*Guidance Document*

# NMR Data Sharing on DATAVERSE

*How to Prepare and Share Raw NMR Data along with Your Publication*

*Enhancing the Transparency and Reproducibility of Structure Elucidation while Improving the Dereplication Process*


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# Overview

Consider in advance that you will need

- The raw NMR data
- The annotated, fully assigned $^{1}H$ and $^{13}C$ NMR spectra in some electronic format.

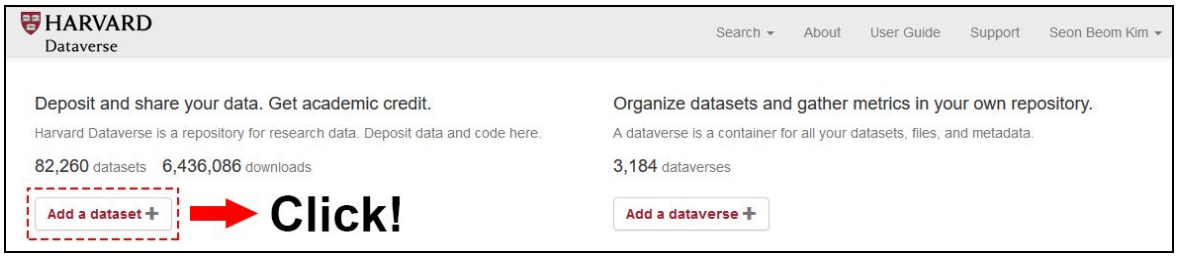
The file names should be chosen such that users will understand intuitively the content of each file.
The steps are as follows:

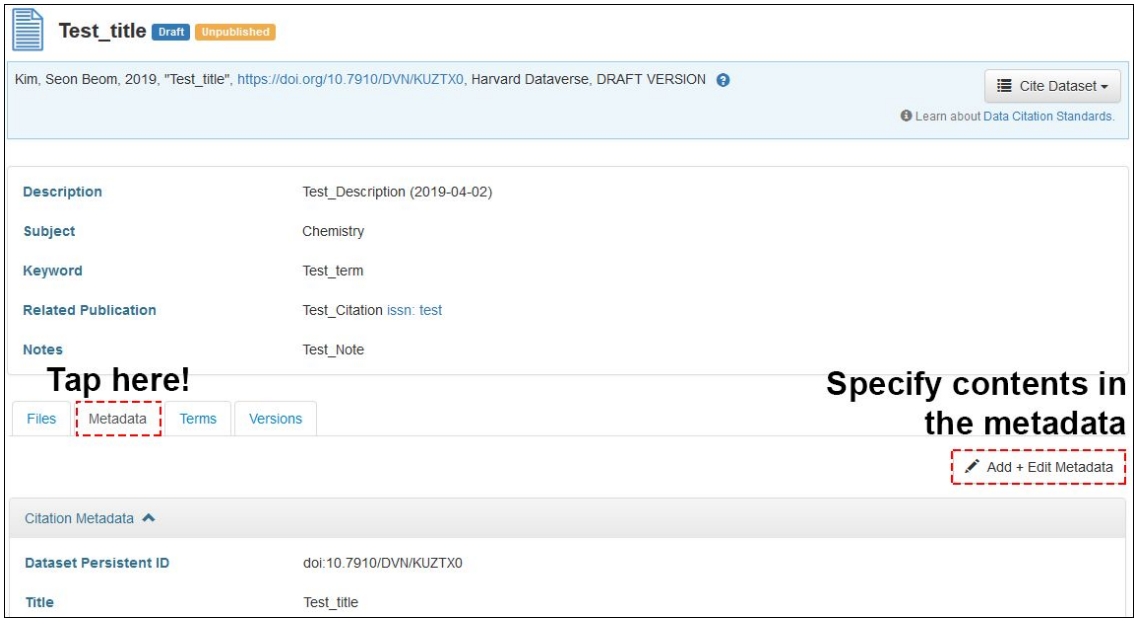
**Step 1** Create a new Dataset
**Step 2** Describe the detailed Metadata
**Step 3** Upload NMR data files on Dataset
**Step 4** Describe the uploaded NMR data

