ECORESPIRA-AMAZON
HANDBOOK v.2

The **ECORESPIRA-AMAZON HANDBOOK** guides you through the practical aspects of the project. Introduced by a very short project philosophy, sampling procedures and sample treatment are described, followed by selected aspects of sample preparation prior to analysis. This handbook does neither replace your communication with supervisors (professors, lab technicians) nor your personal discussions, questions, etc. This handbook has been discussed and explained in detail to all participants at our workshop in Manaus prior to the first field campaign in February 2016 (and was modified subsequently = v.2). It is of critical importance that every team member has a clear understanding of all procedures.

The **ECORESPIRA-AMAZON PROJECT** is highly ambitious for two reasons. Despite many previous studies, the Amazon biome is still largely less well-known territory. High average air temperatures combined with very high average air humidity strains people and material. Therefore, tiredness and exhaustion may easily occur during fieldwork – and must not compromise the quality and diligence in sampling and sample preparation. The environmental conditions are also hard on materials, electronics and mechanics – with resulting potential issues in malfunctioning and high maintenance demand. Whatever is not achieved in the field cannot be compensated for later. The second aspect relates to the topic itself – soil respiration and soil chemistry. Given the vast dimensions of the Amazon basin it is almost impossible to cover the region with any higher sampling density. Therefore, the question of representativity is paramount in both pool estimates (soil chemistry) and dynamics (soil respiration) since results shall be upscaled later. See http://blogs.hrz.tu-freiberg.de/ecorespira

RESPONSIBLE: Jörg Matschullat **STATUS**: May 1, 2016

CONTENT

The **annex** to this handbook is available as a separate file

Soil respiration determination in an Acai plantation (Loc 07) with Laura Medeiros Braga, Gilvan Coimbra Martins and Edinilson Alves Figueiredo (f.l.t.r.), phase 1 of the project

1 PROJECT PHILOSOPHY

The project is largely defined by the following research questions – and their implications

- What are soil respiration (ecosystem respiration) rates in the Amazon basin?
- Does soil respiration change after deforestation and change of land cover? If so, how?
- Is there a relationship between soil carbon and nitrogen pools and respiration fluxes?
- How do those pools and the respiration rates relate to comparable land-cover types outside of the inner wet tropics?
- What does soil respiration in the Amazon reveal about organic matter turnover rates and are those results in line with previous independent studies?
- How does the pedogeochemistry look like in the Amazon basin?
- Does the geochemical composition of the different soils at the intended sites and transects show rather narrow distribution?
- Are there unusual element concentrations in certain major, minor or trace elements?
- How do the new data compare to older ones, e.g. by Georg Irion?

EcoRespira-Amazon is the first Brazilian-German project attempting to determine soil respiration and soil chemistry along smaller transects that represent various types of land cover – from original primary forest to subsequent agricultural use. The pilot project, having started with fieldwork in February/March 2016 aimed at four locations in the central Amazon region near Manaus and another five locations in the southern reaches of the basin in the state of Amazonia. The first four locations also served as training ground assessing measuring and sampling time as well as team building. The now eleven locations will be re-visited at least twice until April 2017 to repeat the sampling procedures at all sites to build a more robust and reliable database for the soil respiration data.

The very fast turnover times of organic matter in inner tropical rainforest environments (ca. 2.5 times per year) and the particular ecological role of the Amazon forest biome (water recycling, oxygen production, biodiversity hotspot, etc.) motivate this project beyond its known vulnerability. Recent European experience has shown that modelling does not yet serve to give satisfactory answers neither on element pool sizes nor on soil and ecosystem respiration. In this joint project with Brazilian (Embrapa, IPAAM, UFAM) and German (TUBAF) scientists, both topics are targeted and shall complement the longer-term commitment of the Brazilian team. Selected field sites that serve Embrapa long-term observation tasks shall be visited and sampled at least three times from February 2016 to April 2017. Ideally, the Brazilian partners will organize additional measurement series in between to obtain an even more robust database.

