REVIEW OF SCIENTIFIC INSTRUMENTS VOLUME 74, NUMBER 7 JULY 2003

# Vacuum bench for the characterization of thermoionization ion sources

C. Guillermier and C. P. Lechene[a)]
*National Resource for Imaging Mass Spectrometry, Harvard Medical School and Brigham and Women's Hospital, Cambridge, Massachusetts 02139*

J. Hill
*J. A. Hill Instrument Services, Arlington, Massachusetts 02474*

F. Hillion
*CAMECA, 103 Blvd Saint-Denis, 92400 Courbevoie, France*

(Received 14 February 2003; accepted 17 April 2003)

We have designed a vacuum bench to study the parameters of thermoionization sources with the ultimate goal of obtaining high spatial resolution for biomedical applications of secondary ion mass spectrometry. In the bench, the source ionizer can be directly heated with an electron gun positioned perpendicular to the axis of the ion beam and focused with an optical system including slit lenses and a magnetic sector. The source cross over diameter is measured by forming the image of the source using an Einzel lens at a 1×magnification. The ion beam current is measured in a Faraday cup placed after a movable diaphragm. The temperature of the diverse elements of the ionizer assembly is measured through a mirror with a micropyrometer. Using the vacuum bench with a cesium carbonate source, we measured a 35 $\mu$m minimum cross over size, and we calculated a 400 A/cm$^2$/sr maximum brightness. We obtained an intense cesium ion beam when heating the ionizer with the electron gun. The vacuum bench will be used to compare the effect of the heating mode of the ionizer (i.e., indirect by filament electron emission or direct by electron beam) on the brightness of the cesium source, and to develop a thermoionization iodine negative ion source. © *2003 American Institute of Physics.* [DOI: 10.1063/1.1584086]

## I. INTRODUCTION

A new generation of secondary ion mass spectrometer combining high spatial and mass resolution with parallel detection of several atomic masses[1,2] opens the use of stable isotopes for imaging the location and measuring the turnover of labeled molecules in biomedical research. The capability for measuring isotope ratios in cellular organelles or in subcellular compartments of cubic nanometer size is of upmost importance for biomedical applications.

In our multi-isotope imaging mass spectrometer (MIMS),[3] an ion beam is scanned over the sample. We use a positive cesium ion beam with a 50 nm minimum diameter to produce negative secondary ions.[4] The cesium ions are produced with a thermoionization source.[5] We can also use an oxygen duoplasmatron negative ion source[6] to form positive secondary ions.[4] The brightness of this negative source is low, and its practical resolution is approximately 200 nm.

Although MIMS capabilities already constitute a breakthrough for biomedical applications of secondary ion mass spectrometry, any gain in resolution would be beneficial. In a secondary ion mass spectrometer with a scanning primary ion beam, a brighter primary ion source translates directly to higher spatial resolution and/or shorter acquisition time.

One of our goals is to attempt to increase the brightness of the cesium source. In the present cesium source, which is heated by electron bombardment from a filament around the periphery of the ionizer assembly, the ionizer plate cannot be the hottest part. As suggested by Slodzian,[7] this may create a cloud of charges that may decrease the production of cesium ions from the source and/or increase the minimum cross over size, both essential determinants of the brightness of the source. In order to decrease this space charge, and thus to potentially increase the source brightness, we decided to heat the source ionizer directly, at its center, using an electron beam.

Another goal is to design an iodine source[8,9] adapted to MIMS to replace the oxygen duoplasmatron negative primary ion source. Indeed, iodine negative primary ion sources have been shown to induce very high positive secondary ions yield.[10]

In this first article, we describe a vacuum bench designed to characterize both positive and negative primary ion sources by measuring the primary ion beam current and the cross over size. We also describe the heating of the source ionizer with an electron beam perpendicular to the ion beam.

