Available online at www.sciencedirect.com
ScienceDirect
Nuclear Instruments and Methods in Physics Research B 259 (2007) 303–307
NIM B
Beam Interactions with Materials & Atoms
www.elsevier.com/locate/nimb

Development of an automatic gas handling system for microscale AMS ^{14}C measurements

Thomas Uhl *, W. Luppold, A. Rottenbach, A. Scharf, K. Kritzler, W. Kretschmer

Physikalisches Institut, Universität Erlangen-Nürnberg, 91058 Erlangen, Germany

Available online 30 January 2007

Abstract

A gas handling technique has been developed for metering CO_2 into the gas ion source of the Erlangen AMS facility. Further investigations and developments have resulted in a system that enables the handling of gaseous samples with carbon masses from 1 μg to 1 mg. The system has been optimized for coupling an elemental analyzer to a gas ion source. Moreover, the system can be connected to any other system that produces CO_2, e.g. gas ampoules, gas chromatographs, etc. The principle of the gas handling system is to first store and isolate CO_2 cryogenically. Then the CO_2 is fed into the ion source by the pressure difference between the CO_2 reservoir and the gas ion source which are connected by a glass capillary. By regulation of the CO_2 pressure, the CO_2 flow into the ion source can be controlled. The outstanding advantage of this easy technique is the possibility of its complete automation. For use of the system with an elemental analyzer we created application software that controls the combustion of the sample, the cryogenic storage and the exact metering of the CO_2 into the ion source. Besides the application of using CO_2 for radiocarbon dating, our gas handling system opens new possibilities of research in fields where only small samples are available or large series of samples have to be measured quickly, e.g. environmental or biomedical research. In addition this system has turned out to be a perfect tool for detailed investigations of gas ion sources.

PACS: 01.30.Cc; 07.77.Ka; 06.60.Ei

Keywords: AMS; Radiocarbon dating; Gas ion source; Gas handling system; V8

1. Introduction

Our standard method to make solid targets for sputtering is to combust carbonaceous samples with an elemental analyzer (EA), to collect the CO_2 cryogenically, to graphitize and then press them into cathode holders [1]. As the effects of contamination of samples rises rapidly with smaller masses, a carbon mass of at least 100 μg is necessary. In addition, the step of graphitization is time consuming and labor intensive. Motivated by research in sectors of environmental science where only small samples are available (e.g. ~10 μg after a preparation time of a year [2]) and intended biomedical applications [3] where many samples have to be measured, the ion source was modified into a hybrid ion source [4] that accepts solid samples as well as gaseous samples. With the possibility of direct CO_2 use, graphitization to form solid sputter targets would no longer be necessary. Because of the higher efficiency of gas ion sources and reduced contamination with carbon due to the minimized preparation line, it is possible to measure samples with carbon masses down to one microgram [4,5]. To be able to use gaseous samples over a very wide mass range we are utilizing two techniques. Since both of the techniques are very simple, full automation was possible.

2. Techniques for microscale ^{14}C AMS measurements

2.1. Injecting CO_2 into a gas ion source with a syringe

As shown in [4] the CO_2-flow into a gas ion source has to be kept very constant at ~2 μl/min. To keep a constant

* Corresponding author. Tel.: +49 9131 8527119; fax: +49 9131 15249.
E-mail address: thomas.uhl@physik.uni-erlangen.de (T. Uhl).

0168-583X/$ - see front matter © 2007 Elsevier B.V. All rights reserved.
doi:10.1016/j.nimb.2007.01.173

gas flow an adjustable CO_2-reservoir that is connected to the gas ion source via a glass capillary is used. The CO_2 flow is then a function of the pressure difference between the reservoir and the ion source as well as the inner diameter and length of the capillary (T = const.). With a variable volume for the reservoir the pressure can be adjusted so that the CO_2-flow is kept at the desired rate. To prevent carbon contamination from the environment, the pressure in the reservoir should be kept above atmospheric pressure, and to be able to measure very low carbon amounts the dead volume has to be very small. For this reason we use a gas-tight syringe (piston material is PTFE) for the CO_2 reservoir. By using connectors with very low dead volumes, a syringe manufactured by our lab and a very small pressure sensor (1.6 mm diameter), we were able to decrease the total dead volume to ~15 µl. This is 1000 times lower than that of the bellows in the gas handling system from NEC.