In principle, the EcoRespira-Amazon project follows ideas and methods applied over the last ten to 20 years in various projects around the world. The soil respiration determination largely draws upon experience gained by the TUBAF team since 2004 in the GREGASO project and its predecessors. The soil investigations draw from the ecogeochemical mapping of Kola peninsula (Reimann et al. 1998), the Baltic Soil Survey (Reimann et al. 2003), the new European GEMAS project (http://www.ngu.no/no/hm/-

Publikasjoner/Rapporter/2008/2008-038/), the USGS mapping project (http://tin.er.usgs.gov/geochem/doc/home.htm), the Australian Geochemical Mapping project (http://www.ga.gov.au/ausgeonews/ausgeonews200706/geochemical.jsp) the BraSol-2010 project (Matschullat 2012; Matschullat et al. 2012; Schucknecht et al. 2012) and the VeLuDeClim project (Erasmi et al. 2014; Schucknecht et al. 2013). EcoRespira-Amazon is in active exchange with the related groups and contributes to the UN International Year of Soils in 2015 and the International Geochemical Mapping Project (IGMP).
Composite soil samples have been taken from eleven locations representing the local land cover with a bias towards secondary forest and post-forest land use. Each location includes (primary/secondary) forest, forest edge and a variety of modified land cover following deforestation (agriculture, agroforestry, pastureland). Care has been taken to include the relevant geological (lithological) units and all typical soil types. Plant community and other eco-environmental information, including weather conditions are being logged on site, wherever possible.

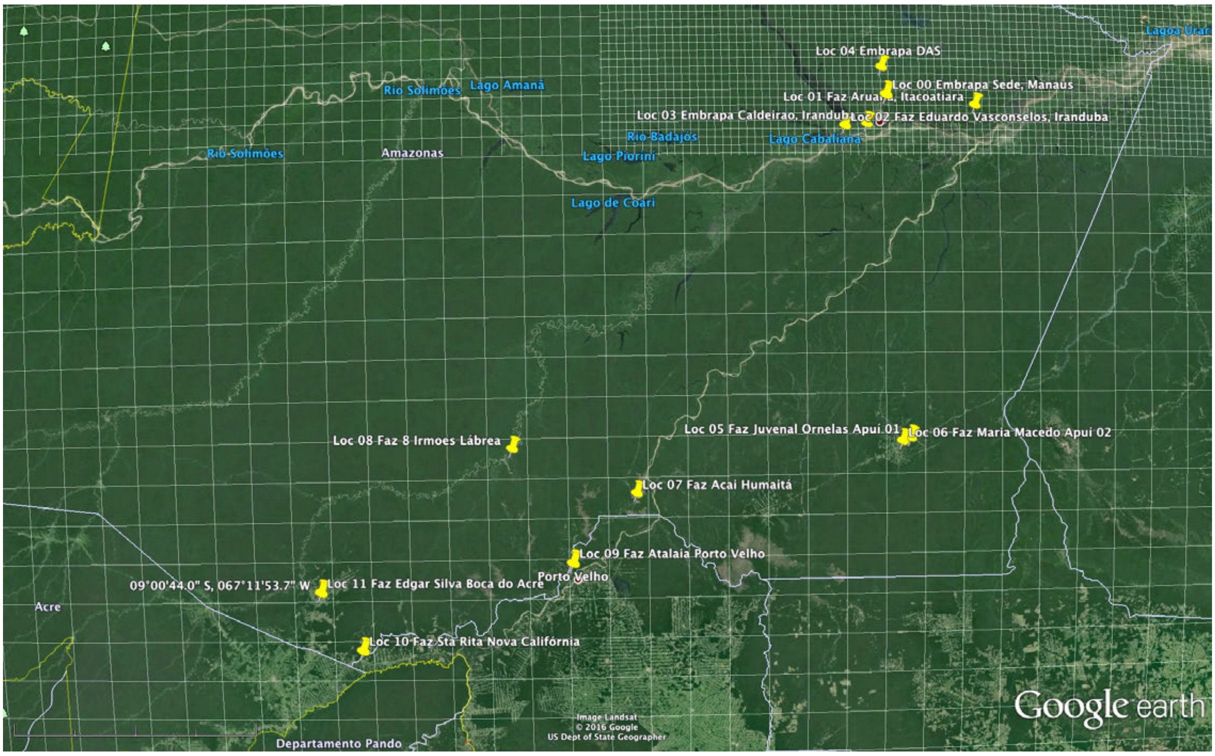


Figure 1. Satellite image of the section of the Amazon basin with the eleven sampling locations (Google Earth). See Logbook EcoRespira-Amazon, phase 1 for more details. Each location encompasses forest and non-forest land cover

Three types of composite soil samples have been taken at each site of every location: the litter layer with its high organic matter content (**ORG**), a surface mineral soil sample (**TOP: 0—20 cm**) and a bottom soil sample (**BOT: 30—50 cm**), both largely representing the prevalent root zone. Each location represents two to three sites with different land cover. Each composite sample consists of original samples at three sub-sites (table 2 in the EcoRespira logbook, phase 1) for each site. Those sites have a size of roughly 10,000 m^2 (≈ 100 x 100 m, 1 ha). The composite samples were split (aliquots) in the Embrapa soil laboratory by our team, and are being analysed for most of the periodic system, including high resolution CNS in Freiberg. Additional analysis is performed in the Embrapa soil laboratories in Manaus. Results are constantly being updated and can be found on the project web site (**http://blogs.hrz.tu-freiberg.de/ecorespira**).