## II. EXPERIMENTAL SETUP

### A. General description of the bench

The vacuum bench configuration is shown in Fig. 1. The vacuum chamber is composed of an 8 in. flange tee and two 8 in. flanges crosses [Fig. 1(a)]. Each cross has been machined with six 2.75 in. port flanges, three on each side. The front openings of the bench are equipped with two viewport

---

[a)] Electronic mail: cpl@harvard.edu

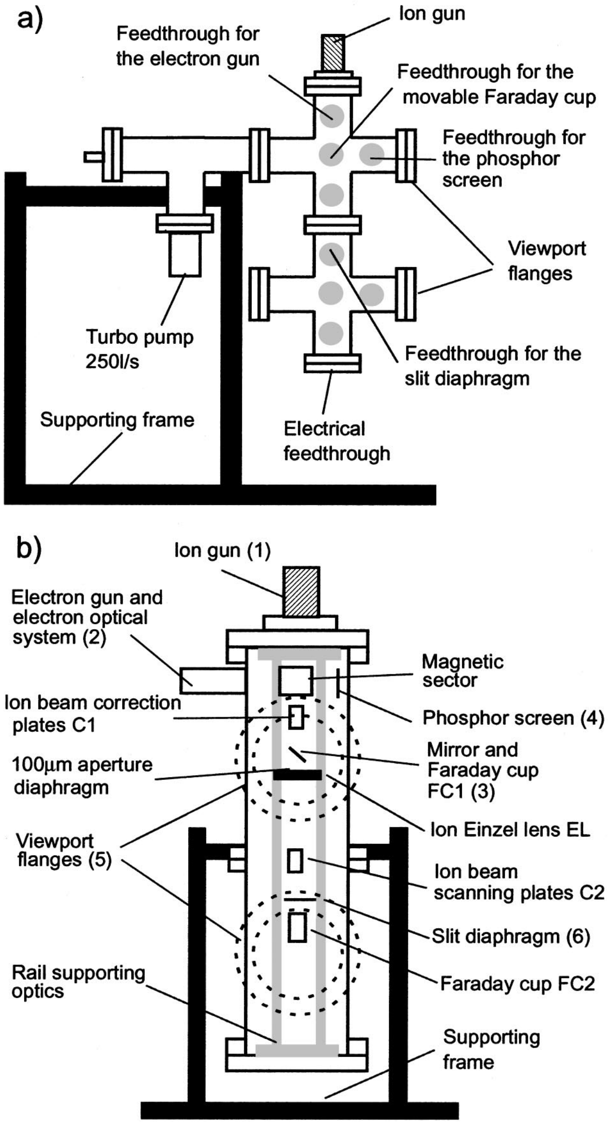

FIG. 1. Vacuum bench overview: (a) Side view and (b) front view.

flanges. The chamber is pumped down to $10^{-8}$ Torr by a 250 l/s Varian turbomolecular pump.

The cesium ion gun is installed on the top of the bench. The ion optics elements are supported on a rail or placed on mechanical movements [Fig. 1(b)]. The image of the virtual source cross over is formed in the plane of a movable slit diaphragm using an Einzel lens (EL). The magnification factor is 1. A 100 $\mu$m aperture, placed immediately before the lens, is centered in the hole of the ion lens grounded electrode. This aperture determines the solid angle (i.e., $3.4\times10^{-4}$ rad half angle) of the cesium ion beam emission. The cesium ion current is measured in a Faraday cup (FC2) placed directly after the slit diaphragm. A set of deflection plates (C1) placed before the Einzel lens can be used to center the beam in the 100 $\mu$m aperture. A set of deflection plates (C2), placed after the Einzel lens, will be used to scan the beam over the slit diaphragm maintained in a fixed position.

The electron-beam heating system is mounted on the bench perpendicular to the ion beam [Figs. 1(b) and 2]. A mirror and a Faraday cup (FC1) are attached to the same mechanical movement and placed between the corrector C1 and the Einzel lens EL. The mirror is oriented to observe the source ionizer assembly through the upper viewport flange. The ionizer temperature can thereby be measured using a micropyrometer equipped with a telescopic lens. The Faraday cup (FC1) can be moved in place of the mirror to measure electron or ion currents. A phosphor screen, mounted after the magnetic sector on the electron gun-slit lenses optical axis, allows us to visualize the electron-beam shape.

