ESPA p4ges PROJECT
Work Package Carbon (WP4-Carbon)

Manual for laboratory works and analyses

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The Laboratoire des RadioIsotopes (LRI) - University of Antananarivo is a research center which conducted researches on soil and plant dynamics in natural ecosystems and cultivated area. It is also a reference laboratory on physico-chemical analyses on soil and plant at national scale.

For this, the following analysis can be performed at the laboratory: soil handling and preparation (drying, grinding, sieving), soil physical analysis (soil texture, soil aggregation), soil chemical analysis (soil pH, soil organic carbon, total and available phosphorus, total and mineral nitrogen, cation exchange capacity, oxalate iron and aluminium, other elements as manganese, silica), and soil biological activity (carbon dioxide, nitrous oxide emission).

1- Sample registration and preparation

Sample preparation and analyses were performed in The Laboratoire des RadioIsotopes which is a laboratory dedicated for soil and plant chemical and physical analyses related to research work or public service delivery.

All samples collected in field were sent to the laboratory and were received and recorded by lab responsible for sample registration and storage.

Samples were then registered with the following information for sample tracking:

- Document number	ID number of the sample package.
- Person in charge	The name who was in charge of the sampling
- Program or project name	The title of the project in which the sampling activities are funded
- Sampling period	Dates of sampling
- Sampling site	Sites of sampling
- Sample type	Soil, plant or others (e.g. lowland soil, forest soil, rice leaf)
- Sampling method	e.g. by hand, core sampler…
- Sampling depth	Soil depth in which samples were collected
- Land cover or cropping system	e.g. forest, savanna, paddy field, agroforestry, rice-upland rotation
- Number of samples	The total number of all samples
- ID list of samples	Labelling for identification of samples (e.g. P01_001 to P01_090)
- Analysis requirement	e.g. Olsen P, NO_3/NH_4, SOC…

An ID number was generated for each sample and all specified information were recorded (e.g. depth of sampling, replication number, precise information of land-use or land cover, topography).

The list of recorded samples was then used to check each individual sample received in order to verify if all sampled listed are arrived in the laboratory. This checking was performed by the person in charge of sampling and the lab technician.

A final electronic list including concordance between the lab ID number and the field ID was generated.

2- Sample analysis

Soil samples for physico-chemical analysis were air-dried in a greenhouse. The dry soils were ground and sieved at 2 mm or 0.2 mm mesh according to the analysis method and bagged thereafter. The sieved soils were ready for the subsequent analysis stage.

Soil organic carbon analysis

Soil organic carbon content was assessed using chemical analyses on a selected samples and Mid-Infrared Spectrometry (MIRS) analyses on the whole samples.

For the chemical analyses, subsample (aliquot) of sieved soil will be weighted for the subsequent analyses. Subsequently, soil will be analyzed according to different standard method. Soil organic carbon (SOC) will be determined using Walkley and Black (1934) method which consist to wet oxidation using potassium dichromate in acid medium (sulfuric acid) followed by redox titration.

For the mid infrared spectrometry analyses, the sieved soils will be scanned using Mid-Infrared Spectroscopy (MIRS) (Agilent 4100 ExoScan FTIR (Danburry, Connecticut, USA)). Spectral data set from MIRS scanning of the selected soil (around 1/3 of total soil samples) will be used to develop prediction model with soil properties values from laboratory measurement. Accurate MIRS model will be used to predict the remaining (2/3 of total soil samples) soil properties.

Soil texture analysis

Soil texture was determined using chemical analyses on a selected samples and Mid-Infrared Spectrometry (MIRS) analyses on the whole samples.

For chemical analyses, subsamples of sieved soil were analyzed for soil particle size analysis based on separation of soil into various size fractions (Afnor, 1999). Organic matter will be removed from soil samples using hydrogen peroxide (H_2O_2) 30% oxidation. The soil–H_2O_2 mixture will be heated until complete evaporation of H_2O_2. Chemical dispersion solution (Sodium pyrophosphate) is added thereafter to help particle disaggregation. Sand fractions

will be determined by dry sieving while silt and clay fractions will be determined by pipette analysis.

