

## Status report of the Trondheim Radiocarbon laboratory

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## **ABSTRACT**

The Trondheim radiocarbon laboratory has evolved from a traditional radiocarbon decay counting laboratory to an accelerator mass spectrometry (AMS) facility primarily measuring  $^{14}\text{C}$ . This evolution required adjustments in sample preparation and data handling to match the capacity of the AMS system and thousandfold reduction in sample sizes. We summarize here the steps involved in dating a sample at the National Laboratory for Age Determination in Trondheim, Norway. These include the structure of our sample database for information handling, sample cleaning procedures for different sample types, our reduction systems, both an automated EA-based system for regular use and a manual system for more challenging samples, and data evaluation. We will also briefly summarize the capabilities of our isotope-ratio mass spectrometer.

## **INTRODUCTION**

The radiocarbon laboratory in Trondheim has a long history starting in the 1950s with the construction of gas proportional counters (GPC) (Nydal 1959; Nydal 1962). Throughout the years, the laboratory has been active in many research fields such as atmospheric research (Nydal 1968), oceanography (Nydal et al. 1992), and archaeology (Nordeide and Gulliksen 2007). With more sensitive measurement techniques requiring much less sample material becoming available, an accelerator mass spectrometer (AMS) was commissioned and officially accepted in May 2009 (Nadeau et al. 2015). Since then, the sample preparation protocols and systems were upgraded to match the increased sample throughput of the new system.

The most important requirement was the need for reduction systems to convert CO<sub>2</sub> samples into graphitic carbon. Yet, also the smaller sample amounts and higher sample throughput needed to be addressed. Here, we present the status of the ongoing development and optimization process to fit the new needs. This covers the data handling within the laboratory as well as processes for chemical sample cleaning, reduction of CO<sub>2</sub> to graphite, target pressing, <sup>14</sup>C measurement, and data evaluation.

## **DATABASE**

When processing large numbers of samples, the flow of information in the laboratory is of key importance to track the samples throughout the treatment and measurement. A relational database provides a powerful tool to collect and distribute information in many

ways, including both manual handling as well as automated communication with instruments. Our database is constructed in two layers. The tables, presently included in a Microsoft Access file, could also be hosted by a database server and accessed via ODBC (open database connectivity) or other communication protocols. User interfaces constitute the second layer of the structure. These are linked to the underlying table-layer but remain independent. Depending on the needs, user interfaces can be designed for a single process (e.g. chemical pre-treatment) or as overviews to track samples through the laboratory.

The database table structure reflects the processes to which a sample is subjected through the laboratory (Fig. 1). The user table contains information about customers, such as addresses. Sample descriptions, names and deadlines are included in the sample table. The pre-treatment table describes the chemical cleaning that was carried out to obtain the datable fractions that are stored in the fraction table. The CO<sub>2</sub> table is used for information about the combustion process, such as weighed-in mass or carbon content. The reduction table stores data such as starting and end pressures of the reaction. The target table's main content is the identifying holder number of the AMS cathode and linking it to the sample. Finally, the result table contains the evaluated results from the measurement. In every step, the one-to-many option that allows multiple continuations of the process, e.g. several different pre-treatment sessions for a single sample or combustions of a prepared fraction. The main tables have a direct link to the sample table, which allows the omission of "missing steps", e.g. there is no need to enter preparation, combustion, and reduction records when preparing machine blanks from a graphite sample.

Data entries about chemical pre-treatment, part of the information regarding combustions, and target pressing are made manually through a Microsoft Access user interface. Different

queries and forms have been developed to have overviews of samples at the different preparation steps. A label printer allows fast printing of identification labels to keep track of the samples during the treatment in the laboratory. The labels include barcodes and the implementation of barcode readers is planned. It is also possible to export data to other systems, such as batch files for the AMS system, or to create result reports. Some programs, such as the control software of the reduction systems (see section 4), automatically connect to the database in order to retrieve sample information and to upload process data, e.g. the carbon content of a sample from the elemental analyzer. The data evaluation process uses a database connection to read sample information from the database, such as the sample material to apply the correct blank correction.

The information provided for all preparation steps is set so that it allows to check the data of the previous step. This is an important part of quality control, assuring that samples are not exchanged during handling in the laboratory and that the results are finally assigned to the correct sample.

## **SAMPLE HANDLING**

A sample submitted to our laboratory will first be registered under a TRa-number (TRa = Trondheim Radiocarbon AMS) in order to track the sample throughout the treatments in the laboratory. The cleaning procedures applied to a sample depend on the sample material, so the first step is always to identify which part or fraction of the sample should actually be dated. Charcoals from archaeological contexts such as cooking pits often consist of many pieces from various trees. We offer species determination of wood to select short-lived species from such samples to get a better age constraint. After the selection

process, the sample is chemically cleaned in the laboratory. The procedures are based on published methods, but have been slightly adapted to the needs of the laboratory.