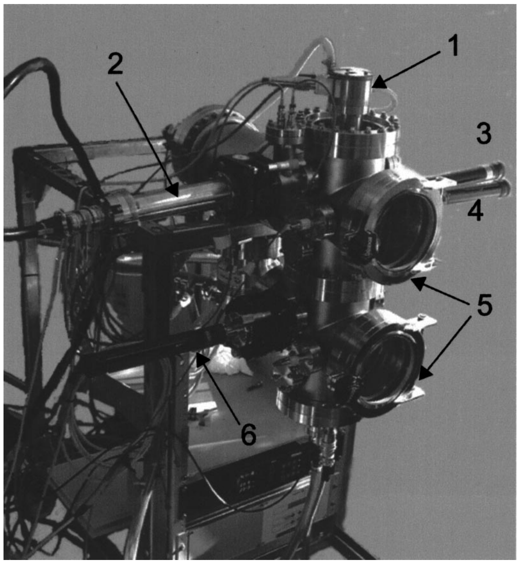

FIG. 2. Photo of the vacuum bench.

## B. Cesium ion source geometry

The ion gun is composed of a cesium thermal-ionization source set at a positive 8 kV potential, followed by one grounded electrode for the ion extraction. The source is composed of a reservoir connected to an ionizer assembly by a narrow tantalum drift tube, as illustrated in Fig. 3. A tungsten disk with four small holes serves as the ionizer plate and is held inside the ionizer assembly in close thermal contact with it. The reservoir and ionizer assembly are each surrounded by a tungsten filament at a near-ground potential. The reservoir and the ionizer are heated by electronic bom-

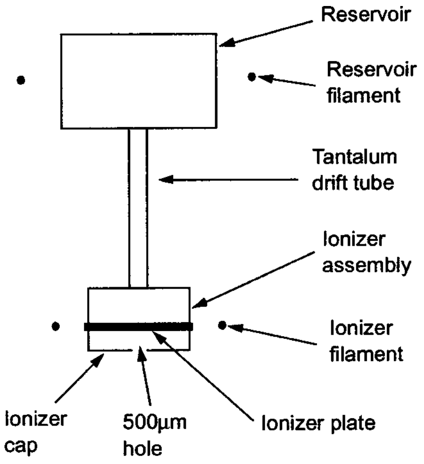

FIG. 3. Cesium ion source diagram.

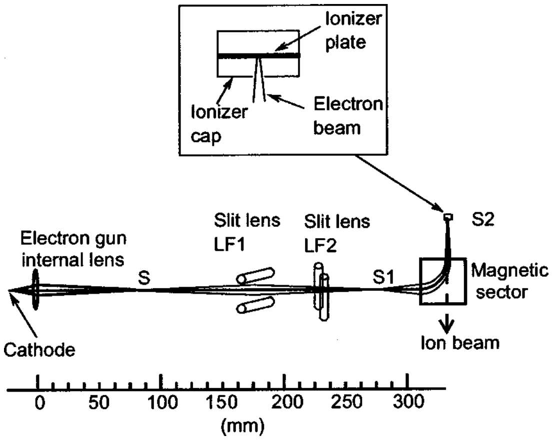

FIG. 4. Electron beam heating optic system.

bardment, and their temperatures are controlled by regulating the electron emission current from the tungsten filaments. The cesium vapor is produced by vaporization of a cesium carbonate salt contained in the reservoir. Cesium ions escape through a 500 $\mu$m diameter hole in the ionizer cap.