# Stepwise Process

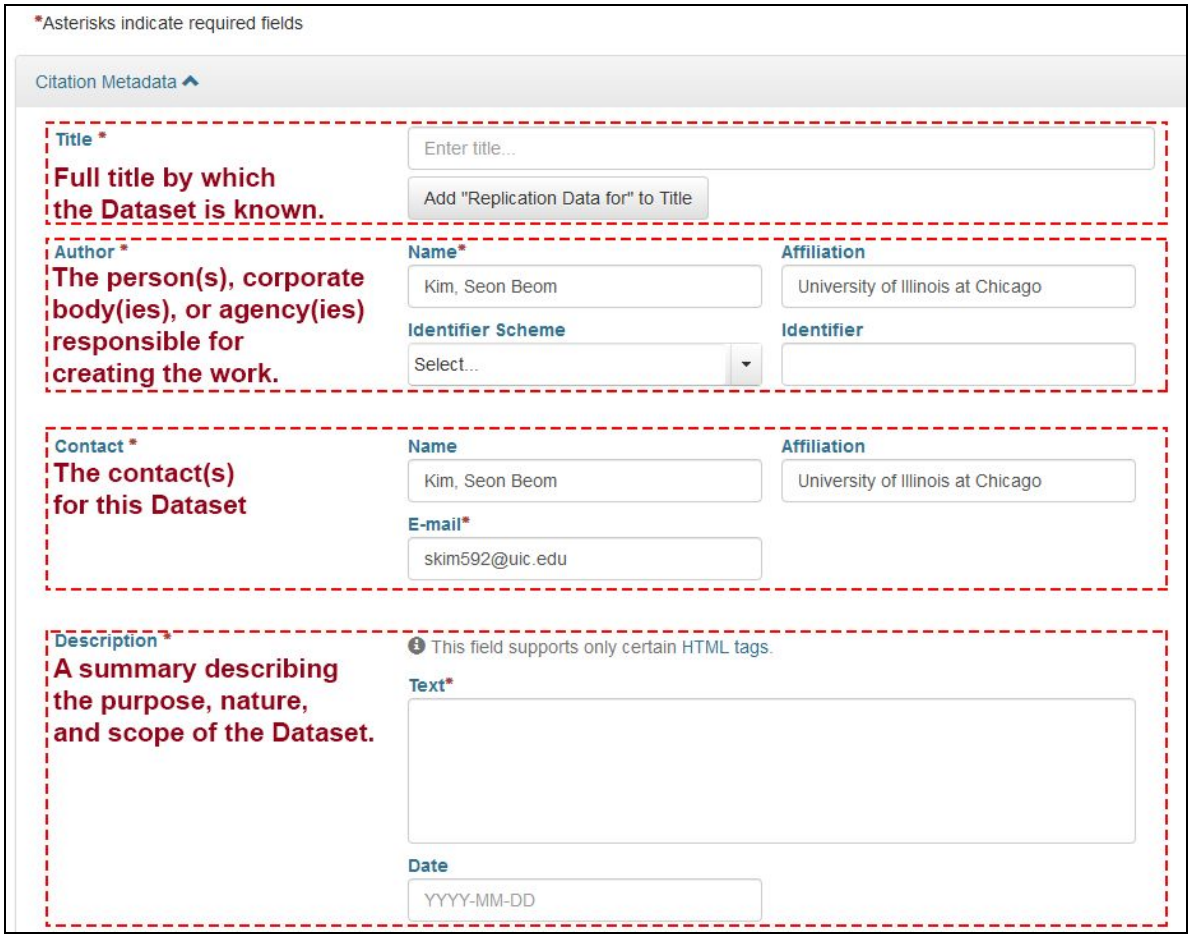
## A. Steps 1-2: Create A New Dataset and Fill in the Metadata