At each sub-site, and following site preparation with PVC tubes, soil respiration/ecosystem respiration is to be measured directly (CO_2) and gas samples to be taken for subsequent laboratory analysis (CH_4 and N_2O by gas chromatography in Freiberg). This, together with other data logged in parallel, allows calculating soil gas flux rates and will thus contribute to a better understanding of sink or source qualities of land-use and land-cover types – a contribution to climate models and to sustainable management practices of the biome.

Both types of information are indispensable for any kind of planning and decision-making to support sustainable long-term development. Without such knowledge, recommendations for land use (agriculture, forestry, urbanization etc.) may easily and irreplaceably compromise the most valuable natural resource soil. Another highly important product is geochemical "background" data for the individual lithologies, soil and land use types. Such baseline data are needed to assess any type of large-scale environmental change. Related data are currently not available, out-dated or not representative. Data, e.g. as those by Irion (1976, 1978) may well be out-dated in respect to trace elements, given the analytical capabilities of that time.

Further purposes in this project relate to knowledge transfer in general. The bi-national project has started training students, post-graduates as well as post-docs from Brazil and Germany and will give ample opportunity to further increase experience and knowledge. Concrete questions are related to our understanding of weathering, alteration of soil chemistry through management practices under tropical conditions, etc. The detection of geochemical anomalies in unknown areas also relates to the land-use (change) aspect.

Our project is a non-commercial, scientific project, executed by Brazilian and German university and state institutional members – students and staff. All results are free and may be accessed upon availability on our project website or by contacting the project representatives in Brazil (Prof. Dr. Celso Azevedo at UFAM, Dr. Roberval Monteiro Bezerra De Lima at Embrapa, Dr. Kikue Muroya at IPAAM; all Manaus, Brazil) or in Germany (Prof. Dr. Jörg Matschullat at TUBAF, Freiberg).

The project is part of NoPa2 (Novas Parcerias), a joint initiative by the German and the Brazilian government, financed through the German Federal Ministry for Economic Cooperation and Development, and the Brazilian government through CAPES. CAPES, DAAD and GIZ jointly organize and oversee the project progress.

The rainy season poses particular challenges and even the best driver can get stuck. This will not happen deeper within a forest, yet other surprises and encounters wait therein …

2 ITEM CHECKLIST FOR FIELD EQUIPMENT in July/August 2016

General items, documentation and data storage

1. This project handbook (ideally one per person)
2. Field books with appropriate pens (one per person)
3. Four (4) handheld GPS receivers Garmin (one per person)
4. One (1) digital camera (min. 1) with spare batteries and chargers (Olympus OMD-E with lenses and cleaning equipment)
5. One (1) digital HD movie camera (Panasonic)
6. One (1) stereo field microphone for nature sound recordings
7. Two (2) plasticized sample site numbering sheets
8. Five (5) plasticized scales (20 cm) with colour control bar (1 per person)
9. Three (3) waterproof site marker pens
10. Two (2) Laptop computers, one Panasonic Toughbook
11. One (1) external hard disc (1 Tb) for data back-up
12. One (1) multi-adapter plug/socket
13. One (1) solar panel multi charger (Revolt) with multi adapters

Soil sampling

14. Two (2) TDR probes (Delta Devices) for sub-site soil humidity determination
15. Disposable gloves, metal free (2 boxes with medium and large size)
16. One (1) Sondaterra Dutch soil auger set with drill head in transport bag
17. RILSAN sampling bags (300 pcs) with locking ties and paper marking boards
18. Cotton sample bags for **ORG** samples and plant material
19. Ten (10) waterproof markers to label vials, sample bags etc.
20. One (1) hygrothermometer (Testotherm) for air humidity and temperature

Gas sampling

21. 24 PVC rings KGU DN250
22. Two SEMACH-FG chamber systems with chamber bodies, including various built-in and attached sensors (all in transport box) and the separate steering units with data logger, operating software and battery back-up and chargers (Peli boxes)
23. 500 evacuated, doubly-sealed gas vials (Exetainer) 5.9 mL, soda glass, flat bottom, labelled with double septum in Peli box
24. 15 syringes with 20 metal needles

