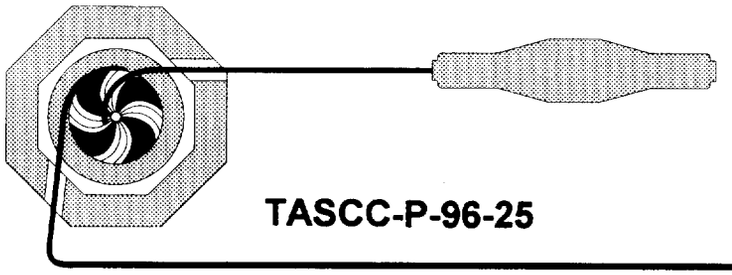

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HOLD-UP AND MEMORY EFFECT FOR CARBON IN A COMPACT MICROWAVE ION SOURCE

R.J. Schneider and K.F. von Reden
Woods Hole Oceanographic Institution, Woods Hole, MA 02543, U.S.A.

J.S.C. Wills[1], W.T. Diamond R. Lewis, G. Savard and H. Schmeing
AECL, Chalk River Laboratories, Chalk River, ON K0J 1J0, Canada

[1]Author to whom correspondence is to be addressed. E-mail address: willsj@crl1.crl.aecl.ca

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R.J. Schneider and K.F. von Reden
Woods Hole Oceanographic Institution, Woods Hole, MA 02543, U.S.A.
and
J.S.C. Wills[1], W.T. Diamond, R. Lewis, G. Savard, and H. Schmeing
Chalk River Laboratories, AECL, Chalk River, Ontario, Canada, K0J 1J0

Abstract

The production of C^+ in a microwave ion source injected with a gaseous CO_2 sample has been investigated as an alternative to sample graphitization. A continuous flow of argon gas maintained the discharge, and 20 μL of CO_2 gas at approximately 1 atmosphere was pulsed into the source through a sample valve closely coupled to the plasma chamber. The C^+ component of the beam fell to 1% of its original intensity less than 10 s after the valve was operated. In a separate experiment, the microwave ion source was operated with pure CO_2 feed gas and the extracted beam was magnetically analyzed. Efficient breakup of the molecules was observed. These results are considered promising for such applications as accelerator mass spectrometry of C isotopes in gaseous samples.

[1] Author to whom correspondence is to be addressed. E-mail address: willsj@crl1.crl.aecl.ca

Introduction

The standard method for producing negative carbon ions for AMS analysis is to reduce carbon dioxide over an iron or cobalt catalyst in a reactor. The resulting mixture of graphite and iron is pressed mechanically into a target for a cesium sputter ion source. This method is efficient and target production has been automated reasonably well for carbon sample sizes from 100 μg to 2 mg. Using manual handling techniques it is possible to produce reliable graphite sputter targets from samples as small as 20 μg. For routine analysis of trace gases in environmental samples or for biomedical samples, it would be convenient to eliminate the graphitization process and to be able to use even smaller samples. We have investigated the possibility of injecting gaseous samples directly into the plasma of a microwave ion source. If it can be shown that this source would be stable, reliable, and have small memory effects, then it could be a good candidate for an AMS ion source (when coupled properly through a charge-exchange canal). In this paper, we present the results of preliminary tests for sample memory and hold-up for μmol samples of carbon dioxide (containing 12 μg of carbon), introduced as pulses into a novel microwave ion source. This is an alternative method to the hybrid ion source developed at Oxford [1], in which the CO_2 sample gas is introduced into a titanium target cone of a conventional sputter ion source.

Ion Source

The ion source used for these tests is a compact permanent magnet microwave-driven ion source [2] being developed to provide intense heavy-ion beams to the Tandem Accelerator Superconducting Cyclotron (TASCC) facility [3]. The discharge is supported by a gas feed, so gaseous samples can be accommodated by injection into the support gas stream. The operating principle of the source is described in references [4] and [5]. A sectional view of the source is presented in Fig. 1. A brief description follows.