All methods have been tested with known-age materials in order to verify their performance. However, the variety of samples cannot always be covered with known-age materials, so that some samples are pre-treated multiple times. This is especially needed for conserved samples when it is unclear if the standard procedure will remove all of the contaminants. In such a case, the procedures may be slightly changed, e.g. more soxhlet repetitions or different solvents, to address possible issues with it.

### **Acid-Alkali-Acid Preparation**

Most sample types, such as charcoals, seeds, plant remains, food residues, wood, hair, feathers and textiles undergo a traditional acid-alkali-acid (AAA) cleaning (de Vries and Barendsen 1954). The samples are first put in 1 % HCl over night to remove carbonates. This also removes acid soluble organic compounds (“fulvic acid” fraction). After washing with deionized H<sub>2</sub>O till pH>4, a 1 % NaOH solution is added to dissolve alkali soluble organic components, the so-called humic acids. The solution is separated from the residue by centrifugation and decanting. The residue is washed with deionized water till pH<10, then acidified with 1 % HCl to remove CO<sub>2</sub> dissolved in NaOH, and washed again till pH>3. This gives the alkali residue or “humin” fraction. The NaOH solution is acidified with concentrated HCl till pH<1. This precipitates the alkali-soluble/acid-insoluble humic acid fraction that is then washed with deionized water till pH>3. Both fractions are then dried in an oven at 60 °C. Details of the process are shown in figure 2.

### **Base-Acid-Base-Acid-Bleach (BABAB) Preparation**

For dating of single tree rings, e.g. for wiggle matching, a cellulose extraction is performed following an adaptation of the BABAB (base-acid-base-acid-bleach) protocol (Nemec et al. 2010). First, the sample is treated with 4 % NaOH which is followed by short steps of 4 % HCl, 4 % NaOH, and 4 % HCl again at 75 °C. A bleaching step with a mixture of 5 % NaClO<sub>2</sub> and 4 % HCl at 75° C (pH≤4) is done, with an ultrasonic bath at room temperature at the end. The last NaClO<sub>2</sub> bleaching step is repeated until the sample material is white.

### **Bone Preparation**

Collagen is extracted from materials such as bones or antlers using a modified Longin method (Longin 1971). The samples are first crushed to small pieces to accelerate the later extraction process. The samples are cleaned in an ultrasonic bath with H<sub>2</sub>O and then acetone to remove fat. Following, the material is demineralized in 3.6 % HCl at room temperature until pH remains <1. The sample is then washed with deionized H<sub>2</sub>O till pH>3 and 0.5 % NaOH is added for 4 hours at room temperature to dissolve humic acids. After washing till pH<10, the residue is acidified again with 3.6 % HCl to remove atmospheric CO<sub>2</sub>, washed till pH=3 and hydrolyzed at 70 °C overnight. Finally, the gelatin is filtered hot through a pre-combusted quartz filter (Merck Millipore, AQFA04700) and freeze dried.

### **Calcined Bones and Carbonate Samples**

Calcined or burnt bones are crushed to powder before the cleaning begins. Organic material is removed by reaction with 1.5 % NaOCl for 48 h at room temperature. Soluble carbonates are afterwards removed with 5.7 % acetic acid and the sample is dried after washing with H<sub>2</sub>O (Lanting et al. 2001).

Carbonate samples undergo a soft leaching with 30 % H<sub>2</sub>O<sub>2</sub> to remove organic surface coating and the top carbonate layer of the sample.

### **Organic Solvent Extraction**

Samples that were conserved or are otherwise subject to significant hydrophobic pollutants are subjected to a soxhlet type procedure with an elution sequence of solvents (Bruhn et al. 2001), which can remove a large variety of contaminants. Yet it may not be successful in all cases. The samples are extracted (3x 10 minutes each) in sequence in boiling tetrahydrofuran (THF), chloroform, petroleum ether, acetone, and methanol. The apparatus we use for this is a 4-fold soxhlet type extractor “fexIKA 200” sold by LFH Laborfachhandel, Waldkirch, Germany. It is computer controlled and allows us to treat relatively small amounts of material.

## **COMBUSTION AND REDUCTION**

When a sample has been cleaned and the datable material has been isolated, it is combusted and reduced to produce homogenous graphitic material for the AMS measurement. Two different reduction lines are used in Trondheim.