3 GENERAL FIELDWORK PREPARATIONS

- In preparation for the sampling campaign, all members of the field team have read this handbook and acknowledged to fully understand the information. Should any question arise at any time, this is to be announced to the accompanying supervisors and answered by them on the spot.
- From October 2015 to February 2016, the German participants have participated at a preparatory seminar under the guidance of Prof. Jörg Matschullat. They were trained on a theoretical basis to prepare for the boundary conditions in Amazônia and have read and discussed most details of the forthcoming expedition. Laura Medeiros Braga gave an important talk about Brazilian perception of the Amazon region.
- As of the successful field campaign in February/March 2016, Laura Medeiros (UFB), Sophie von Fromm and Jörg Matschullat (TUBAF) bear the necessary experience and will train Carolin Schröder and Thomas Drauschke for the second field campaign.
- Every day, before leaving the respective "camp", the materials checklist (p. 6) is controlled and all items that are foreseen to be needed during the day for the sampling campaign packed for completeness and easy access.
- Every person is responsible for proper personal gear. Responsibility for specific tasks will be allocated during the workshop in Manaus.
- After finishing the daily fieldwork, all equipment is to be cleaned and checked for proper functioning. This includes battery checks, charging and organization of a stock of fresh batteries where needed. The other important task to be fulfilled daily is finishing the field protocols – checking for completeness, and for general understanding of the sites (exchange of observations, experiences). All photographs or videos related to the project will be uploaded daily to two independent storage media to be taken care of by the supervisors.

Nothing like a great team when the going gets tough. Bruno Scarazatti, Joel Gomes, Laura Medeiros Braga and Sophie von Fromm (f.l.t.r.) pack up the equipment to be transported to another site (1st field campaign 2016)

4 SAMPLING

General. The sampling campaign follows a predefined path, including a definition of places where the field teams may stay overnight or have a lunch break. Figure 1 shows the locations, where work will be undertaken; Figures 2–10 in the Annex show close-ups of each location.

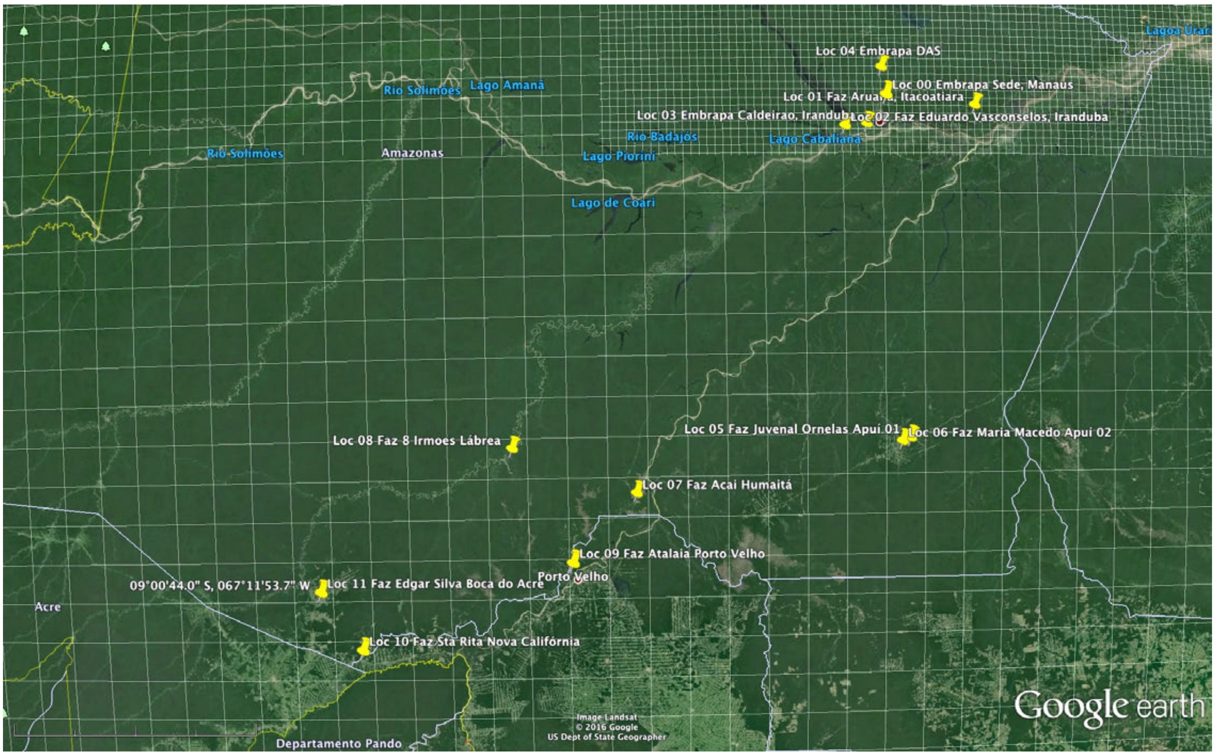


Figure 1. Satellite image of the section of the Amazon basin with the eleven sampling locations (Google Earth). See Logbook EcoRespira-Amazon, phase 1 for more details. Each location encompasses forest and non-forest land cover

In general, and in each sampling campaign, the four sites near Manaus shall be sampled first. This helps new team members to become acquainted with boundary conditions. Since Manaus is within easy reach, possible mistakes can more easily be compensated for. Yet, we do not wish to make mistakes.

