DMP title

Project Name Catalytic and solid state NMR spectroscopic study of bimetallic catalysts: synergisms between Lewis acid and transition metal sites in carbonylation and transfer hydrocyanation reactions (FWO DMP) - DMP title

Grant Title G066522N

Principal Investigator / Researcher Dirk De Vos

Description Catalytic coupling of two small molecules may proceed much faster if each molecule is activated on a discrete metal centre. Especially combinations of transition metals (TM) and Lewis acids (LA) can generate a special synergy in heterogeneous catalysis. In carbonylation of alcohols (or epoxides), CO and the alcohol (epoxide) are activated resp. by TM and LA sites; in transfer hydrocyanations, the slow steps are accelerated by binding of nitriles on LAs. So far, there are hardly any techniques showing how organic molecules are activated in such an interplay of 2 active centres (TM + LA). The project explores powerful solid state NMR spectroscopy techniques to map the proximity of LA and TM sites, and to detect which molecules bind on which active centres (LA, TM). Experiments comprise single- and double-resonance spectroscopy, possibly at elevated temperature and/or pressure. The proximity of paramagnetic centres is detected via large paramagnetic shifts and significantly shortened relaxation times. Isotopes used are 1H, 13C, 15N, 119Sn, 27Al, 29Si, some of them after enrichment. For transfer hydrocyanation, LA centres are dispersed on a support, and combined with a Ni phosphine TM. For carbonylation, both TM and LA are introduced into a porous material, e.g. a zeolite or a metalorganic framework. The NMR insights will enable a rational design of improved catalysts.

Institution KU Leuven

1. General Information

Name applicant

Professor Dirk De Vos

FWO Project Number & Title

G066522N - Catalytic and solid state NMR spectroscopic study of bimetallic catalysts: synergisms between Lewis acid and transition metal sites in carbonylation and transfer hydrocyanation reactions

Affiliation

- KU Leuven

2. Data description

Will you generate/collect new data and/or make use of existing data?

- Generate new data

Describe in detail the origin, type and format of the data (per dataset) and its (estimated) volume. This may be easiest in a table (see example) or as a data flow and per WP or objective of the project. If you reuse existing data, specify the source of these data. Distinguish data types (the kind of content) from data formats (the technical format).

Procedures for performing reactions, for synthesizing catalysts and initial observations of these experiments will be gathered. Such procedures will also result in raw data files containing chromatographic data (GC-FID, GC-MS) and material characterization data (XRD, physisorption and chemisorption data or spectroscopic analysis via **NMR** etc.). These will be processed in excel files, summaries and presentations, finally culminating in manuscripts. The combined virtual volume of these datasets will be limited (< 15 GB), and will consist mostly of data files containing NMR Spectra/GC chromatograms (up to 10 GB).

Examples:

Type of Data	Format	Total volume	How Created
X-Ray diffraction patterns	.xrdml	<500 MB	XRD Spectroscopy of synthesized catalysts
NMR spectra	/	1-5 GB	Raw and processed spectra of synthesized catalysts
Reactor design schemes	.svg/.pptx	<500 MB	Design schemes for the catalytic reactors
GC chromatograms	.gcd	1-2GB	GC analysis of worked up reaction samples
Data analysis	.xlsx	<1GB	data analysis of formed products
FT-IR-data	calcmet.spectrum	<1GB	FT-IR analysis of gaseous products.

3. Legal and ethical issues

Will you use personal data? If so, shortly describe the kind of personal data you will use. Add the reference to your file in KU Leuven's Register of Data Processing for Research and Public Service Purposes (PRET application). Be aware that registering the fact that you process personal data is a legal obligation.

- No

Are there any ethical issues concerning the creation and/or use of the data (e.g. experiments on humans or animals, dual use)? If so, add the reference to the formal approval by the relevant ethical review committee(s)

- No

Does your work possibly result in research data with potential for tech transfer and valorisation? Will IP restrictions be claimed for the data you created? If so, for what data and which restrictions will be asserted?

- Yes

The methods and reactor configurations developed in this project have potential for valorization, which will be subjected for discussion during the project. Patents will be submitted, transferred and valorized with the assistance of the intellectual property unit of KU Leuven Research & Development (LRD).

Do existing 3rd party agreements restrict dissemination or exploitation of the data you (re)use? If so, to what data do they relate and what restrictions are in place?

- No

4. Documentation and metadata

What documentation will be provided to enable reuse of the data collected/generated in this project?

Details to reproduce reaction procedures are described in a personal lab booklet. Upon presentation or publication of data, procedures are described in sufficient detail to enable a reproduction of the generated results for an experienced user. ReadMe-files or recording methods for advanced characterization are stored in parallel with generated data.

In Ljubljana, G. Mali is responsible for the preservation of the raw NMR data. The analysis will be provided to KULeuven and stored additionally to ensure proper documentation and data transfer.