### **Trondheim Oxidation and Reduction System (TOR)**

Most of the samples are reduced in the automated reduction system TOR (Trondheim Oxidation and Reduction-system). The system was originally built at the University of Erlangen (Ohneiser 2006). After the radiocarbon laboratory in Erlangen closed, the system was moved to Jena and finally installed in Trondheim. A new elemental analyzer (EA, Elementar microcube) suited for mg-sized samples was coupled to it for sample combustion. A LabVIEW program (Elliott et al. 2007) controls the hardware and the reduction process. The program was modified slightly to incorporate communication with the EA and our

sample database. The hardware was mostly unchanged although some parts have been replaced to prevent ageing or to fix issues probably originating in transport and installation. The system performs the iron-catalyzed hydrogen reduction of CO<sub>2</sub> (Vogel et al. 1984), freezing out the residual water with Peltier coolers similar to other automated reduction systems (Wacker et al. 2010). Ten reactors are arranged in circular shape and are run in parallel. A selector valve sends the gas coming from the EA to one of the reactors (Fig. 3). The reactors are isolated by two valves: the inlet connected to the selector valve and the outlet valve connected to the pumping system. A vertical freezing finger allows for freezing the CO<sub>2</sub> gas with liquid nitrogen. A cross-piece is connecting to a tube in the Peltier cooler, which is cooled down to about -30 °C, as well as the removable reduction tube where the reduction oven is mounted manually, and a pressure transducer (Mouser Electronics, 1240-015A-3L).

Once the system has been readied by manually replacing the reduction tubes (Kimble, 6x50 mm, borosilicate glass) pre-filled with 3 mg Fe powder (VWR, 24088.232, ≥99.5 % powder, reduced by hydrogen), mounting the ovens, and placing the samples in the EA wheel, the software controls the automated processes of iron cleaning, sample combustion, and reduction. A manual mode can be used to accommodate special procedures. The iron catalyst (of all the reactors) is first cleaned by heating it to about 350 °C in hydrogen gas for 40 minutes after which the system is pumped and filled with Ar gas. Each sample is combusted in the EA, the sample CO<sub>2</sub> is channeled to the desired reactor by the selector valve and captured in reactors by freezing. Liquid nitrogen is filled in a Dewar and moved to the freezing finger of the reactor being prepared for reduction to freeze CO<sub>2</sub> during sample loading and the addition of hydrogen. Once the CO<sub>2</sub> peak from the EA has passed, the inlet is closed and the remaining He carrier gas is pumped. The loading procedure takes about 10

minutes per reactor. The CO<sub>2</sub> pressure in the reactor is used to determine the sample size and adjust the amount of hydrogen that is needed to obtain a ≈10 % hydrogen excess, while the CO<sub>2</sub> is frozen again. We aim for 1.0-1.5 mgC, but smaller samples can be reduced as well. Once the hydrogen has been added, the Peltier coolers and, with some delay, the ovens (550 °C) are activated for the reduction process. The program logs the pressure data of the reactors and checks the end pressure and rate of change in order to suggest the end of the reaction. A typical reduction takes about 4 hours, while larger samples might need more time. This allows us to prepare two batches per day.

### **Fe-Zn Manual Reduction System**

The other reduction system is a manually operated, in-house built system which uses a Zn-Fe reduction process (Jull et al. 1986; Slota et al. 1987). The design of the system is based on the reduction system at Scottish Universities Environmental Research Centre (SUERC) (P. Naysmith, G. Cook, personal communication, March 4, 2013).

As this system does not include means to produce CO<sub>2</sub>, sample CO<sub>2</sub> is prepared on separate systems. Challenging samples, such as liquids or sediments requiring a large volume of material, are sealed in quartz tubes with 500 mg CuO pellets (VWR, Copper(II)oxide wire, 11005CK) and a piece (ca. 100 mg) of Ag foil (Alfa Aesar, 00501 silver foil) for combustion in a muffle oven running at 850 °C for 6 hours. Carbonate samples are hydrolyzed in borosilicate glass H-shaped tubes made of two Louwers glass valves (12 mm diameter). The samples are placed in one of the two interconnected tubes while 85% phosphoric acid is placed in the other. Both combustion quartz tubes and the carbonate H-tubes are evacuated in a 19-port glass manifold.

Sample CO<sub>2</sub> from both types of reactions is transferred to glass bottles using a 6-fold dedicated system for multi-step cryogenic gas cleaning to remove water and non-condensable gases. This system allows for the partitioning of the gas as well as its distribution to multiple gas bottles for transfer to the reduction system.

Each reduction cell is made of two 125 mm long tubes (6 mm diameter, borosilicate glass, manufactured by the university glass blower), which are connected to adjacent ports of a union cross (Swagelok, SS-4-UT-4) for both the Fe and Zn reagents. This construction ensures a good separation of both Fe and Zn reagents which is necessary for a complete reduction of the CO<sub>2</sub> without fractionation. As for the automated system, we use 3 mg Fe (VWR, 24088.232, ≥99.5 %) in addition to 70 mg Zn (Merck, <45 μm, 1.08789.0500). Seven of these reduction reactors are connected to a glass pumping manifold by O-ring sealed glass valves.