### C. Electron beam heating system

The electron beam heating system has been developed for surface ionization of both positive and negative ion sources. Depending on the nature of the source, however, the heating power required to heat the ionizer can vary widely. Since the goal of the system is to heat only the ionizer plate, the electron beam spot diameter, ideally, should be less than 500 $\mu$m. The EGG-3H Kimball electron gun was the best adapted to our experimental requirements. This gun, equipped with an internal Einzel lens, delivers an electron beam with an independently adjustable 100 eV to 14 keV energy range and a 0.2 to 10 mA emission current range. In preliminary experiments, we used the vacuum bench to characterize the electron beam parameters. From electron beam intensity profiles and beam spot size measurements, we found the experimental conditions for which 50% of the beam intensity was contained within a 300 $\mu$m spot.

The optical system, presented in Fig. 4, was designed to focus and center the electron beam on the source ionizer plate. This system consists of the mechanically alignable electron gun positioned perpendicular to the ion beam direction, a 3 mm aperture in front of two slit lenses (LF1 and LF2)[11] and a rectangular magnetic sector. The magnetic sector, with a 7 mm gap size, a 90° deflection angle, and a 23.5 mm radius of deflection, is made of two $35\,\text{mm}\times 37\,\text{mm}$ M$\mu$-metal rectangular pieces. The magnet is excited by two coils with 130 turns on a M$\mu$-metal core. For an electron-beam energy of 8 keV, a coil current of 1.5 A produces a deflection field of 125 G. A 3 mm circular aperture installed in front of the slit lens system limits geometrical aberrations. The entire optical system was designed and tested with the Simion software.

This system allows us to produce an image S2 with a $1\times$ magnification of the electron-beam focus spot S. Both S2 and the source ionizer are positioned in the magnetic sector image plane. The electron beam is focused with the slit lens LF2, producing (from S) the image S2 in the magnetic sector image plane. The electron beam is focused in the radial plane of the magnet so that the slit lens LF1 produces (from S) the image S1 located in the magnet object plane.

The electron-beam spot is directly observable on the ionizer cap through the mirror placed behind the first corrector [Fig. 1(b)]. Visual observation provides a convenient means of tuning the optical system to optimize the focus of the electron beam at the level of the ionizer cap. The electron-beam current is measured through the current in the high voltage power supply of the source. The magnetic field used for the electron-beam deflection has a very weak influence on the cesium ion beam trajectory.

## III. EXPERIMENTAL RESULTS

### A. Cesium ion source characterization

We calculated the cesium ion source brightness from the ion source cross over diameter and the ion intensity produced in a given solid angle:

$$B=\frac{I_{\mathrm{Cs}}}{\frac{\pi^2}{4}\cdot d_0^2\cdot \alpha_0^2}. \tag{1}$$

In Eq. (1), $B$ is the brightness of the source and $I_{\mathrm{Cs}}$ is the ion current emitted in a solid angle $\alpha_0$ from a virtual surface of diameter $d_0$, defined as the cross over (measured as *CO*).

To measure the cross over diameter, a real image of the virtual cross over is formed in the plane of the slit diaphragm with the ion lens. The slit diaphragm is moved across the ion beam, and the ion current intensity variation is recorded with the Faraday cup (FC2). A width $L_{10-90}$, corresponding to a variation of the intensity from 10% to 90% of the full beam intensity, is determined from the intensity profile. The $L_{10-90}$ width is measured at various ion lens voltages. The minimum value of the width $L_{10-90}$ is a good estimate of the source cross over diameter. Indeed, Simion calculations with our lens parameters have shown that corrections due to geometrical and chromatic aberrations of the lens could be neglected, given the 100 $\mu$m diaphragm aperture placed in front of the lens.

Beam sections of $L_{10-90}$ width, measured for various ion lens voltages, are presented in Fig. 5. For this experiment, the ionizer plate was set at 1280 °C, and the $I_r$ filament reservoir current was adjusted to produce a 200 pA ion beam current. The $L_{10-90}$ minimum value is obtained for a 4.25 kV voltage of the ion Einzel lens EL. For this particular 200 pA ion current value, we measured a 37 $\mu$m cross over diameter.

