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Development of a commercial Automated Laser Gas Interface (ALGI) for AMS

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ABSTRACT

National Electrostatics Corporation (NEC), Massachusetts Institute of Technology (MIT), and GlaxoSmithKline (GSK) collectively have been developing an interface to introduce CO_2 produced by the laser combustion of liquid chromatograph eluate deposited on a CuO substrate directly into the ion source of an AMS system, thereby bypassing the customary graphitization process. The Automated Laser Gas Interface (ALGI) converts dried liquid samples to CO_2 gas quickly and efficiently, allowing 96 samples to be measured in as little as 16 h. ^{14}C:^{12}C ratios stabilize typically within 2 min of analysis time per sample. Presented is the recent progress of NEC's ALGI, a stand-alone accessory to an NEC gas-enabled multi-cathode source of negative ions by Cs sputtering (MC-SNICS) ion source.

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1. Introduction

Graphitization has long been a time-consuming and yet necessary procedure for carbon AMS. While the technology behind CO_2-based ion sources has certainly advanced in the past decades [1], these sources are not yet comparable to solid graphite ion sources in terms of efficiency, ion currents, and susceptibility to sample contamination. Based on the patented work [2] done by MIT's Department of Biological Engineering, National Electrostatics Corporation (NEC), Massachusetts Institute of Technology (MIT), and GlaxoSmithKline (GSK) have been working together to design and build a prototype of an interface capable of producing CO_2 by the laser combustion of dried liquid samples, such as the output of a liquid chromatograph. While laser ablation-coupled AMS systems have been in use for several years [3], this is the first system in development for commercial carbon AMS analysis capable of analyzing a large number of samples automatically using a 96 sample well plate. Presented is the recent progress of NEC's Automated Laser Gas Interface (ALGI), a stand-alone accessory to an NEC gas-enabled multi-cathode source of negative ions by Cs sputtering (MC-SNICS) ion source.

2. Sample preparation and methods

A 96 sample plate consists of a 12 column by 8 row grid and is similar to plates often used in biomedical applications. Loaded into the titanium plate are stainless steel mesh wells, each containing a bed of 20 mg of CuO powder, which serves as an oxidizing reactor to convert sample carbon to CO_2. After the wells are packed with CuO, the plate is heated in an oven at ~750 °C for several hours with a continuous flow of O_2 through the oven to oxidize any organic material contaminating the CuO and maximize the amount of adsorbed O_2 [4]. The baked plates are then stored in argon to avoid atmospheric contamination. Fig. 1 shows the sample plate resting in the ALGI chamber with the lid and laser removed.

To simulate the liquid samples obtained from pharmacokinetic or metabolism studies, individual liquid samples are combined with aqueous ^{12}C sodium benzoate (SB) with a carbon content of ~300 μg. This compound was chosen due to its solubility in water, non volatile nature, and commonality as a ^{12}C carrier in the pharmaceutical industry. Additionally, various amounts of aqueous ^{14}C enriched SB can be added to wells for increased modern carbon during ALGI testing. Once the samples are added to the CuO wells by a pipette, usually 5–10 μL of total liquid, plates are heated briefly (to dry the liquid samples and remove volatile components) and inserted into the ALGI chamber through a side door (not visible in Fig. 1).

For experimentation, four types of samples were used in plates prepared by both NEC and MIT: Australian National University (ANU) sucrose wells, CuO wells, Dead SB wells, and Hot SB wells:

(1) An ANU sucrose aqueous solution (IAEA-C6) containing ~300 μg of carbon per well was used as the normalization standard.

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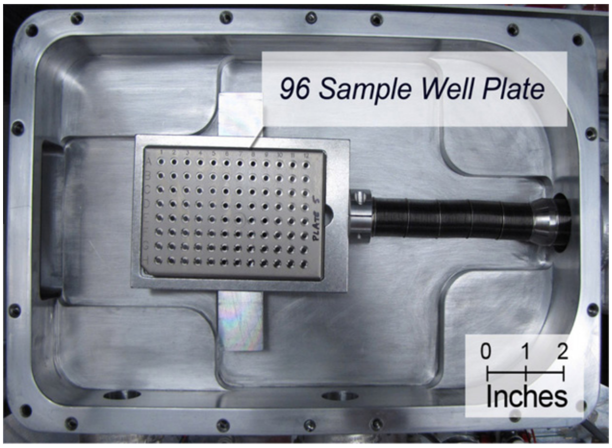

Fig. 1. The interior of the ALGI sample chamber with the lid and laser removed. The 96 sample Ti plates are inserted and removed through a door on the left side of the chamber (not shown).

(2) CuO wells that contained neither ^{12}C SB nor ^{14}C SB dried samples. These wells were used for system background measurements.
(3) Dead SB wells contained ~300 μg of ^{12}C SB and were used as processed background.
(4) Hot SB wells also contained ^{12}C SB along with various amounts of enriched ^{14}C SB, ranging from 0.001 disintegrations per minute per gram (dpm/g) to 0.2 dpm/g.