Initially the Fe and Zn reagents are cleaned by heating them to about 380 °C and 260 °C respectively while pumping. Gas transfer bottles are also connected to the reactors and the sample CO<sub>2</sub> is transferred to the reactors by freezing with liquid nitrogen freezing. Two large ovens are moved to cover the reduction tubes during the reaction. The temperatures are set to 560 °C (Fe) and 430 °C (Zn) and the oven for tubes containing the Fe is applied one hour after the Zn oven. The temperature in the ovens is not perfectly homogenous and shows a decrease of about 10 % towards the outer reactors.

### **Target Pressing**

Sample graphite is pressed into the target pieces which have been laser engraved with a unique number (holder number) to identify the sample afterward. The target piece is fixed in a screwing tool with its front facing a ceramic ball (diameter: 5 mm, ZrO<sub>2</sub>/YPSZ) blocking one side of the graphite cavity (diameter: 1 mm). On the other side, a funnel is screwed into the

target piece which facilitates the sample transfer into the cavity. Once the graphite has been transferred into the target piece, the graphite is pressed through the funnel for about 3 s at a pressure of 5-6 bar. A copper pin is added in back to seal the target and the target is pressed again deforming the copper pin. Targets are stored in Eppendorf vials until they are measured.

## **MEASUREMENT**

The  $^{14}\text{C}$  measurements are done at our 1 MV AMS system made by High Voltage Engineering Europa B.V. A detailed description of the system can be found in several publications (Klein et al. 2006; Klein et al. 2007; Nadeau et al. 2015) and only a short overview is given here. The main elements before the accelerator are the SO-110 Cs sputter source and a bounced  $90^\circ$  magnet. We usually operate the Cs ion source at about  $40 \mu\text{A } ^{12}\text{C}$  current. The source has a 50-position wheel. The terminal voltage is usually set to 1 MV for routine measurements, which provides an ion transmission of 49 % in the 2+ charge state. Another  $90^\circ$  magnet is used for mass separation after the accelerator. The stable isotopes are injected into off-axis Faraday cups for current measurement, which is used for normalization of the  $^{14}\text{C}$  signal. The  $^{14}\text{C}$  ions are injected into a  $120^\circ$  electrostatic analyzer, which provides an energy filter and redirects the ion beam into the gas ionization chamber where the single ions are identified by a two-anode energy spectrum.

A usual sample wheel consists of 10 oxalic acids 2 (NIST SRM-4990C) reference samples (Mann 1983), 5 secondary standards with different nominal values and 5 process and machine blanks. The remaining 30 positions of the wheel are filled with unknown samples. After an initial burn-in time of 5 minutes for every sample, all the samples are sequentially measured for 8 minutes. The sequence is repeated to achieve a measurement time of

80-90 minutes for each sample so that a pace of 2 wheels per week can be sustained. The recorded data is saved in separate measurement files for each measurement. The whole dataset is combined in an Excel spreadsheet, which is used for the evaluation. The data evaluation follows the general principles of radiocarbon dating (Mook and van der Plicht 1999). The first step in the evaluation is determining of the normalization ratios for  $^{13}\text{C}$  and  $^{14}\text{C}$  by using the reference samples. A current or time dependent correction can be applied at this stage if needed. A background correction to the initial data is not done because the machine background, determined with unprocessed natural graphite (Alfa Aesar, graphite powder, natural, briquetting grade, -200 mesh, 99.9995 % (metals basis)), is measured at a level corresponding to a radiocarbon age of >60 kA, which is significantly lower than the process blanks. The individual measurements of each sample are then normalized and fractionation corrected to calculate the  $F^{14}\text{C}$  value. The mean result is then corrected for process blank depending on mass and material of the sample for the final result. The correction is scaled with the  $^{14}\text{C}$  content of the sample assuming the contaminating material is modern (Donahue et al. 1990). The size dependence of the blank is determined by a set of measurements with different sample size. For the AAA treatment, a piece of coal from Svalbard is used as test material. The blank material undergoes the same cleaning, combustion, and reduction steps as a regular sample and is regularly repeated. The  $^{14}\text{C}$  blank results show an inverse proportionality with the combustion mass (Fig. 4) which is interpolated for the blank correction (Schleicher et al. 1998; Ruff et al. 2010).