At each site. The following procedure will be followed since sites are now defined:

a) Short introduction for the new team members to each (sub) site.

b) Deployment of PVC rings for the chamber with precise (as possible) GPS coordinates, trying to retrace the February/March 2016 positions.

c) Soil samples might be taken – depending on lab results for the Feb/March samples.

d) About 24 hours later, gas measurements and samples with the chambers

Almost two days are required per location.

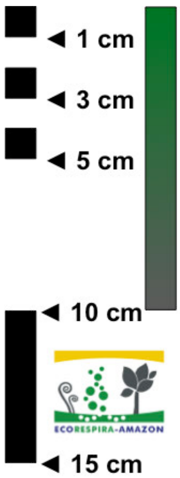
Work Steps. Upon arrival at a sampling (sub) site, the following work is to be done in this sequence:

I. The geographical position will be taken by at least two hand-held GPS receivers independently but at the same position. These positions will be documented as well as the number of satellites and the ground resolution. All GPS will be taking data based on WGS 84 – this is binding and has to be checked (settings on the GPS receivers). The position is to be marked as precise as possible onto the geographical maps – ideally repeating the Feb/Mar 2016 positions.

II. Digital overview photographs will be taken with the individual site number clearly in the foreground (plasticized numbering). This helps to identify all pictures unmistakably for each site.

III. The field team splits into pre-defined sub-groups – each with at least two persons and each doing its particular task on the site:
   - The team defines sub-sampling positions for soils sampling and set-up of the PVC rings for subsequent gas flux measurements.
   - One sub-team performs the sampling procedure for the three soil composite samples from each sub-site.
   - A second sub-team sets the PVC rings and prepares those sub-sites for gas flux measurements on the following day.
   - A third sub-team (if available) performs a rough "mapping and documentation" procedure for each site, registering vegetation and land use, including photo-documentation.

4.1 Site description. Each site is to be described as precise as possible:



Photographic documentation (always with a scale – a person for larger, landscape-like pictures, the EcoRespira-Amazon scale for close-up, right). This documentation demands writing explanatory notes for each photograph, including direction of photograph, representativeness of the object, etc. A minimum of six photographs is to be taken from each site: 1) general overview with site code number, 2) vegetation, 3) landscape, and 4–6) soil samples with provided scale. Great care has to be taken with technical quality of the photos. Check your camera setting for high-resolution images, proper white balance and ISO. Ideally check each picture straight after taking it so that you can reproduce it if need be.

A written description of the typical land use of the sampling site and its surroundings is needed with as much detail as possible (this may include e.g., meteorological conditions, plant communities, forest type, observed animals, availability and access to open water bodies, etc.).

Left: Photo documentation is not necessarily a light-hearted affair – although it may look like it (Gilvan Coimbra Martins in action)

4.2 Soil sampling

Soil samples have been taken successfully at all sites. To check reproducibility, individual repetitions appear useful, ideally only at sites near Manaus to reduce expensive overweight in the transport to and from Porto Velho, Rondônia. Samples will be taken with a Sondaterra soil auger after removing the litter layer carefully with lab glove-protected hands. Litter material is collected separately; see below. The samplers will have to wear disposable gloves whenever handling soil material. No finger rings or other jewellery is to be worn by anyone performing sampling and having potential direct or indirect sample contact (e.g., bending over sample with sweat dropping down etc.).