➢ Testing the creation of a dataset only try on the **Demo Version Dataverse** (https://demo.dataverse.org)
➢ you can create your own dataverse (for your laboratory publications for example) and create a **new dataset** (https://dataverse.harvard.edu/) within this dataverse.
➢ Your dataset will now have an attributed DOI



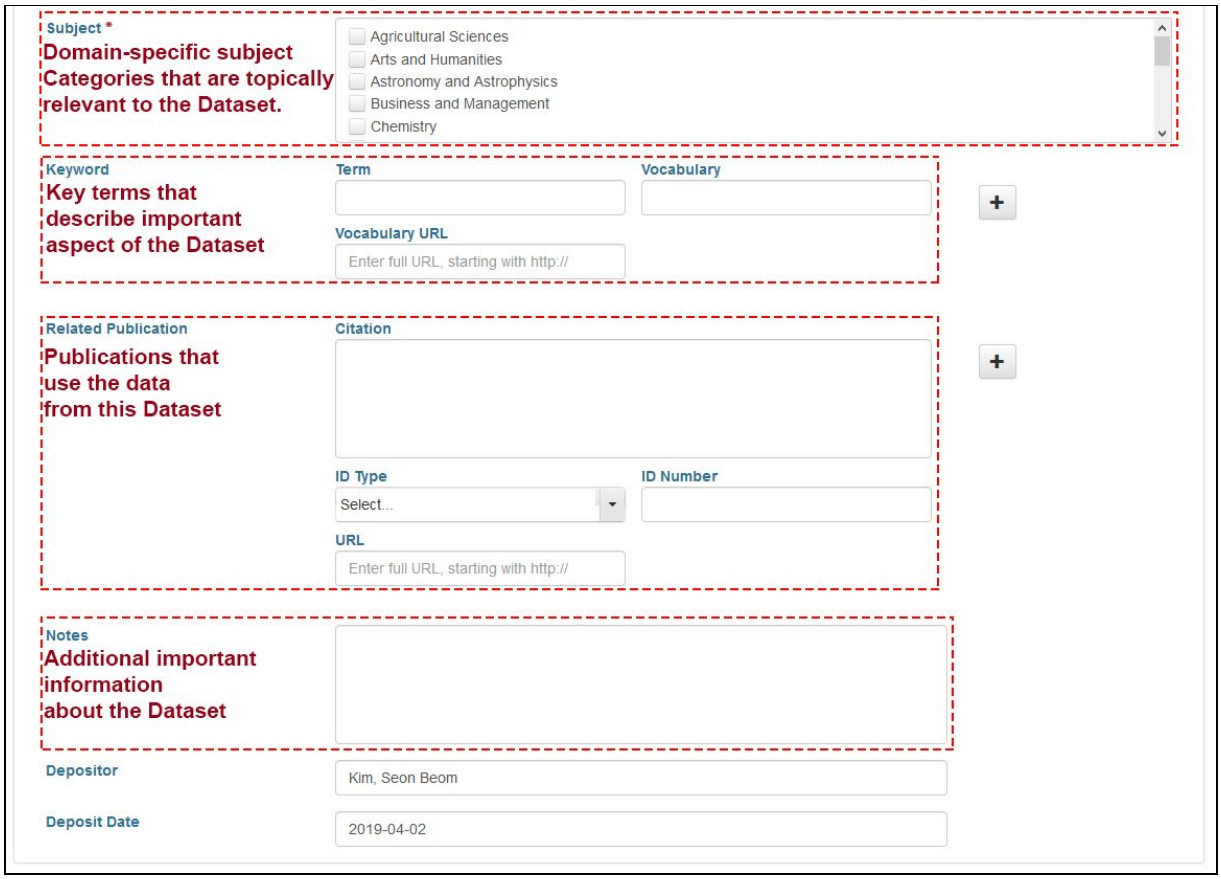



**METADATA**




- **Title:** The title can be related to the type of compounds described in the NMR data or to your research article.
- **Author:** Add authors with name, affiliation, identifier (e.g ORCID #). Use your ORCID only, avoid the other author identifier schemes such as ISNI. If you do not have a ORCID, create one!
- **Contact:** Indicate who among the authors will be the contact for this dataset.
- **Description:** Indicate what this dataset contains
  - Purpose of the research (see also Related publication below)
  - Describe the NMR dataset (1D; $^{1}H$ and $^{13}C$, 2D; COSY, HSQC, HMBC) with the name of your structure, its Pubchem ID if known (with **http://link**)
  - Indicate whether you also performed a purity determination of the corresponding compounds
- **Subject:** Choose the specific subject categories : eg. Chemistry
