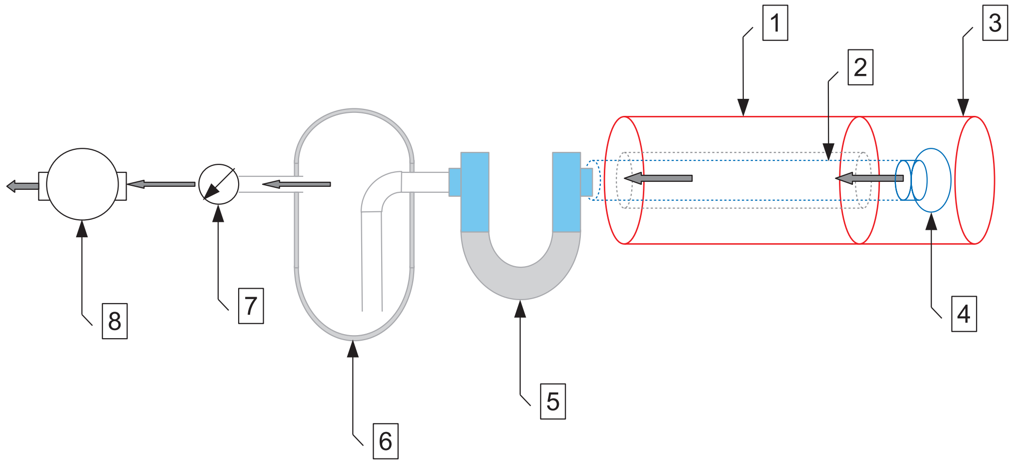
The low-pressure environment fulfills two key roles; a) helps to avoid adverse chemical reactions by limiting the number of collisions between the individual molecules. b) it decreases the boiling point of the precursors in the preheater unit, helping the starting material to be distilled/sublimated through the hot reaction zone.

CONTINUOUS LIQUID SPRAY VAPORIZATION

In case of the CLSV method, the spray nozzle disperses the solution of the starting material into the heated reaction zone. A carrier gas (typically an inert gas, e.g. nitrogen) is employed to ensure the continuous transport of the substrate through the hot tube. Therefore, in the CLSV mode the presence of vacuum is not required, and there are no constraints by the volatility of the substrate. Finally and similarly to the FVP method, the product is collected in a cold trap.

WETTED PARTS OF THE FLASH PYROLYSIS PLATFORM:

- Stainless Steel
- Quartz Glass
- Borosilicate Glass
- Rubber sealings (cold trap side)



1) FURNACE
2) REACTOR TUBE
3) PRE-HEATER
4) SAMPLE VIAL
5) COLD TRAP 1.
6) COLD TRAP 2.
7) PRESSURE SENSOR
8) VACUUM PUMP

TECHNICAL SPECIFICATION:

Reactor Module:

Preheater Temperatur Range:	RT-400 °C
Furnace Temperature Range:	RT-1000 °C
Operating Pressure:	3×10^{-3} mbar (hPa) vacuum (FVP)
Quartz Reactor Tube:	standard outer diameter: 35 mm
Max outside diameter:	55 mm
Furnace heated length:	450 mm
Physical length:	600 mm
3 controlled zone	
Electric Supply:	110 VAC 60 Hz or 230 VAC, 50 Hz, max. 2800 W
Weight:	34 kg
Dimensions (WxHxD):	1040 × 545 × 545 mm

VFP mode part:

VACUUM PUMP

Ultimate pressure:	x 10^{-3} mBar (hPa) vacuum
Dimensions:	423 × 176 × 127
Electric Supply:	110 VAC 60 Hz or 230 VAC, 50 Hz, max. 240W
Weight:	18 kg

CLSV mode part:

SPRAY NOSE

Pressure:	maximum 10 bar
Wetted parts:	316SS, quartz glass
Carrier medium:	gas (e.g.: nitrogen)
Nozzle operation range:	Liq. 0.1-5 mL/min, Gas 0.3-10 L/min

APPLICATION AREAS

- Monomolecular reactions
- Pericyclic reactions
- Cycloadditions and retro-cycloadditions
- Sigmatropic reactions
- Extrusion of small molecules (e.g. CO, CO_2, N_2, etc.)
- Cleavage of the weakest single bond
- Ring expansion reactions
- Ring closing reactions

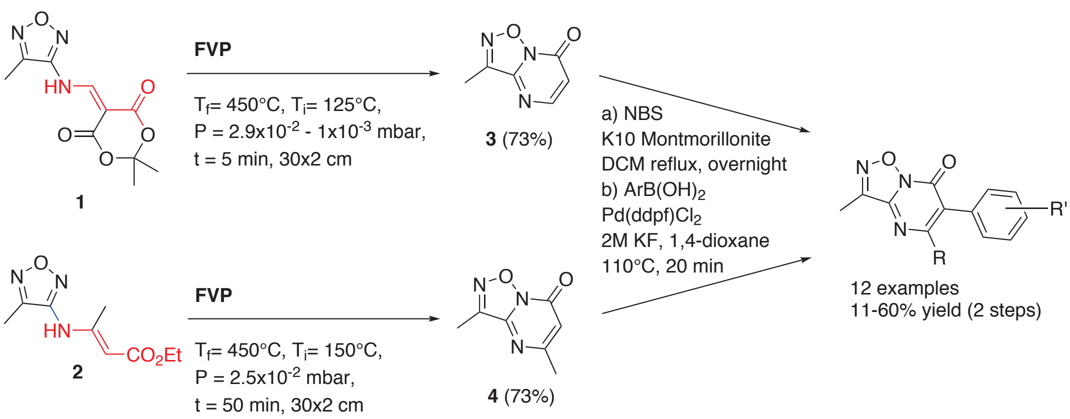
Flash vacuum pyrolysis, the key step in the synthesis of a novel oxadiazolo-pyrimidinone library

The synthesis of previously unexplored ring systems is of particularly high interest, especially in the pharmaceutical industry. Yet, it has been shown that medicinal chemists employ a relatively small chemical technology toolbox, and only a minor part of the synthetically tractable small aromatic systems can be found in the literature.

State of the art: Thermal cyclizations in batch are normally performed in solvents with high boiling points (e.g.diphenyl ether, b.p. 258 °C). However, such processes are often hampered by side-reactions and by difficult workup. Beneficially, high temperature / high pressure flow chemistry (e.g. in the Phoenix Flow Reactor) allows to apply low boiling point solvents (e.g. CH3CN, THF) in combination with the precise control of residence time.

Although flash vacuum pyrolysis is not a generally accepted method in pharmaceutical R&D, it is often advantageous when other methods afford low selectivity or the synthesis is problematic (e.g. due to clogging) in micro- or mesofluidic flow reactors.

Below we show how the flash vacuum pyrolysis technique is used as a key step in the synthesis of a novel oxadiazolo-pyrimidinone library.



ThalesNano

For more information on how flow chemistry can benefit you, please visit **www.thalesnano.com**

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