A tapered waveguide directs 2.45 GHz microwaves into the plasma chamber through a ceramic window. The water-cooled copper plasma chamber has an inner diameter of 5 cm and a length of 7 cm. The support gas enters the chamber through a tube adjacent to the microwave window. The requisite solenoidal magnetic field, having a peak axial value of about 900 G, is provided by a permanent magnet assembly. Positive ions are extracted from the microwave-driven discharge through a 5 mm diameter aperture in a copper plasma electrode. The extraction system is a triode structure with an acceleration gap variable from 2.5 to 10 mm and a deceleration gap of 2 mm. For injection into a Tandem accelerator, the positive-ion beam is converted into a negative-ion beam by charge exchange in an alkali vapour.

The source has good gas efficiency; for example, a 93 mA positive hydrogen ion beam was generated using 3 sccm (5 μg/second) hydrogen, giving an efficiency of 22%. The

source has also demonstrated reasonable breakup of molecular species. For example, atomic fractions of up to 80% have been obtained with hydrogen and oxygen feeds. No measurable X-ray fields have been observed in the vicinity of the ion source at up to 40 kV extraction voltage.

For the hold-up time measurements, the source was installed on the injector deck of the TASCC Tandem. The layout of the injector is shown in Fig. 2. The positive ion beam was extracted from the source at up to 30 kV. The beam was mass-separated by a dipole magnet. The mass-separated ion beam current was measured by a suppressed Faraday cup, 3.9 m from the ion source. For this experiment, the charge-exchange canal heaters were not turned on. Only the positive-ion beam was analyzed and all focusing elements in the beamline were turned off. As a result, the analyzed beam current measured at the Faraday cup was approximately a factor of 100 smaller than the extracted beam current deduced from the high voltage power supply current.

For studies of molecular breakup, the source was installed on a test stand. The extracted ion beam was analyzed with a two-slit emittance measuring device [6] with a dipole magnet for mass analysis.

Hold-up Time

A 6-port valve with interchangeable sample loops [7], designed for gas chromatography applications, was used to inject CO_2 samples into the source gas stream. The valve was located within 20 cm of the plasma chamber. For these tests, a 20 μL sample loop (the smallest available) was used. The sample loop was filled at atmospheric pressure through a low-pressure regulator. The sample loop was then switched into the source gas stream. To maintain the discharge during sample switching, a bypass line fed a constant stream of source gas around the sample valve (Fig. 3). The source discharge was started with 0.6 sccm ^{40}Ar through the bypass line and 0.4 sccm through the valve. A 6 mA 17 kV Ar^+ beam was extracted. The C^+ and Ar^+ components of the mass-analyzed beam were each measured on the Faraday cup while the sample valve was cycled. Results are shown in Fig. 4. When the sample loop was switched into the source gas line (after allowing sufficient time for the loop to fill with CO_2), the inrush of gas from the sample loop at near-atmospheric pressure briefly extinguished the discharge, after an initial transient. The carbon signal (Fig. 4a) then recovered to a peak value of approximately 7 μA before decaying to near the base-line value. The $^{40}Ar^+$ signal (Fig. 4b) stabilized at a higher current (possibly due to lower Ar gas flow into the source) while the sample loop was switched into the gas stream. When the loop was switched out and the Ar flow had stabilized, the Ar^+ current returned to its original value.

Memory Effects

To test for memory effects (source contamination by carbon deposition) the source was operated continuously on pure methane (CH_4) with a total extracted beam current of 5.5 mA for 1.5 hours. The source gas was then changed to Ar and the decay of the C^+ and O^+ peaks was observed. After 5 min (the time taken to switch feed gases), the C^+ peak had decreased from 10 μA to 50 nA, and after 30 min of operation, it had reached 25 nA.

The same test was performed with CO_2. The source was operated on CO_2 for 3 h, with a total extracted beam current of 6.5 mA. Five minutes after switching to Ar, the C^+ peak had decreased from 11 μA to 12.5 nA, corresponding to a background of 49 000 years (assuming a 1× modern sample).