The secondary standards are chosen to be similar to the unknown samples in size, material, and age. Most of the samples measured in our laboratory are charcoals, wood, and bones from archaeological sites with ages of about 2'000 years. A well suited sample for these conditions is sample H from the FIRI intercomparison (Scott 2003). Since the laboratory was

still running the GPC when the FIRI intercomparison was conducted, the amount of material supplied is enough for many AMS measurements. In 2017 and up to March 2018, the FIRI H sample has been measured a total of 90 times. The mean value of all measurements  $F^{14}C=0.75755\pm 0.00017$  (radiocarbon age:  $2230.5\pm 1.8$  y BP) is in good agreement with the consensus value of  $2232\pm 5$  y BP. The FIRI D sample was also measured several times during this period ( $n=19$ ) and its mean value  $F^{14}C=0.57088\pm 0.00029$  (radiocarbon age:  $4503\pm 4$  y BP) is statistically consistent with the consensus value of  $4508\pm 3$  y BP.

## **IRMS**

In addition to the AMS system, the laboratory operates an isotope ratio mass spectrometer (IRMS) for stable isotope measurements, mainly  $\delta^{13}C$  and  $\delta^{15}N$ . The Thermo Delta V Advantage is equipped with a multiport dual inlet system for gas from bottles and is also connected to an EA (Thermo Flash 2000) by Thermo Conflo IV for the measurement of solid samples. The system can run automatically, processing a total of 32 samples which usually contain about 20 unknowns. The sample size can easily be below 0.5 mg, but might need to be increased to get an accurate result from samples with a low carbon or nitrogen content.

## **CONCLUSION**

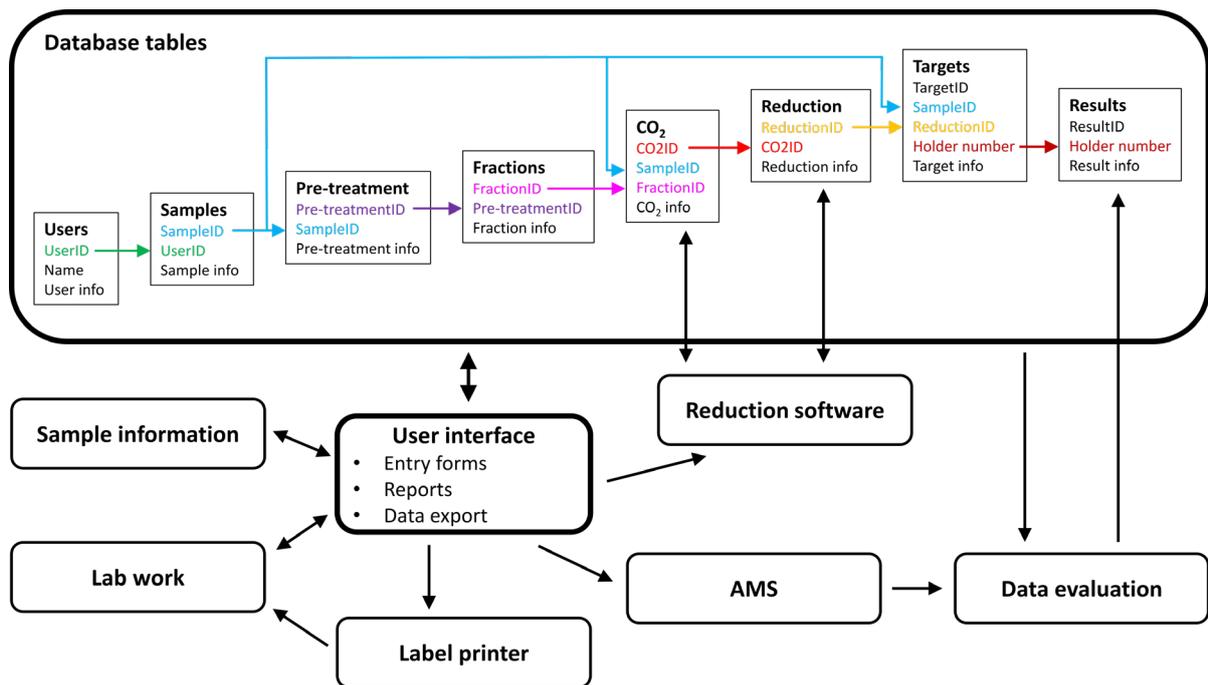
In the past years, the National Laboratory for Age Determination in Trondheim completed the transition from traditional decay counting to AMS measurements. This included testing adapted procedures for handling, cleaning, and measuring for mg size samples. New systems allow a higher sample throughput with a limited need of manpower. The infrastructure and systems for handling sample information and results were created for an efficient and well documented work flow. The addition of an IRMS is also extending the capabilities of the lab, both for checking  $^{14}C$  related procedures and for independent research.