- **Keyword:** will help scientists to find your datasets in the repository (structure elucidation, quantitative NMR, NMR raw data). Just fill Term, unless you are using ontologies.
- **Related publication:** **IMPORTANT**

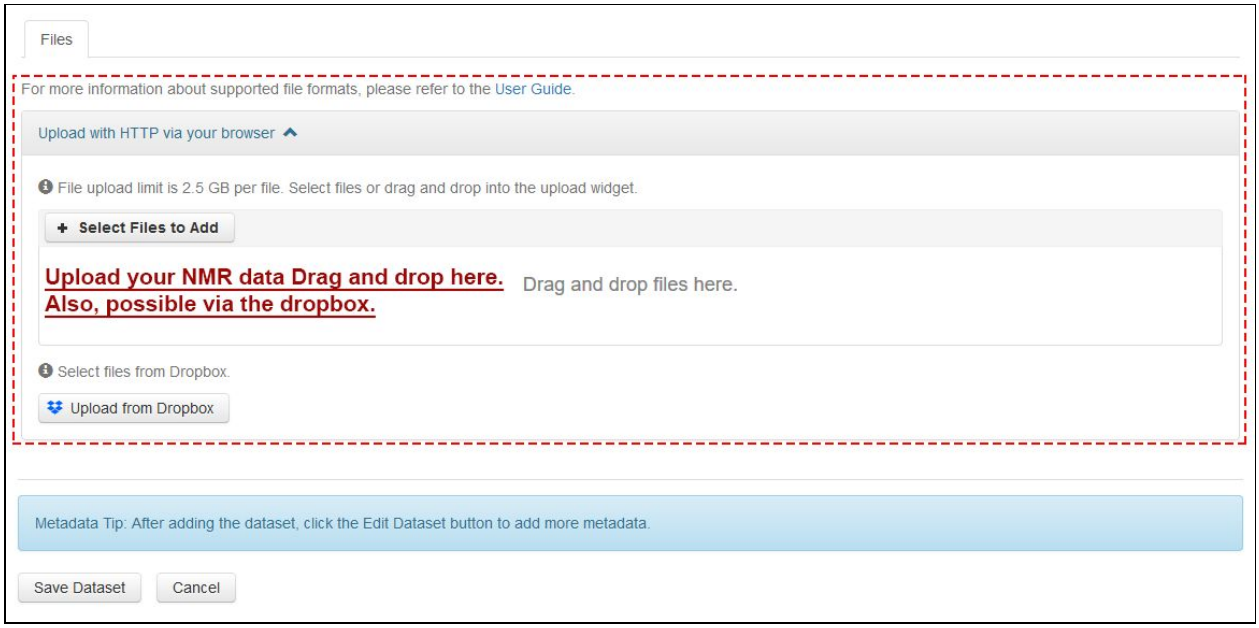
**HERE you can actually link your dataset to your (just published) research article by adding its DOI. Also if your article is not yet published when you release your data, you can still add this information later. Everything stays together and related to each other.**






- **Subtitle: you can possibly** add a secondary title or certain limitations on the main title.
- **Alternative Title:** A title which commonly referred or abbreviation of the title.
- **Producer:** Person or organization with the financial or administrative responsibility over this Dataset
- **Producer Date:** Date when the data collection or other materials were produced (not distributed, published or archived)
- **Production Place:** The location where the data collection and any other related materials were produced.
- **Contributor: DON'T FORGET TO ACKNOWLEDGE ANYBODY WHO PARTICIPATED IN DATA ACQUISITION!**