3. Design and control system

Fig. 2 exhibits the entire ALGI including the touch screen for local system control. The plate is positioned in the ALGI by two stepper motors, which control the rotational and translational axes. A series of 12 solenoid valves control the vacuum and gas-related aspects of the system, while two mass flow controllers inject helium, modern CO_2, and background CO_2 into the ion source.

In order to aid the user in performing common tasks, the touch screen contains a series of automated Control Modes. The system begins in Park Mode, with the chamber door and each of the valves closed. ALGI also sets both mass flow devices to zero flow, to avoid pressurizing gas lines unnecessarily. When a sample plate is ready to be tested, the user activates Plate Load Mode, at which time ALGI vents the chamber to atmosphere, moves the plate holder into its load position, and opens the chamber door automatically. Once this operation is complete and the plate is loaded, the user selects Run Mode, which prepares the ALGI for normal measurements. The chamber door is closed and the plate is re-initialized to the home position to confirm that each plate starts in the same location. The appropriate solenoid valves are activated to evacuate the chamber to <5 torr and fill it to 40 torr of helium; three cycles are used to purge atmospheric gases. Helium is also routed through the mass flow controller at 0.05 standard cubic centimeters per minute (sccm), and is used as a carrier gas for the combustion products. Fig. 3 shows a schematic of the ALGI touch screen, including gas line routing for Run Mode and associated valves.

When an individual well is selected to be combusted, stepper motors locate the coordinates of the well and an o-ring sample shield closes around the well to isolate the sample from the rest of the plate. The helium carrier gas flow rate is adjusted to roughly equal the chamber pressure, thereby minimizing the effect that sample changing (i.e. sample shield movement) has on the source vacuum. Laser combustion of the sample produces gas products, including CO_2, which are then transported by the carrier gas through a Nafion water separator to a slotted Ti cathode in the ion source [5,6]. NEC's proprietary DMAN software (located on NEC's AccelNET control systems, not on the ALGI) controls the entire data collection process and computes ^{14}C:^{12}C and ^{13}C:^{12}C isotope ratios, as well as other counting statistics.

By attaching the ALGI to an NEC 134 sample MC-SNICS, sample cross-contamination is reduced significantly by using a one-cathode-per-sample configuration. Before each sample is measured, DMAN software indexes to an unused Ti cathode and calculates the average ^{14}C event rate to ensure cathodes are not contaminated by residual carbon in the ion source. This is known as the "pre-laser cathode monitoring process." Once the event rate passes below a user-defined threshold, the laser is fired, combusting the dried sample in the well; CO_2 and other combustion products are released into the source. In these studies, the user-defined threshold was typically fewer than 15 ^{14}C events per minute for a new cathode, or less than 0.0025 dpm per well. The criterion for the threshold can be modified for individual experiments, ^{14}C content, and precision required.

After the laser is fired, there is approximately an 8–10 s delay before the gas is introduced into the ion source due to the carrier gas filling the gas lines. The ^{14}C events and high energy $^{12}C^+$ current rise sharply in the next minute followed by an exponential

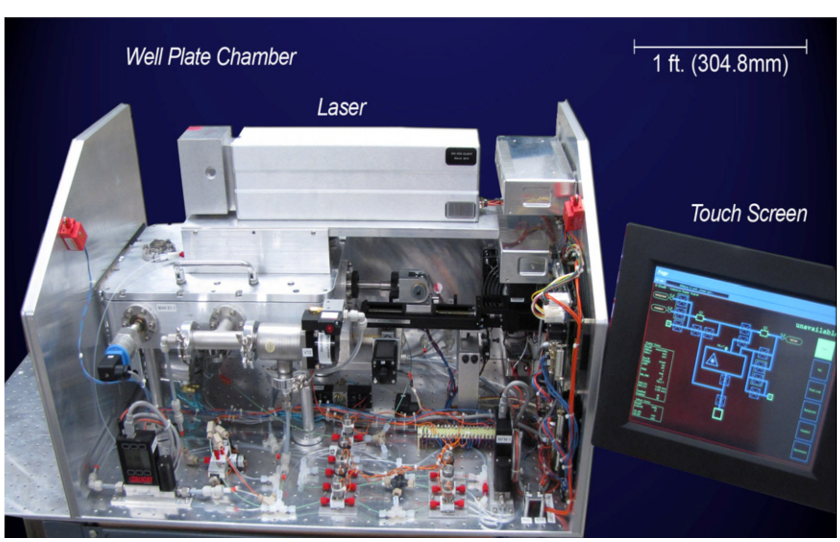

Fig. 2. NEC's Automated Laser Gas Interface (ALGI) with touch screen monitor. The laser is positioned above the plate chamber using a 90° mirror.

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