Molecular Breakup

To study the degree of breakup of the CO_2 molecule, the source was installed on the test stand and operated on 0.2 sccm CO_2 with 170 W microwave power. A 10 mA beam was extracted at 24.5 kV extraction voltage. A mass analysis of the beam is presented in Fig. 5. Molecular breakup of the CO_2 is clearly demonstrated, with 20 % of the total beam current composed of $^{12}C^+$.

Discussion

The pulsed-CO_2 injection tests clearly demonstrate that injecting 1 μmol (20 μL at STP) of CO_2 into the microwave ion source gives a useful carbon current pulse, and that the pulse amplitude can be 1000 times larger than the background current.

When operating under conditions of steady gas flow, the microwave ion source efficiently converts carbon atoms flowing into the plasma generator into carbon ions. For the example quoted earlier (0.2 sccm CO_2 flow, 10 mA total beam current), the ratio of extracted $^{12}C^+$ to $^{12}C^0$ is ~14%. However, any comparison with other ion sources must take into account losses due to the charge-exchange process and transmission losses through the machine. The estimated losses for the microwave ion source coupled to the WHOI Tandetron are summarized in Table 1. We have assumed an equilibrium charge-exchange fraction of 15% for C^+/C^- at 20 kV. Fractions of 15 to 25% have been reported for carbon with various charge-exchange media at this energy [8]. We have also estimated a transmission of only 35% for this positive ion beam through the charge-exchange canal. This figure is based on our present experience with a conventional canal closely coupled to the diverging positive-ion beam extracted from the microwave source. The transmission could possibly be improved by focusing the beam to a waist in the midplane of the canal with electrostatic or magnetic lenses, or by designing a new canal to accommodate the positive-ion beam with lower losses. The stripping efficiency at 2.5 MV is assumed to be 47%. Thus, ignoring transmission losses through the machine, the estimated global

efficiency for an accelerated carbon beam produced from a gaseous CO_2 sample is approximately 0.3% at the detector for this system.

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Table 1: Estimated Transmission Efficiency for Microwave Ion Source and Tandetron System

Parameter	
CO_2 Flow Rate	0.2 sccm (6 μG/sec)
C^+ Production Efficiency	14%
Charge-Exchange Canal Transmission	35%
Equilibrium Charge-Exchange Fraction, C^+/C^-	15%
Stripping Efficiency at 2.5 MV	47%
Global Efficiency	0.3%