## B. Steps 2-3: Prepare and upload your NMR data




- **Prepare your NMR data on your personal computer:**

**Create your data folder containing the following information**

1. **Folder with all the raw 1D and 2D NMR data**
    a. Name your folder with Compound_Magneticstrength_Solvent_NMRdataformat
    Example Naming: NameOfCpd_RawNMRdata_TypeOfInstr_Solv_MagFld
    Basically the name of your folder should contain essential information describing your NMR data
    b. Each spectrum/subfolder within this folder should have its title:
    Example: $^{1}$H, gCOSY, HSQC, $^{13}$C_DEPT135

2. **Folder containing the "jdx" formats of all your 1D and 2D NMR data (optional)**
    **a.** With the same name scheme and title as described above
    Example Naming: NameOfCpd_NMRdata_TypeOfInstr_Solv_MagFld.jdx
    Offering you NMR data as jdx. format should allow a more "universal" use of your data with different NMR software or platforms.

3. **PDF/ png files with fully assigned/annotated $^{1}$H and $^{13}$ NMR spectra**
    Although it is not absolutely necessary for sharing your NMR data, this type of document will facilitate the interpretation of your data. The annotated spectra go hand in gloves with any of your publication related to the description of new structures. Such document will promote transparency, and is educational at the same time!
    a. If the structure is complex, create a document with expansion of the region of interest for more clarity, and then save/export your compiled documents in PDF (or png) formats.

b. Name your document with essential information:
Example Naming: NameOfCpd_annotated_NMRspct_Solv_MagFid.pdf

4. **The structure of your compound as a ".mol" file**
   a. Name of compound (e.g. TestCpd01)
   Example Naming: NameOfCpd.mol

5. **Folder with quantitative NMR analysis**
   If you use quantitative NMR (qNMR) for purity determination or quantitation of compounds. This folder should contain all the elements necessary to reproduce or calculate the purity of your compound.
   a. $^{1}$H NMR spectrum acquired under quantitative conditions (Bruker format or *jdf, and *jdx)
   b. The calculation spreadsheet used to determine the purity, using a calibrant or the 100% method
   See also https://gfp.people.uic.edu/qnmr/content/qnmrcalculations/index.html
   c. The annotated $^{1}$H NMR spectrum with the integrales for each considered signals (*pdf or *png)

**Example of your dataset on your personal computer:**

| Name | Date modified | Type | Size |
|---|---|---|---|
| TestCpd01_HiFSA_Solv_MagFld | 4/3/2019 11:12 AM | File folder | |
| TestCpd01_jdx_Solv_MagFld | 4/3/2019 1:03 PM | File folder | |
| TestCpd01_TestInstr_Solv_MagFld | 4/3/2019 11:11 AM | File folder | |
| TestCpd01.mol | 11/13/2017 3:52 PM | Molecule Definition | 3 KB |
| TestCpd01_Annot_1HNMRspec_Solv_Magfld.pdf | 2/23/2018 3:29 PM | Adobe Acrobat D... | 48 KB |
| TestCpd01_Annot_1HNMRspec_Solv_Magfld.png | 12/7/2017 5:35 PM | PNG File | 59 KB |
| TestCpd01_Annot_13CNMRspec_Solv_Magfld.pdf | 2/23/2018 3:35 PM | Adobe Acrobat D... | 71 KB |
| TestCpd01_Annot_13CNMRspec_Solv_Magfld.png | 2/23/2018 3:25 PM | PNG File | 46 KB |

Note: HiFSA refers to $^{1}$H iterative full spin analysis, i.e the complete description of your NMR spin parameters detailing altogether the chemical shifts, coupling constants and line width of your $^{1}$H NMR spectrum.