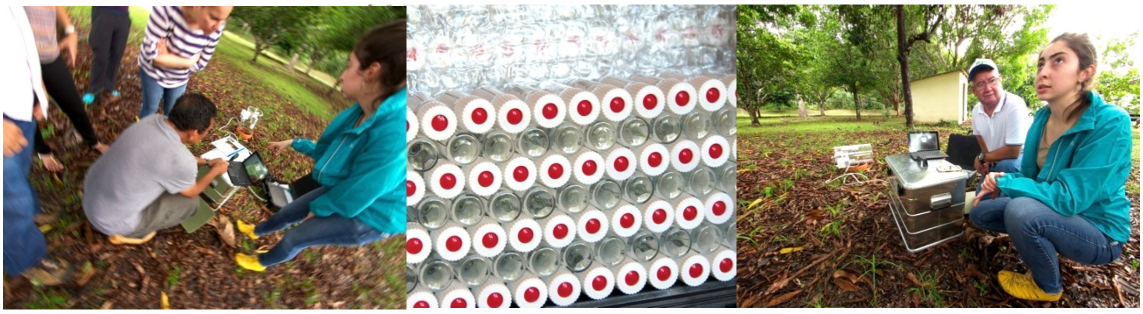
- Step 1: the organic debris (litter) etc. is sampled by hand at each site, representative for the entire site. Only larger living plants, stones and larger roots are to be removed from the sample. This potentially most organic-rich material shall be transferred to a cotton bag.
- Step 2: the first approximately 20 cm are drilled using one of the pre-cleaned Sondaterra soil augers. The auger is cleaned prior to any sampling by drilling nearby the final drill site to the desired depth, discarding that sample and manually cleaning the auger before taking the "real" sample. Small visible stones or larger roots are to be discarded, too. The remaining "good" mineral soil material is to be filled into pre-coded RILSAN® sampling bags. Marking the RILSAN bags shall exclusively be done with a waterproof marker. In addition, a cardboard batch with the same code written with a high-quality ball-pen sealed in a PE-Ziploc bag shall be inserted into each RILSAN bag prior to final closure. Close the bag tightly with the provided strips (!Watch, bags cannot be opened thereafter until arrival in Manaus!).
- Step 3: drill to 30 cm depth and discard that material (later to fill the hole).
- Step 4: each sampling hole shall be "overdrilled" to the final depth of about 50 cm (watch marking on drill rod). The next steps follow the same procedure. Care has to be taken that the coded RILSAN bag does prevent misunderstandings (sample mix-up).
- Step 5: after having retrieved the last sample from each of the sub-sites, each site will be "cleaned", refilling excess material into the dug hole and camouflaging the sub-sites with organic litter material to prevent accidents. This last step shall not serve beauty, but rather follow the codex: "Take only memories, leave only footsteps". While we take samples, we shall not make this very visible to avoid any kind of impression that we are making a mess or destructive holes that could injure animals.
- Step 6: after each of the three sampling steps, the full and marked RILSAN bag will be transported into a vehicle and carefully stored. It is very important to check the bags closures for a very tight fit prior to storing them away in the vehicle. This last step will be registered in the site protocol, too.

Left: Soil sampling with an auger (José Maria and Jörg Matschullat); storage and transport in Rilsan bags; Right: Soil profile with Terra preta do Indio (Indigenous black earths) at location 03

4.3 Gas sampling (soil and ecosystem respiration)

For operating the SEMACH-FG dynamic closed chambers systems for gas sampling, please refer to the respective manual for the operation of the SEMACH-FG and SEACH-FG chamber systems.

A first gas sample is to be taken right at the beginning of the CO_2 measurements (sample 0 min). After the CO_2 measurements are finished for each sub site and a subsequent chamber “cleaning” to get back to initial CO_2 concentrations, a 30 min gas sampling starts with samples taken in 5-minute intervals. Using the luer-lock interface on top of the chamber, an appropriate 12 mL disposable syringe and needle, samples are taken and injected into Exetainer® vials. After each location, syringe and needle are discarded and a new pair used for the next location.



Left: At first, the Earth seems to start twirling, since there is so much to remember and take care of. Center: Yet, there is order behind the perceived chaos (Exetainer® vials). Right: And with the right teacher (Laura Medeiros Braga), the most complex issue becomes reasonably easy to tackle.

4.4 Plant sampling

- In addition to the ORG samples, fresh plant material, preferably leaves from the same species shall be sampled at each sampling location, preferably in the forest only (= **PLT** sample). It is mandatory to sample this material with disposable gloves.
- Similar to the soil samples (ORG, TOP, BOT), the plant samples need to be documented in the field (genera or species identification where possible. Ideally, leaves from a tree should be sampled rather than grass or shrub vegetation.
- The leaf material is to be sampled from the entire site into a cotton bag, and registered with the site code and a sequential PLT number.
- Ideally, the samples shall lie in the unclosed bag until the group progresses to the next site. The reason being that some air-drying effect can take place and sample decay can be minimized.
- Photographic plant documentation is mandatory (overview and close-up).

5 SAMPLE PREPARATION AND ANALYSIS IN THE LABORATORY

Summary of analytical procedures for soil characterization (WRB 2007)

This annex is mostly taken from WRB (2007) and has not been screened in respect to EcoRespira-Amazon. Yet, additional points have been added. It provides summaries of recommended analytical procedures to be used for soil characterization for the World Reference Base for Soil Resources. Full descriptions can be found in *Procedures for soil analysis* (Van Reeuwijk 2006) and the USDA *Soil Survey Laboratory Methods Manual* (Burt 2004). ➔ Not all procedures will be applied in the EcoRespira-Amazon project.