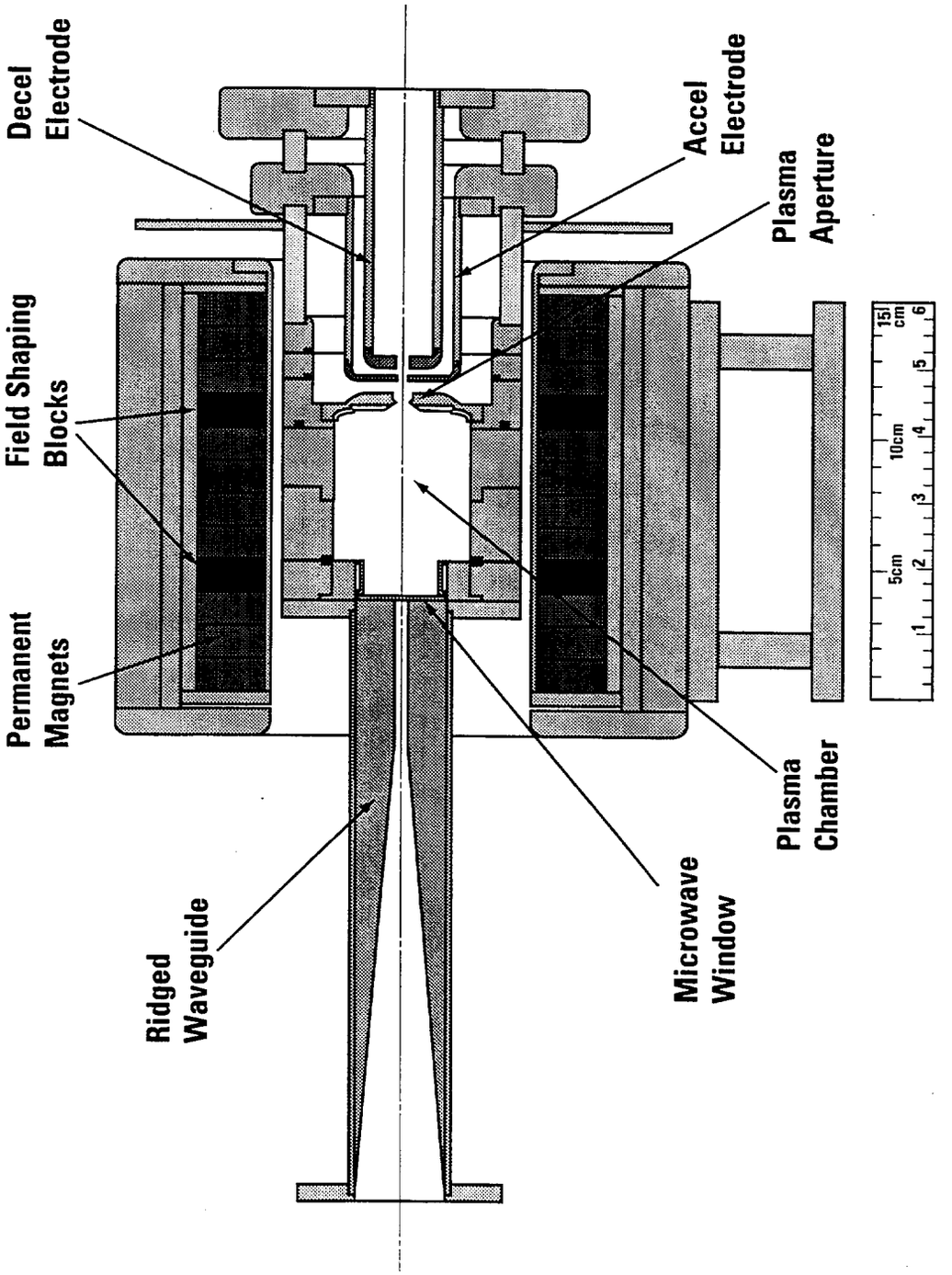

Figure 1 Compact permanent-magnet microwave ion source.