## **ACKNOWLEDGMENTS**

We thank Gordon Cook and Philip Naysmith from SUERC for the possibility to visit their laboratory which played a key role in developing our Zn reduction system. We also want to thank Axel Steinhof and Martin Göbel from MPI Jena for their support in transporting and commissioning the reduction system from Erlangen.

## Figures

Figure 1 Schematics of the database main tables and links. Minor tables, e.g. the operator table, which identifies the individuals working on samples in every step of the procedure, or the system table, which contains the reduction reactors, were omitted for better visibility.



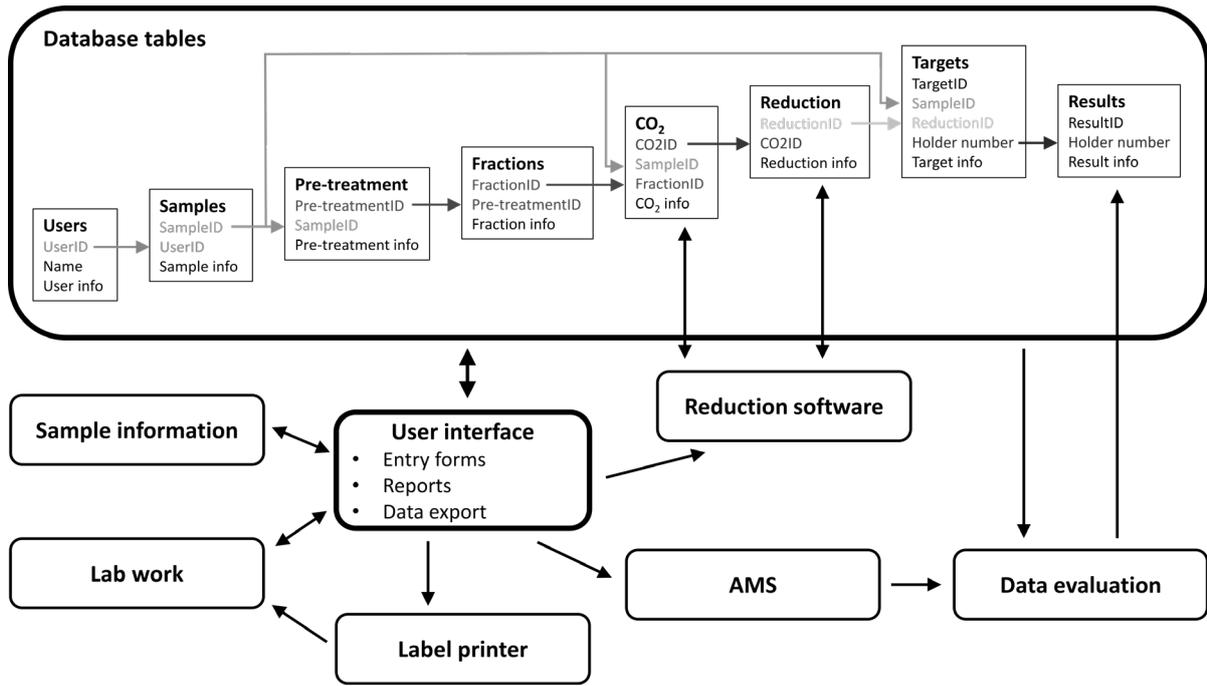


Figure 2 Process flow of sample cleaning processes for different sample materials. The given amount of material for sample types might vary, e.g. depending on how degraded a sample is.

