On January 1st, 2018 a new laboratory dedicated to Molecular Chemistry has been created. Gathering a roughly 90 collaborator staff, the Laboratoire d’Innovation Moléculaire & Applications (UMR7042-LIMA) is three-tiered: Université de Strasbourg, Université de Haute-Alsace and CNRS laboratory, located both in Strasbourg and Mulhouse.

The Laboratoire d’Innovation Moléculaire & Applications (LIMA) is a decisive actor for the scientific site coherence and becomes the largest laboratory dedicated to research in molecular chemistry in Alsace. One of the LIMA’s priorities is to support the emergence of new research fields, to promote fundamental research and to stimulate fruitful interactions with the socio-economic environment. The overall scientific aim of the LIMA is to develop innovative methods dedicated to molecular synthesis, to validate new therapeutic strategies and to apply them in different fields (pharmaceutical and agro-chimie, eco-responsible chemistry and materials chemistry).

The laboratory is located at both the ECPM (Strasbourg) and ENSCMu (Mulhouse) engineering schools. It is organized in 9 research teams (6 in Strasbourg and 3 in Mulhouse) related to specific scientific domains. Three primary scientific aims emerge:

1) Exploratory research in synthetic organic chemistry
   • Catalysis, asymmetric synthesis, organometallic chemistry

2) Exploratory research in biological chemistry
   • Parasite imaging, click chemistry
   • Proteomics, metabolomics
   • Multivalence
   • In-situ target research, enzymology

3) Application of biological chemistry in Life Sciences
   • Mode of action of biological processes
   • Medicinal chemistry and agrochemical products
   • Natural substances, from isolation to total synthesis
   • Design of molecular and supramolecular probes

The teams:
   • Chimie Medicinale et Phytopharmaceutique (Sébastien Albrecht – Mulhouse)
   • Biomolécules, Synthèse et Méthodologie (Nicolas Blanchard – Mulhouse)
   • Chimie Organométallique Appliquée (Michael Chetcuti – Strasbourg)
   • Synthèse et Catalyse Asymétrique (François Colobert – Strasbourg)
   • Synthèse Organique et Molécules Bioactives (Philippe Compain – Strasbourg)
   • Chimie Bio(IN)organique et Médicinale (Elisabeth Davioud-Charvet – Strasbourg)
   • Chimie Radicale, Hétérocyclique et Interfaces (Jean-Philippe Goddard – Mulhouse)
   • Chimie Organique et Hétérochimie Appliquées (Frédéric Leroux – Strasbourg)
   • Chimie des Matériaux Moléculaires (Jean-François Nierengarten – Strasbourg)

In addition to this, a strong technical support is provided by an analytical platform at the cutting edge of modern techniques, dedicated to molecular chemistry and biochemistry, which is spread over the two Alsatian sites.

From fundamental research to leading innovations

Our teams are specialized in numerous fields of organic chemistry, methodology developments to their applications in the fields of life science, agriculture, catalysis, materials …

The laboratory is world recognized for its work on chirality, synthesis of naturally and/or biologically related compounds, glycochemistry, fluorine chemistry, radical chemistry and material synthesis with application in electronics and photovoltaics.

Through that research process, many universities and companies already collaborate with us to create knowledge but also to develop the innovations of the future.

… And training the leaders of tomorrow

Fully integrated in the French and international research landscape, our teams are also dedicated to the programs of partner universities. Teaching in chemistry faculties or in the two chemical engineering schools (ECPM & ENSCMu), our staff give high level courses to train the chemistry leaders of tomorrow into a European research campus, EUCOR.

Keywords: Molecular chemistry | Catalysis | Chirality | Glycochemistry | Fluorine chemistry | Heterocyclic chemistry | Radical chemistry | Medicinal chemistry | Photochemistry | Total synthesis | Phytopharmaceutical Chemistry | Materials chemistry

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**FLASH PYROLYSIS PLATFORM**

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

**Introduction**

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,[1] and only a minor part of the synthetically tractable small aromatic systems can be found in the literature.[2]

**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 work-up.[3,6] Beneficially, high temperature / high pressure flow chemistry (e.g. in the Phoenix Flow Reactor) allows to apply low boiling point solvents (e.g. CH₂CN, THF) in combination with the precise control of residence time.[6] 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.[3]

In this application note we show how the flash vacuum pyrolysis technique is used as a key step in the synthesis of a novel oxadiazolo-pyrimidinone library.[3,4]

**Instrumentation**

Flash vacuum pyrolysis (FVP) experiments are carried out by sublimation or distillation of a substrate in vacuum through a heated reactor tube (typically quartz). The product is collected after the reactor tube in a cooled trap (dry ice / acetone or liquid nitrogen).

FVP is well suited for monomolecular transformations. Due to the low pressure (vacuum) the number of collisions between the molecules is low. Moreover, the vacuum increases the speed of migration of particles through the reactor zone resulting in short residence times (typically in the millisecond range). These properties collectively minimize the chance for side-product formation.

**Experimental**

For the experimental part, please consider the literature or visit the website of ThalesNano on www.thalesnano.com.

**Results and discussion**

The synthesis of 3 was carried out by vacuum distillation of substrate 1 through the heated quartz reactor tube (450 °C furnace temperature, 30×2 cm tube dimensions) of the FVP unit of the Flash Reactor Plus™. The ring-closed product (3) was collected after the reactor tube in a cooled trap at −78°C (dry ice/acetone). The structural confirmation of compound 3 was established by detailed NMR spectroscopic analysis (1H, 13C[1H ], 1H-13C HMBC, 1H-13CN HSQC, 1H-13C HMQC), and its elemental composition was confirmed by HRMS spectrometric analysis. All analytical data was in correspondence with the proposed structure. Synthesis and characterisation of compound 4 was done analogously. Next, compounds 3 and 4 were treated with N-bromosuccinimide under standard batch conditions to afford the brominated products (not shown on the scheme). Subsequent Suzuki-coupling with aryl boronic acids afforded 5.

**Conclusion**

We demonstrated how the flash vacuum pyrolysis technique can afford previously non-described heterocycles. These heterocycles were then derivatized to afford a library of more than 20 oxadiazolo-pyrimidinones.[3,4]

**Acknowledgement**

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**References**

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