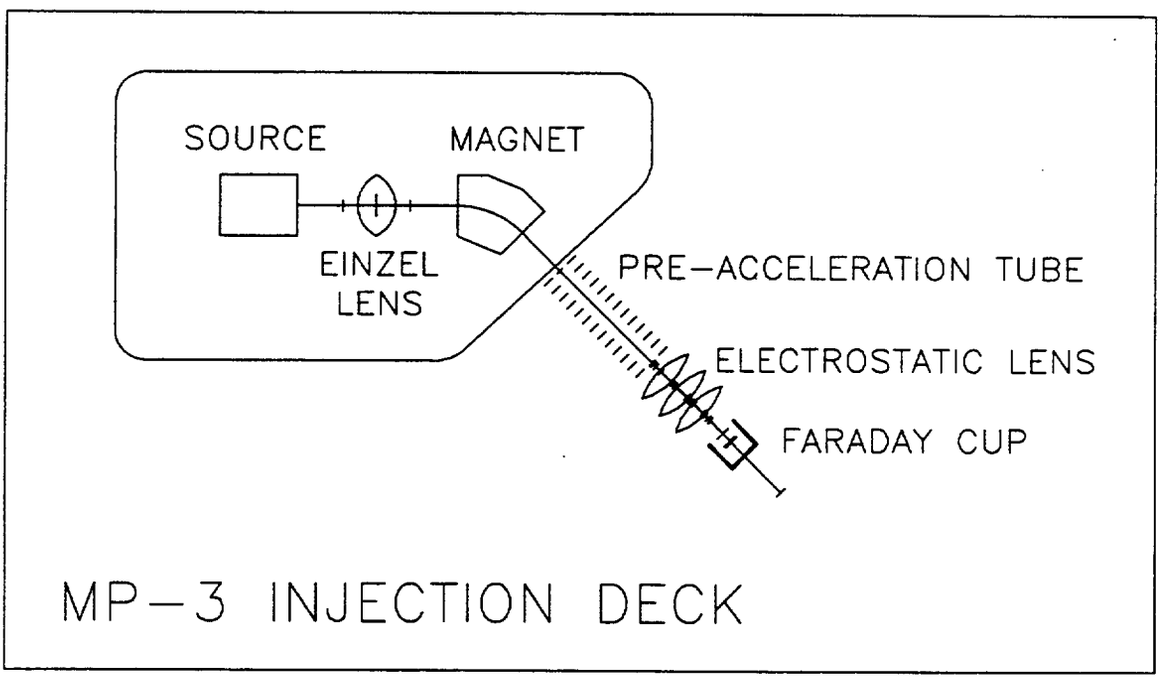

Figure 2 TASCC Tandem injector deck.

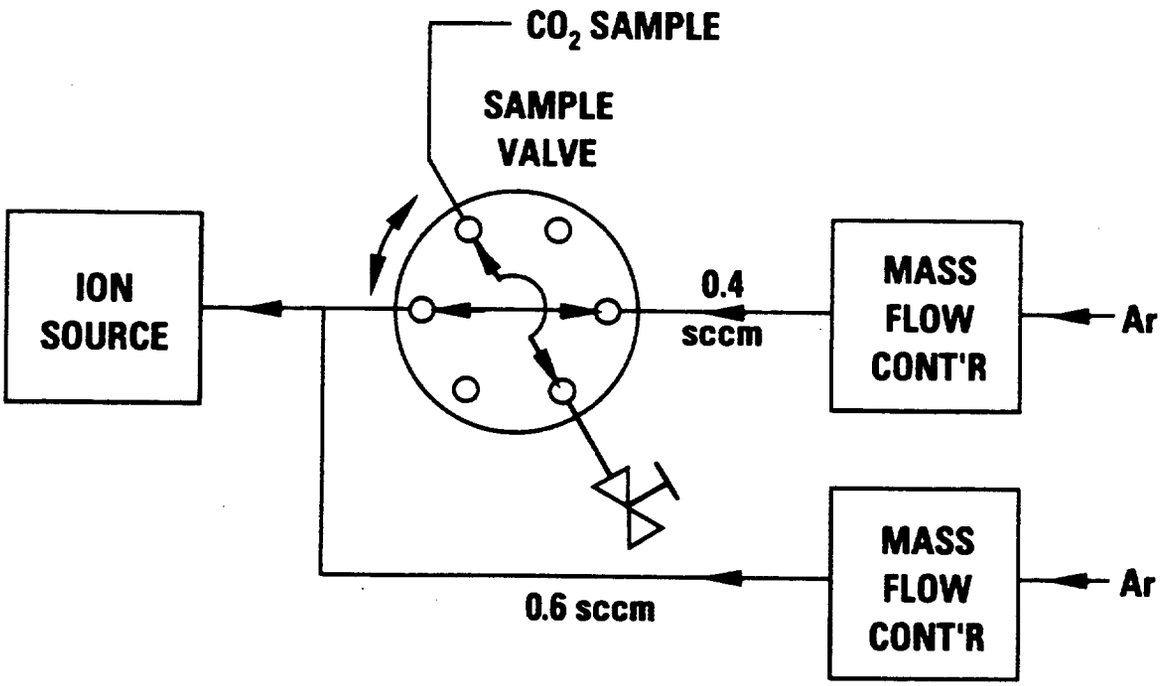

Figure 3 Sample injection valve.