The advantages of this technique are:

- It is an easy technique for metering CO_2 into the ion source;
- It can be completely automated;
- It has a very low dead volume of 15 µl.

For optimum operations we have to maintain a reservoir pressure of 40 psi (see Fig. 1 for capillary dimensions). But, because of the remaining internal volumes inside the valves, the pressure sensor and the connectors, this pressure cannot be maintained for carbon masses below 30 µg. In this low mass range the CO_2-pressure can not be maintained at optimal conditions for the ion source; there is a need for an additional technique for samples in the mass range of 1–30 µg. Through the use of capillaries with different dimensions it would be possible to decrease the needed pressure to 20 psi. Under these circumstances, the additional technique would only be necessary for samples from 1–15 µg. However, because a change in the capillary dimensions would require a change in the dimensions of the syringe (the volume must be increased if the pressure is dropped for the same maximum CO_2 amount) and the manufacturing of a real gas-tight syringe is rather difficult, the dimensions of capillary have not been changed. Optimization of such components would be considered during the future construction of a newer model apparatus.

2.2. 'Cryogenic storage and release' technique for the handling of CO_2-samples (1–30 µg)

To analyze carbon masses from 1 to 30 µg we use a revised 'cryogenic storage and release'-technique that was presented in [4]. The principle is to store the CO_2 in a 'freezing tube' (0.75 mm ID) by dipping the tube into a Dewar vessel filled with liquid nitrogen (LN_2). The CO_2 is distributed homogeneously over the whole inner surface of a stainless steel tube. A low helium flow of 0.1 ml/min

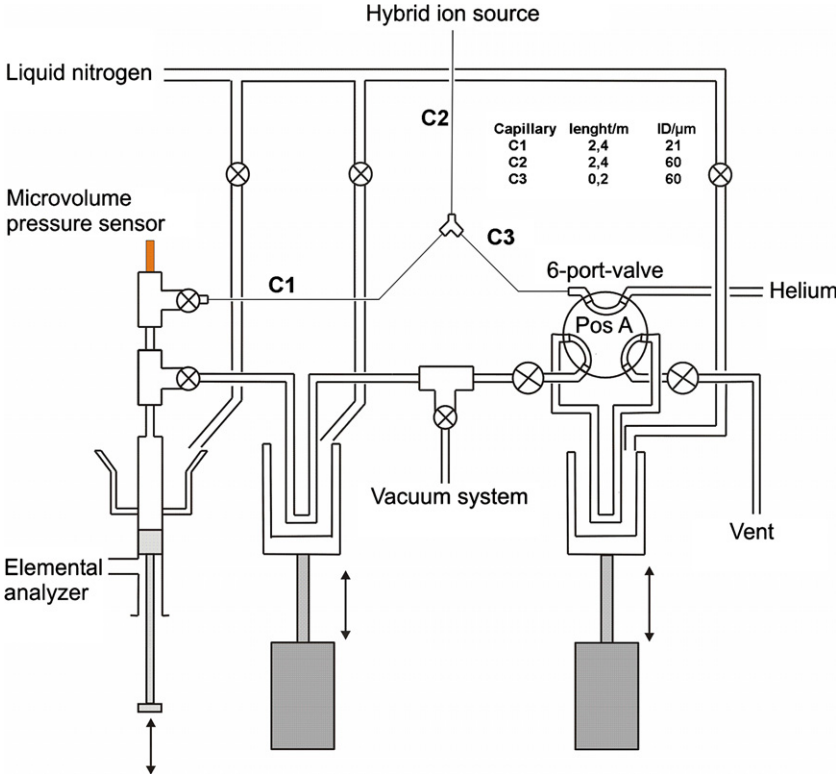

Fig. 1. Schematic of the gas handling system coupled with an EA. Two modes of operation are possible: (a) CO_2 is stored in syringe and introduced into ion source by pressure difference (b) CO_2 is stored cryogenically in U-Trap and released into Helium flow (Multiport valve must be in Pos B), that transports the CO_2 into the ion source, by slowly lowering the dewar vessel.