For the mid infrared spectrometry analyses, the sieved soils will be scanned using Mid-Infrared Spectroscopy (MIRS) (Agilent 4100 ExoScan FTIR (Danburry, Connecticut, USA)). Spectral data set from MIRS scanning of the selected soil (n=132) were used to develop prediction model with soil texture values from laboratory measurement. Accurate MIRS model will be used to predict the remaining soil texture.

Bulk density and Natural ^{13}C abundance analysis

Soil bulk density was determined from dry soil weight after removing the coarse fraction >2 mm in the soil profile.

For other supplementary analysis as natural ^{13}C abundance ($^{13}\delta$), selected sieved soils (n=150) were sent to other specialized external laboratory for chemical analyses.

For the mid infrared spectrometry analyses, the sieved soils will be scanned using Mid-Infrared Spectroscopy (MIRS) (Agilent 4100 ExoScan FTIR (Danburry, Connecticut, USA)). Spectral data set from MIRS scanning of the selected soil (n=150) were used to develop prediction model with soil $^{13}\delta$ values from laboratory measurement. Accurate MIRS model will be used to predict the remaining soil $^{13}\delta$.

3- Data publication

All datasets which were generated by methodology process described within this manual have been also archived at Environmental Information Data Centre - EIDC (http://eidc.ceh.ac.uk). These obtained data were published or in process for publication, including the following papers:

- Andry Andriamananjara, Jennifer Hewson, Herintsitohaina Razakamanarivo, Andrisoa Riana Hary, Ranaivoson Ntsoa, Nantenaina Ramboatiana, Mieja Razafindrakoto, Nandrianina Ramifehiarivo, Marie Paule Razafimanantsoa, Lilia Rabeharisoa, Tahiana Ramananantoandro, Andriambolantsoa Rasolohery, Nantenaina Rabetokotany, Tantely Razafimbelo, 2016. Impact of land use changes on aboveground and soil carbon stocks in a humid tropical forest of Madagascar. Agriculture, Ecosystems and Environment. 233: 1–15

- N. Ranaivoson, A. Andriamananjara, T. Razafimbelo, J. Hewson, A. Rasolohery, R. H. Andrisoa, M. A. Razafindrakoto, N. Ramifehiarivo, M. P. Razafimanantsoa, N. Rabetokotany, R. H. Razakamanarivo. Toward a better understanding of soil organic carbon variability in eastern humid region of Madagascar. Under review in European Journal of Soil Science.

- Mieja Razafindrakoto, Andry Andriamananjara, Tantely Razafimbelo, Jennifer Hewson, Riana Hary Andrisoa, Julia Jones, H.J. Ilja van Meerveld, Alison Cameron, Ntsoa Ranaivoson,

Nandrianina Ramifehiarivo, Nantenaina Ramboatiana, Ravo Nantenaina Gabriella Razafinarivo, Tahiana Ramananantoandro, Andriambolantsoa Rasolohery, Marie Paule Razafimanantsoa, Christophe Jourdan, Laurent Saint-André, Gabrielle Rajoelison, Herintsitohaina Razakamanarivo. Organic carbon stocks in all pools following land cover change in rainforest of Madagascar. In: Munoz M.A., Zornoza R., Editors. *Soil Management and Climate Change: Effects on Organic Carbon, Nitrogen Dynamics, and Greenhouse Gas Emissions*, Elsevier. Under review.

References:

AFNOR. (1999). Recueil de normes: qualité des sols. Paris: Association Française de Normalisation.

Walkley, A., Black, J. 1934. An examination of the Degtjareff method for determining soil organic matter and a proposed modification of the chromic titration method. Soil Sci. 37 (1), 29–38.