

Call for participation to IS-NMR-ILC 001_2022

1. AIMS OF THIS INTERLABORATORY COMPARISON

The main purpose of the present ILC is to assess the reproducibility of qNMR and non-targeted NMR methods by comparing different quantification protocols, one based on the use of an internal standard molecule and another one based on the use of an external references.

In particular, the aims of the Interlaboratory Comparison are:

1. Validation of a non-targeted NMR method for classification of wheat samples;
2. Validation of a non-targeted NMR method for classification of pasta samples;
3. Validation of a qNMR method by both internal and external standard.

The Interlaboratory Comparison is organized in the framework of the project IPERDURUM (Project title: "Filiera frumento duro: innovazione varietale, qualità e tracciabilità delle produzioni pugliesi", CUP: B39J20000160009 funded by Regione Puglia under the Programma di Sviluppo Rurale (PSR) 2014-2020 Puglia – Misura 16 "Cooperazione" – Sottomisura 16.2 – "Sostegno a progetti pilota e allo sviluppo di nuovi prodotti, pratiche, processi e tecnologie" – DdS n. 94250037697).

2. MATERIAL DESCRIPTION

The total number of NMR tubes provided to participants will be 12.

- n. 4 sealed NMR tubes will contain buffered aqueous extracts of wheat samples added of sodium azide as biocide and 3-(trimethylsilyl)-2,2,3,3-tetradeuteropropanoic acid sodium salt in D₂O, as internal reference.
- n. 4 sealed NMR tubes will contain buffered aqueous extracts of pasta samples added of sodium azide as biocide and 3-(trimethylsilyl)-2,2,3,3-tetradeuteropropanoic acid sodium salt in D₂O, as internal reference.
- n. 3 sealed NMR tubes will contain aqueous solutions of reference materials.
- n. 1 sealed NMR tube will contain methanol-d₄ as NMR thermometer.

3. SOLUTION NMR EXPERIMENT

1D ¹H-NOESY experiment with presaturation of the residual water signal is required for the NMR tubes containing aqueous solutions. Five repetitions per tube are required.

A 90° single pulse experiment is required for the NMR tube containing methanol-d₄. One repetition is required.

The total experimental time needed is approximately 30 h.

Acquisition and processing parameters will be detailed in “Guidelines and contract terms” available in the NMR Spectrometer Profile (see IS-NMR-ILC Scheme).

4. COSTS

Participation is **free of charge** for all participants (Sample delivery is free of charge only for EU participants. Sample delivery costs for extra-EU participants will be quoted after participation request).

Electronic version of the final report will be free of charge for all participants.

5. REGISTRATION

Link: <https://innovative-solutions.it/4th-nmr-interlaboratory-comparison/>

6. ILC COORDINATOR:

S.A.Mer Servizio Analisi Chimico-Merceologiche,

via Emanuele Mola 19, 70121, Bari BA.

Contact person: *Katia Antonicelli*

E-mail: amministrazione@samer.it

7. ILC CUSTOMER (FUNDER):

Innovative Solutions S.r.l.,

zona H 150/B, 70015 Noci (BA), Italy

Contact person: *Maurizio Triggiani*

E-mail: ilc@innovative-solutions.it

8. SCIENTIFIC COMMITTEE

Vito Gallo, Polytechnic University of Bari and Innovative Solutions S.r.l., Italy

Piero Mastrorilli, Polytechnic University of Bari and Innovative Solutions S.r.l., Italy

Mario Latronico, Polytechnic University of Bari and Innovative Solutions S.r.l., Italy

Biagia Musio, Polytechnic University of Bari and Innovative Solutions S.r.l., Italy

Rosa Ragone, Polytechnic University of Bari, Italy

Maurizio Triggiani, Innovative Solutions S.r.l., Italy

Stefano Todisco, Polytechnic University of Bari, Italy

Marica Antonicelli, Polytechnic University of Bari, Italy

Trisolini Maria, Innovative Solutions S.r.l., Italy

9. RIGHTS AND OBLIGATIONS OF THE PARTICIPANTS

Participant must follow instructions contained in the “Guidelines and contract terms” available in the NMR spectrometer profile. If instruction will not be complied, data will not be considered for final elaboration.

Participants (only those who have complied with the instructions) will be mentioned in the final report and in any kind of publication containing NMR data they produced.

In case of publication in peer review journals, participants will co-author the manuscript according to the rule: 1 spectrometer, 1 co-author.

Innovative Solutions will be the owner of the NMR data of the present ILC.

Raw NMR data will be not distributed to participants, neither entirely nor partly.

Minimum number of participants: **10**.

Maximum number of participants: **50**.

9. TIMETABLE

2022/07/15 – 2022/10/05: Call open and registration (<https://innovative-solutions.it/invitation-to-the-4th-interlaboratory-comparison-of-nmr-techniques/>);

2022/10/06 – 2022/11/05: Actions of Innovative Solutions (Recognition of the spectrometers available for the ILC and publication of the “Guidelines and contract terms”, sample preparation, NMR sample homogeneity assessment and sample stability control);

2022/11/06 – 2022/11/30: Sample delivery

2022/12/01 – 2023/01/15: NMR experiment registration and data submission

2023/01/16 – 2023/02/10: Data elaboration

2023/02/11 – 2023/02/28: Publication of the report.

10. SUBCONTRACTS

The subcontract regulates:

- selection of NMR laboratories;
- sample preparation;
- shipping of samples to NMR laboratories;
- communication of the guidelines for running NMR experiments;
- organization and management of all activities and communications necessary for the collection of results.

11. SUBCONTRACTOR

S.A.Mer Servizio Analisi Chimico-Merceologiche, via Emanuele Mola 19, 70121, Bari BA.