- **Upload your NMR data on your created Harvard Dataset**


**To keep your Bruker raw NMR data together in one single folder you may need to double zip* your folder before uploading it. Otherwise a single zip is preferred for uploading your folder.**


## C. Step 4: Describe your data

**.....As important as your Overarching Metadata section.....**

1) **Describe the NMR information for your uploaded raw NMR data**

- Type of NMR spectroscopy (e.g. 1D and 2D)
- Type of NMR file (e.g. *.jdx, Bruker fid and *.jdf)
- NMR tube and Solvent (provider, batch, lot)
- Qty of compound analysed in the NMR tube
- Precise whether your compound has been isolated from natural source, synthetized, or whether it is a commercial compound
- Pubchem ID of structure (If already known compound)

2) **Describe the qNMR information for your uploaded quantitative analyses**

- Method used for purity determination : EC (External Calibration), IC (Internal Calibration) or 100% Methods. See related article here.
- Identity of the NMR calibrant, qty in the NMR tube
- Qty of compound in the NMR tube
- Volume of solvent in the tube
- Purity certificate of your chosen NMR calibrant (e.g. CRM or 100% method qHNMR spectrum
- Calculation spreadsheet utilized for the determination of purity
- Annotated NMR spectrum with integrales used for purity determination

➔ **You can also check a few examples of uploaded NMR datasets at :**
https://dataverse.harvard.edu/dataverse/cenaptnmr

## D. Final step: Publish your dataset!

**You can save a "draft" of your dataset before publishing it, and add gradually more information to metadata and data at a later time when they are ready.**

**Once everything has been uploaded (and your research article is accepted for publication) and you are happy with everything, you can push on the "publish" button.**

**Your NMR data can now be used and cited by other scientists thanks to the attributed DOI: you just have given a second life to your data, while benefiting the scientific community and enhancing your impact!**

**Congratulations !!**

## E. Some References


1. Choules, M. P., Bisson, J., Gao, W., Lankin, D. C., McAlpine, J. B., Niemitz, M., ... Pauli, G. F. (2019). Quality Control of Therapeutic Peptides by $^{1}$H NMR HiFSA Sequencing. *The Journal of Organic Chemistry*, *84*(6), 3055–3073. https://doi.org/10.1021/acs.joc.8b02704
2. McAlpine, J. B., Chen, S.-N., Kutateladze, A., MacMillan, J. B., Appendino, G., Barison, A., ... Pauli, G. F. (2019). The value of universally available raw NMR data for transparency, reproducibility, and integrity in natural product research. *Natural Product Reports*, *36*(1), 35–107. https://doi.org/10.1039/C7NP00064B
3. Bisson, J., Simmler, C., Chen, S.-N., Friesen, J. B., Lankin, D. C., McAlpine, J. B., & Pauli, G. F. (2016). Dissemination of original NMR data enhances reproducibility and integrity in chemical research. *Natural Product Reports*, *33*(9). https://doi.org/10.1039/c6np00022c
4. Pauli, G. F., Chen, S., Simmler, C., Lankin, D. C., Go, T., Jaki, B. U., ... Napolitano, J. G. (2014). Importance of Purity Evaluation and the Potential of Quantitative 1 H NMR as a Purity Assay. *Journal of Medicinal Chemistry*, *57*, 9220–9231. https://doi.org/10.1021/jm500734a
5. Wilkinson, M. D., Dumontier, M., Aalbersberg, Ij. J., Appleton, G., Axton, M., Baak, A., ... Mons, B. (2016). The FAIR Guiding Principles for scientific data management and stewardship. *Scientific Data*, *3*, 160018. Retrieved from https://doi.org/10.1038/sdata.2016.18
6. Wilkinson, M. D., Dumontier, M., Jan Aalbersberg, I., Appleton, G., Axton, M., Baak, A., ... Mons, B. (2019). Addendum: The FAIR Guiding Principles for scientific data management and stewardship. *Scientific Data*, *6*(1), 6. https://doi.org/10.1038/s41597-019-0009-6


***Zip or double Zip**, note from Mercè Crosas: the latest version of Dataverse maintains the folder structure when you upload the data in a zip file. If you want to take advantage of this feature, you don't want to double zip. Only if you have a very large number of files in the zip, double zip could have an advantage since extracting the folders and files from a zip could be a performance issue in some cases. If performance is not an issue, extracting the zip file into the folder structure is recommended for transparency and preservation.