5.1 Sample preparation (to be applied)

Samples are air-dried or, alternatively, oven-dried at a maximum of 25°C to reserve e.g., mercury (Hg). The fine fraction is obtained by sieving the dry soil sample with a 2 mm sieve. Clods not passing through the sieve are crushed (not ground) and sieved again. Gravel, rock fragments, larger root parts, etc. not passing through the sieve are treated separately.

5.2 Soil physical parameters

Particle-size analysis. The mineral part of the soil is separated into various size fractions and the proportion of these fractions is determined. The determination comprises all material, i.e. including gravel and coarser material, but the procedure itself is applied to the fine earth (< 2 mm) only.

Water-dispersible clay. This is the clay content found when the sample is dispersed with water without any pre-treatment to remove cementing compounds and without use of a dispersing agent. The proportion of natural clay to total clay can be used as a structure stability indicator. ➔ We may not apply this method.

Bulk density. Soil bulk density is the mass per unit volume of soil. As bulk density changes with water content, the water status of the sample must be specified. Two different procedures can be used:

Undisturbed core samples. A metal cylinder of known volume is pressed into the soil. The moist sample mass is recorded. This may be the field-moist state or the state after equilibrating the sample at a specified water tension. The sample is then oven-dried and weighed again. The bulk density is the ratio of dry mass to volume at the determined water content and/or the specified water tension.

Coated clods. Field-occurring clods are coated with plastic lacquer (e.g., Saran dissolved in methyl ethyl ketone) to allow determination of underwater mass. This gives the volume of the clod. The moist sample mass is recorded. This may be the field-moist state or the state after equilibrating the clod at specified water suction. The sample is then oven-dried and weighed again. The bulk density is the ratio of dry mass to volume at the specified water suction.

Note: The determination of bulk density is very sensitive to errors, particularly caused by non-representativeness of the samples (stones, cracks, roots, etc.). Therefore, determinations should always be made in triplicate. ➔ We may not apply this procedure in EcoRespira-Amazon.

5.3 Soil (physico)chemical parameters

pH-value (to be determined in the lab). The pH of the soil is potentiometrically measured in the supernatant suspension of a 1:2½ soil:liquid mixture. The liquid is either distilled water (pH_{H2O}) or a 1 M KCl solution (pH_{KCl}). To save valuable field time, we will perform pH-determinations in the lab.

Cation exchange capacity (CEC) and exchangeable bases (to be done). The ammonium acetate pH 7 method is used. The sample is percolated with ammonium acetate (pH 7) and the bases are measured in the percolate. The sample is subsequently percolated with sodium acetate (pH 7), the excess salt is then removed and the adsorbed Na exchanged by percolation with ammonium acetate (pH 7). The Na in this percolate is a measure for the CEC. Alternatively, after percolation with ammonium acetate, the sample can be washed free of excess salt, the whole sample distilled and the evolved ammonia determined. Percolation in tubes may also be replaced by shaking in flasks. Each extraction must be repeated three times and the three extracts should be combined for analysis.

Note 1: Other procedures for CEC may be used provided the determination is done at pH 7.

Note 2: In special cases where CEC is not a diagnostic criterion, e.g., saline and alkaline soils, the CEC may be determined at pH 8.2.

Note 3: The base saturation of saline, calcareous and gypsiferous soils can be considered to be 100 percent.

Note 4: Where low-activity clays are involved, the CEC of the organic matter has to be deducted. This can be done by the graphical method (FAO 1966), or by analysing the CEC of the organic matter or the mineral colloids separately.

Cation exchange capacity should certainly be determined in the EcoRespira-Amazon project, likely in the lab in Freiberg.

Exchangeable acidity. This is the acidity (H + Al) released upon exchange by an unbuffered 1 M KCl solution. It may also be designated actual acidity (as opposed to potential or extractable acidity). It is used to determine the so-called effective cation exchange capacity (ECEC) defined as: sum of bases + (H + Al), with bases being determined by ammonium acetate extraction. When the exchangeable acidity is substantial, the Al may be determined separately in the extract as it may be toxic to plants.