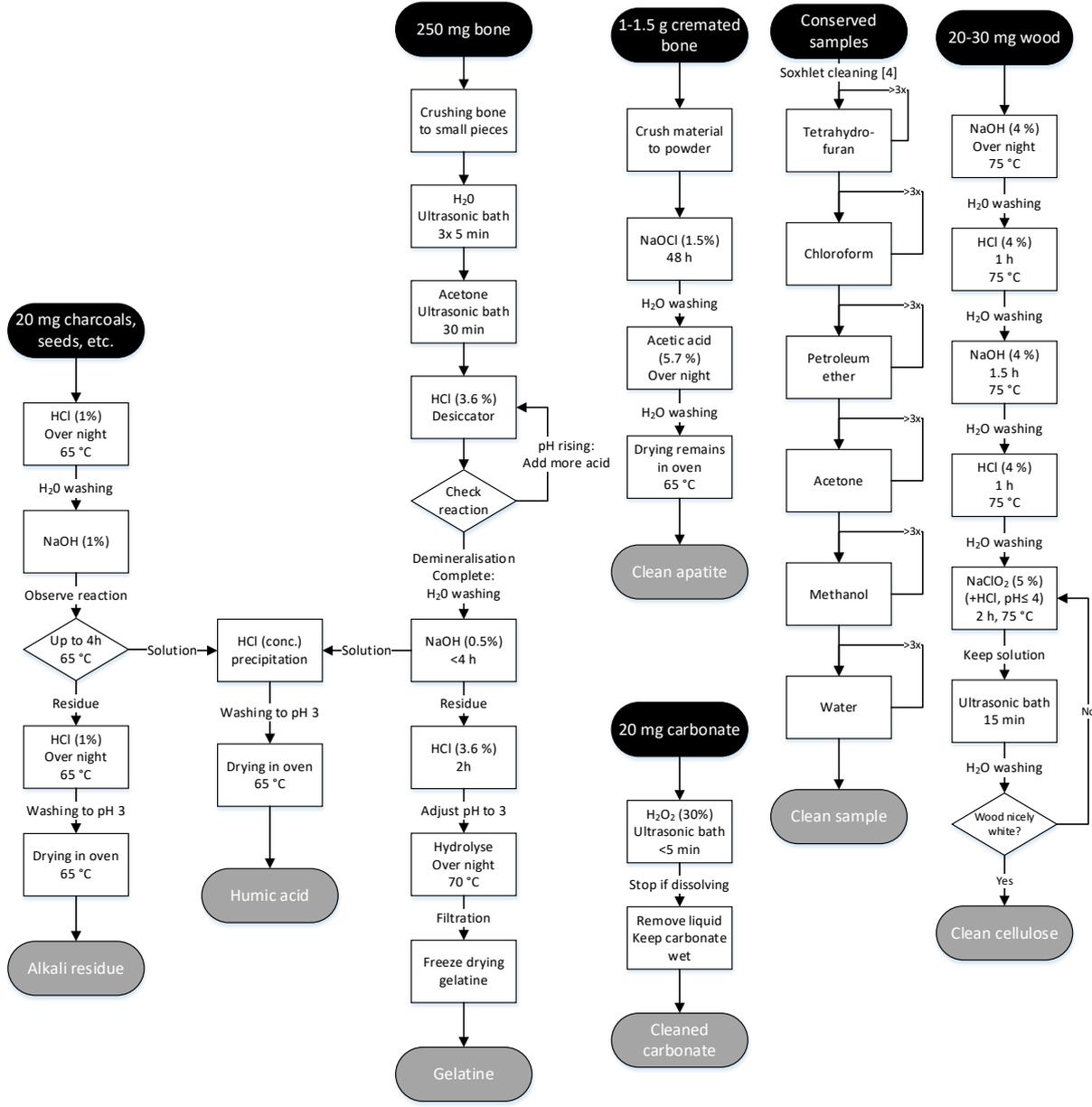


Figure 3 Schematic of the TOR system.