Will a metadata standard be used? If so, describe in detail which standard will be used. If no, state in detail which metadata will be created to make the data easy/easier to find and reuse.

- No

Although no metadata standard is available for the advanced characterization in this project, most techniques store a non-standardized ReadMe-file containing recording parameters and file

information in parallel with recorded datasets. Alternatively, some datasets contain metadata headers on recording parameters (e.g. XRD diffractograms). The data will be stored and named in a consistent manner with unambiguous identifiers. Descriptions of experiments and materials are kept in physical lab notes for each identifier. The data will be processed and summarized in a comprehensive excel worksheet with references to the identifiers. The resulting summaries will culminate in presentations, and finally in manuscripts and/or patents.

5. Data storage and backup during the FWO project

Where will the data be stored?

Physical hand-notes of procedures and initial observations are kept in lab books. Digital data, with unambiguous identifiers for reactions and materials, are kept in cloud storage from **MS OneDrive for companies**. KU Leuven has a long term agreement offering up to 2 TB of storage for each user, coordinated through their general IT department (SET-IT).

In addition to digital acces and easy collaboration through shared folders, two additional copies of all data are stored on hard drives.

Similar strategies apply at the National Institute of Chemistry: NMR routines and raw data are stored on a storage disk at the NMR center; NMR raw and processed data, as well as input and output files of first-principles calculations are stored on both the central servers and a storage disk of the Department of Inorganic Chemistry and Technology.

How is backup of the data provided?

The data is automatically stored in the cloud and on the university servers through OneDrive synchronization. Most raw data is also stored on the researcher computer (monitored through SET-IT) and frequently backed up off-line on external hard drives.

Is there currently sufficient storage & backup capacity during the project? If yes, specify concisely. If no or insufficient storage or backup capacities are available then explain how this will be taken care of.

- Yes

Thanks to the efforts of the KULeuven IT department there is up to 2 TB of cloud acces via OneDrive. As the project is not expected to generate more than 15 GB of data this is more than sufficient.

What are the expected costs for data storage and back up during the project? How will these costs be covered?

These costs are covered university wide through the IT department. No additional costs are expected for the storage of data. In case additional costs do arise, they will be covered by the project budget or reserve funds.

Data security: how will you ensure that the data are securely stored and not accessed or modified by unauthorized persons?

The data will be stored with the same precautions as the high safety standard provided by the KULeuven IT department.

6. Data preservation after the FWO project

Which data will be retained for the expected 5 year period after the end of the project? In case only a selection of the data can/will be preserved, clearly state the reasons for this (legal or contractual restrictions, physical preservation issues, ...).

All data will be retained for the (KULeuven recommended) 10 year period after the end of the project.

Where will the data be archived (= stored for the longer term)?

Upon termination of the contract, in additon to long term storage on the OneDrive cloud, the data will be backed-up and stored on an external hard drive (Samsung Portable SSD T5 1 TB), managed by Annelies Van Vlasselaer.

What are the expected costs for data preservation during the retention period of 5 years? How will the costs be covered?

The high capacity of the cloud storage, in additon to the available external hard drive (1 TB) enables the preservation of data
from multiple terminated or finished projects. Currently, an average of 15 GB is used for the

finished projects of each user, which allows to divide its cost over approximately 60 users. Given the cost of the available hard drive of 120 EUR, the expected costs are negligible. The involved IT-expenses are included in the project's consumable expenses or covered by reserve funds.

7. Data sharing and reuse

Are there any factors restricting or preventing the sharing of (some of) the data (e.g. as defined in an agreement with a 3rd party, legal restrictions)?

- No

Which data will be made available after the end of the project?

Upon publication of the research results, the full datasets will be made available upon reasonable request

Where/how will the data be made available for reuse?

- In a restricted access repository

For KULeuven researchers or project collaborators, the raw data will be available for reuse through the shared folders.
For external users this will be available after publication of the papers or public announcement of any approved patents, upon reasonable request.

When will the data be made available?

- Upon publication of the research results

Who will be able to access the data and under what conditions?

Only uses for research purposes will be allowed and commercial reuse will be excluded.

What are the expected costs for data sharing? How will the costs be covered?

No additional costs are expected as the data can be shared via online platforms (e.g. WeTransfer) or the already available shared folders in the storage cloud from OneDrive. If additional costs would occur they will be covered from reserve funds.

8. Responsibilities

Who will be responsible for data documentation & metadata?

The PhD student involved, in close collaboration with his Supervisor (Prof. Dirk De Vos).

Who will be responsible for data storage & back up during the project?

The PhD student involved, in close collaboration with his Supervisor (Prof. Dirk De Vos) and the administration office under coordination of Annelies Van Vlasselaer.

Who will be responsible for ensuring data preservation and reuse ?

Prof. Dirk De Vos and Annelies Van Vlasselaer.

Who bears the end responsibility for updating & implementing this DMP?

Prof. Dirk De Vos bears the end responsibility of updating & implementing this DMP.