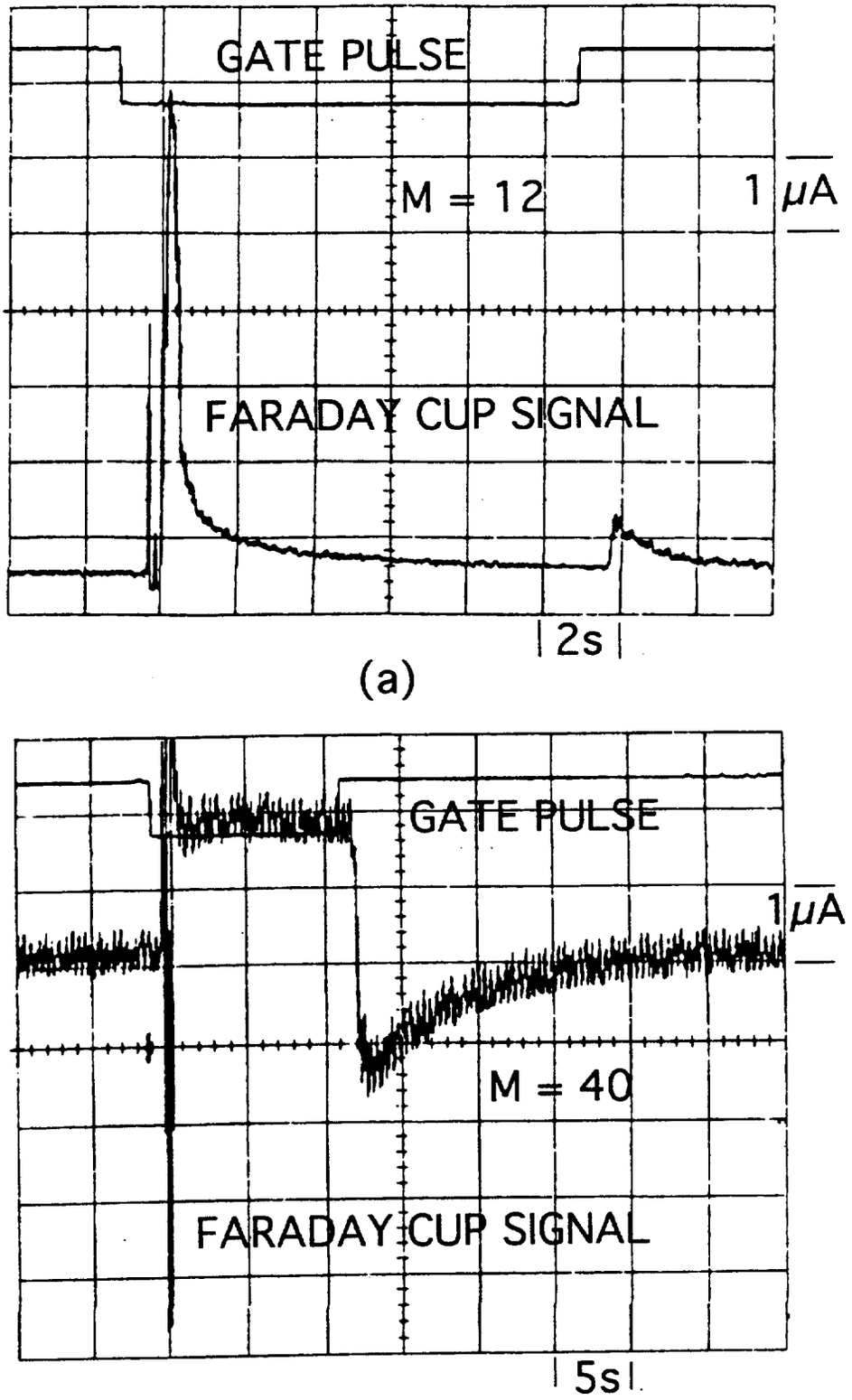

Figure 4 *Mass-analyzed Faraday cup currents during sample injection. Vertical scale is 1 μA/division, horizontal scale is (a) 2 s/division (b) 5 s/division.*