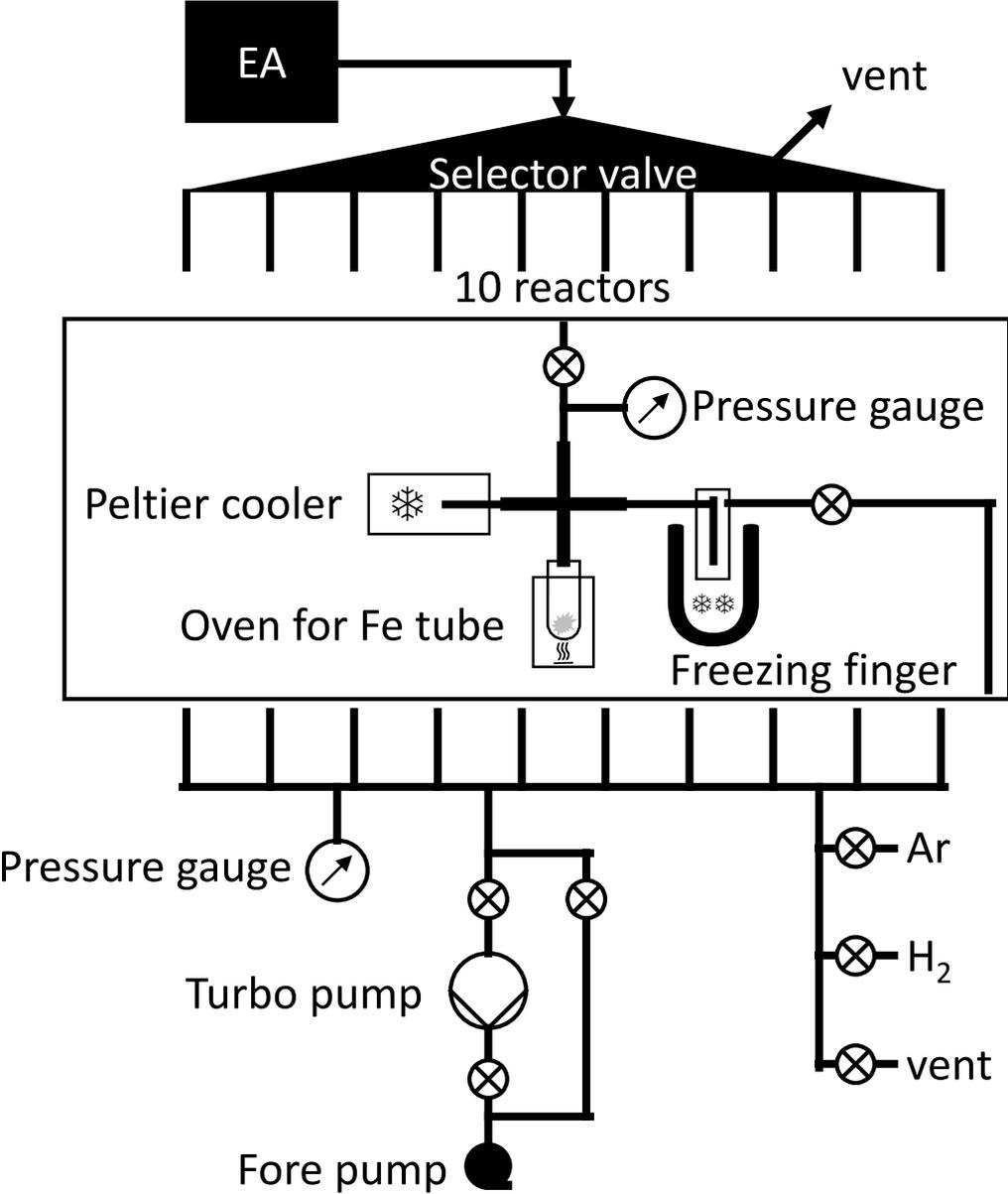
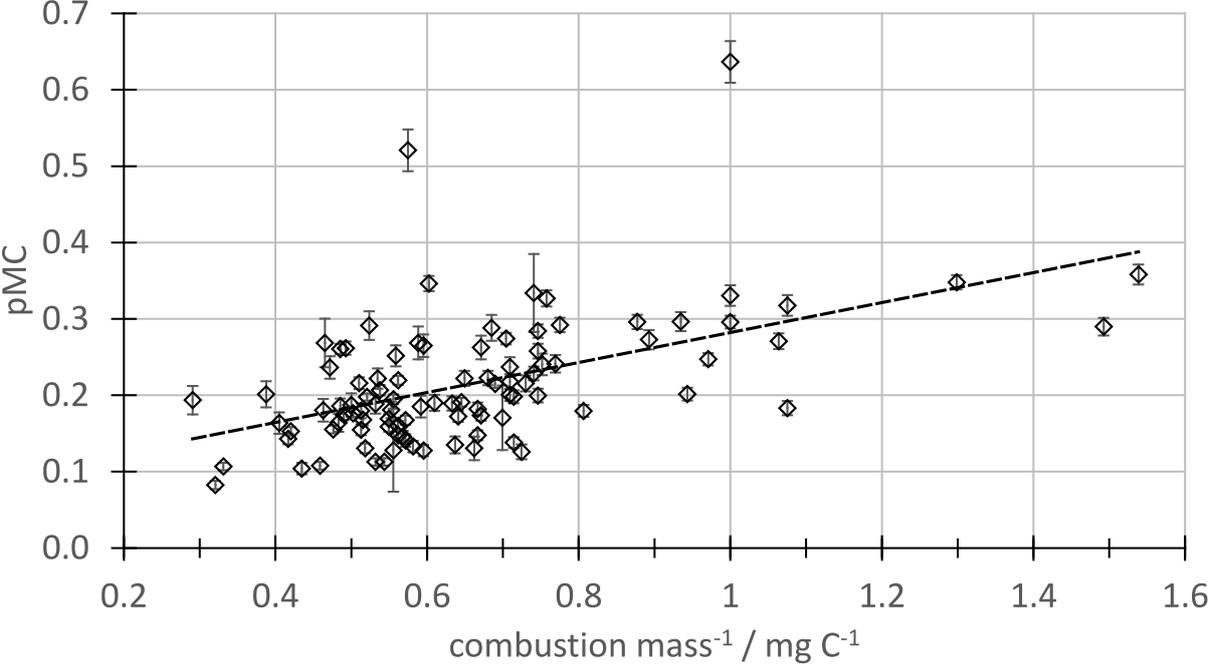


Figure 4 Size dependence of the process blank for AAA treatment with reduction at the automated reduction system.



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