Note: Because the contribution of H^+ is often negligible, some laboratories only determine exchangeable Al. In that case, the ECEC is calculated as: *sum of bases + Al.*

Extractable iron (Fe), aluminium (Al), manganese (Mn) and silicon (Si). These analyses comprise:

- *Free* Fe, Al and Mn compounds in the soil extracted by a dithionite-citrate solution. (Both the *Mehra and Jackson* and *Holmgren* procedures may be used)
- *Active*, *short-range-order* or *amorphous* Fe, Al and Si compounds extracted by an acid oxalate solution.
- *Organically bound* Fe and Al extracted by a pyrophosphate solution.

Salinity. Attributes associated with salinity in soils are determined in the saturation extract. The attributes include: pH, electrical conductivity (EC), sodium adsorption ratio (SAR) and the cations and anions of the dissolved salts. These include Ca, Mg, Na, K, carbonate and bicarbonate, chloride, nitrate and sulphate. The SAR and the exchangeable sodium percentage (ESP) may be estimated from the concentrations of the dissolved cations.

Phosphate retention. The Blakemore procedure is used. The sample is equilibrated with a phosphate solution at pH 4.6 and the proportion of phosphate withdrawn from solution is determined.

X-ray diffractometry (XRD). The clay fraction is separated from the fine earth and deposited in an oriented fashion on glass slides or porous ceramic plates to be analysed on an X-ray diffractometer. Unoriented powder specimens of clay and other fractions are analysed on the same apparatus or with a Guinier X-ray camera (photographs).

Elemental analysis (CNS). Completely homogenized sample aliquots (<63 µm) are used (20 mg per sample) and mixed with 60 mg of tungsten$^{(VI)}$oxide (as oxidant) in a tin-foil sample container. This carefully sealed container is thereafter placed in a sample carousel of the El Cube Elemental analyser (Elementar, Germany) to determine the concentrations of total carbon, total nitrogen and total sulphur.

To obtain the concentration of organic carbon (C_{org}), another aliquot needs to be treated with a drop of 10 % HCl solution. After the gas release (CO_2), the sample will be treated as before and the resulting C-concentration equals the amount of C_{org}, prerequisite to calculate the C/N ratios. ➔ This is an EcoRespira-Amazon procedure.

Loss on ignition (LOI). To determine the volatiles, a powdered sample aliquot is weighed in precisely into a porcelain beaker. The sample is then to be heated in a muffle oven at a temperature of 900°C. After cooling to room temperature, the sample is weighed once again. The weight difference equals the amount of volatiles.

Total element concentrations (WD-XRF). The powdered sample (<63 µm) will undergo two steps of analysis with wavelength-dispersive X-ray fluorescence analysis (WD-XRF).

a) Glass fusion disks. A precise amount of roughly 100 mg inweight is thoroughly mixed with a defined amount of lithium tetraborate and filled into platinum vessels. This powder mixture is to be heated to a completely homogenous melt and then filled into platinum receptacles. The resulting glass fusion disk is rapidly cooled and labelled on its rougher side. This sample will then be analysed by WD-XRF for major and minor and some trace elements (the volatiles are gone). The amount of volatiles is put individually per sample into the program and (depending on the total concentration of each element in a specific sample) the total concentrations from lithium (Li) to uranium (U) will be determined quantitatively. A precise instrument calibration, appropriate reference materials and other lab quality control procedures are paramount.

b) Pressed powder pellets. Again, a precise amount of powdered sample (ca. 1 g) is mixed thoroughly with a wax (ca. 5 g). This admixture is then pressed into a solid powder pellet with a pressure of 20 tons per square centimetre. The resulting pellet can then be used directly in the WD-XRF to determine the total concentrations of all elements, including the volatiles. Mind that highly volatile elements such as mercury (Hg) may be underdetermined due to Hg-losses in the entire chain from sampling to analysis.

➔ These are EcoRespira-Amazon procedures.

Trace and ultratrace-element concentrations (ICP-MS). Following a multi-acid full digestion of sample aliquots (ca. 100 mg each), the diluted and acid-free fumed aqueous solution is analysed by inductively-coupled plasma mass-spectrometry (ICP-MS). The very low limits of determination permit a quantification of even minute element concentrations. ➔ This is an EcoRespira-Amazon procedure.

Below: What may appear as cloudy still, will soon give way to high-quality data, since the sampling campaign of project phase 1 has been performed in a most rigorous and successful manner.

6 MISCELLANEOUS

The project web site **http://blogs.hrz.tu-freiberg.de/ecorespira** will continually be updated and serve for public information, for internal information exchange, and as a platform to communicate about the project.

In Case of Emergency: you might want to contact the national representatives (German Embassy in Brasília and consulate in Manaus):

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Variable cracker butterfly (Hamadryas feronia) resting on a tree bark – one of very many species to be encountered especially within the forest environments

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ANNEX

The annex to this document is available as a separate document upon request.

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