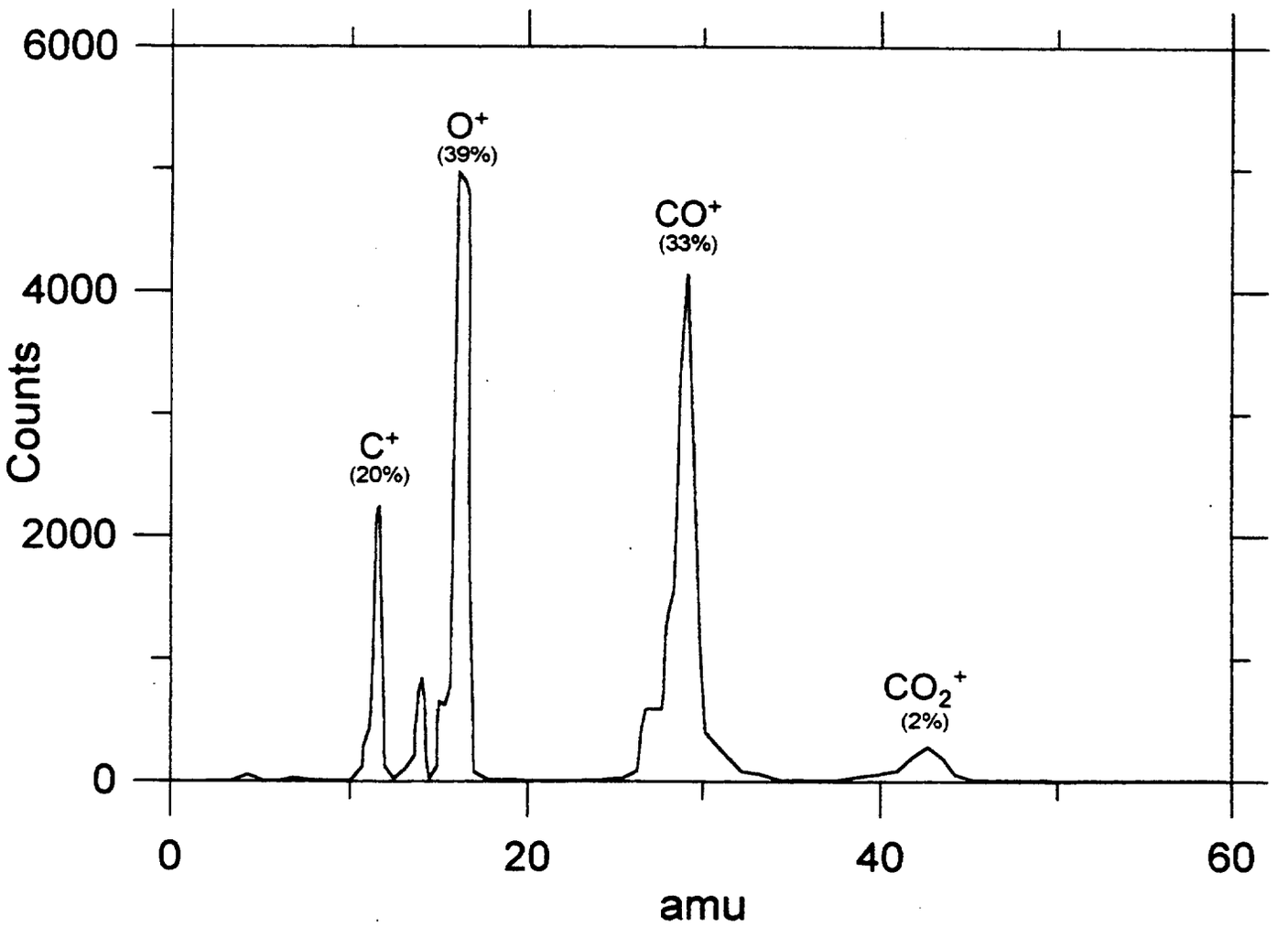

Figure 5 Mass spectrum from pure CO_2 feed. 0.2 sccm CO_2, 170 W microwave power, 24.5 kV extraction voltage, 10 mA beam current.