Add, Mix, Heat, Filter - Repeat, Repeat, Repeat

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1. Introduction

The HiWAY-2-MAT platform (*Méthodes d'exploration haut-débit autonome et combinatoire en chimie du solide / High-throughput combinatorial and autonomous pathways in solid state chemistry*) is part of a large France-wide collaboration for the acceleation of materials development (the PEPR-DIADEM), and is a decentralised synthesis platform set up in multiple labs in France, involving syntheses robots and high-throughput characterisation instruments. Ours is a multi-stage platform including a robotic system for inorganic wet-chemical synthesis and sample preparation, which prepares solid powder samples suitable for rapid XRD, photoluminescence, and Raman analysis.

Herein we discuss the setup and integration of the platform into an existing traditional chemical lab, as well as its currently active projects, producing diverse materials, including luminescent perovskites, phosphates and silicates as catalytic supports, and thermoelectric chalcogenides.

2. Design, setup, use

2.1 Synthesis platform

The synthesis platform is a North Robotics N9 arm and associated modules. The robotic arm handles vials, with modules capable of adding and weighing liquid and solid reagents, heating, mixing, pipetting and filtering materials, to produce X-Y sample grids which are directly transferable to characterisation instruments without further sample preparation.

2.2 Defining, sequencing, and scheduling

To work with different groups, projects, and chemistries, and to simplify the creation and execution of experiments, ontologies for the robots capabilities which apply across different synthesis protocols, such as "Add", "Mix", "Heat", and "Filter" have been defined. This helps to use the platform with a chemical orchestration system, of which many are being actively developed today, as well as assisting with experimental scheduling. Tasks defined in terms of how they occupy various portions of the robot, coupled with predictions of the completion time for a given occupancy, enables the platform to "look ahead" to the next step, helping avoid bottlenecks during parallel syntheses. The method by which reactions are defined has been created in such a way as to be compatible with existing and online tools for experimental generation and Bayesian optimisation.

2.2.1 Combinatorial inorganic syntheses

Previous work in our group has focused on the doping of phosphor materials to control emission wavelength[1], and this work is being expanded to generate large grids of multiply-doped samples, as we seek to exploit and understand complicated interactions of codopants in materials. This codoping is also being investigated in the synthesis of heterogeneous catalysts for the production of fuels from biomass. In addition to perovskite and phosphate syntheses, metal organic frameworks have been synthesised on the platform, making use of appropriate-dimension pressure vessels, and larger pressure grids, enabling batch hydro- and solvothermal syntheses to be conducted.

2.3 Figures

Fig. 1: Looping high-throughput combinatorial synthesis, rapid characterisation, and machine learning.

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References

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