In order to fully characterize the source, the cross over diameter *CO* and the ion current ($I_{\mathrm{Cs}}$) were measured for different reservoir filament heating currents ($I_r$). The results are reported in Fig. 6; the brightness was derived from Eq. (1). The $I_{\mathrm{Cs}}$ ion current increases quickly with the reservoir filament current $I_r$. For a 0.7 mA $I_r$ value, the ion current reaches a maximum of 1.6 nA. Beyond this $I_r$ value, $I_{\mathrm{Cs}}$ exhibits a decrease, probably due to the lowering of the work function resulting from an excess in cesium coverage. The cross over diameters varied slightly with the reservoir filament current, going through a minimum of 35 $\mu$m. Thus,

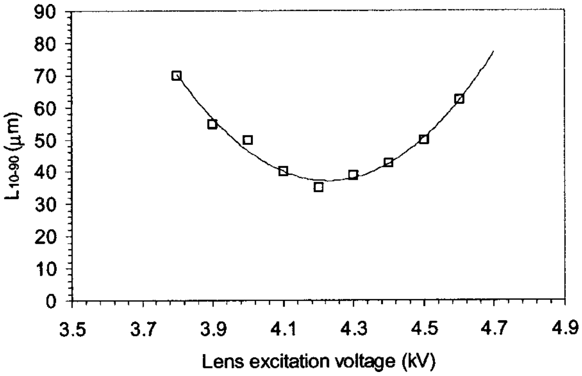

FIG. 5. Ion beam cross sections (width $L_{10-90}$) as a function of the ion lens excitation voltage. The minimum value of the $L_{10-90}$ width of the curve corresponds to the source cross over diameter. The ionizer was heated with the filament, and the source produced a 200 pA cesium ion current ($I_r$ $=0.2$ mA, ionizer temperature: 1225 °C).

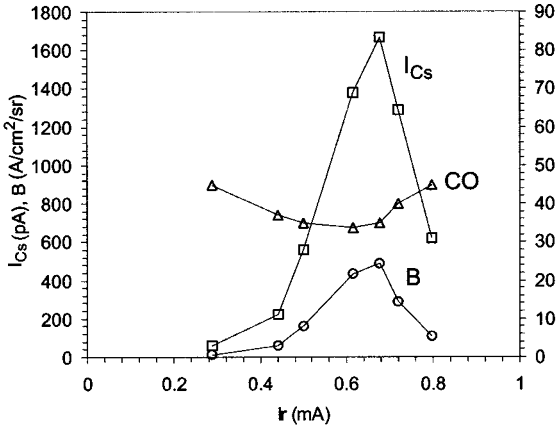

FIG. 6. Cesium ion current ($I_{\mathrm{Cs}}$), cross over size ($CO$) and source brightness ($B$) as a function of the reservoir heating, for a 100 $\mu$m aperture diaphragm. Ionizer temperature: 1225 °C.

under our conditions and with a cesium carbonate source, the brightness of the ion source was a direct function of the ion current. The maximum brightness value was 400 A/cm$^2$/sr.

Similar results were obtained under other experimental conditions. Indeed, we measured both a minimum cross over diameter of 35 $\mu$m and an increase in the brightness of the ion source with an increase in $I_{\mathrm{Cs}}$ using a 400 $\mu$m or a 1 mm diameter aperture diaphragm. We do not know why our results differ from the results of Slodzian *et al.*[5] and Hillion,[12] who observed that the cross over diameter directly increased with the cesium ion current. We should note, however, that we used a cesium carbonate salt instead of the cesium chromate salt used by the authors in Refs. 5 and 12.

## B. Electron beam heating: Measure of the ionizer plate and cap temperature

In order to increase the brightness of the source, our goal is to heat the ionizer plate directly with an electron beam, so as to decrease the putative space charge that may be created by the indirect filament heating presently used with the cesium ion source. We have measured the ionizer plate and the ionizer cap temperatures when heating with the electron beam. The ion source was polarized at 8 kV and the electron beam energy was 8 keV. The ionizer tungsten plate and cap temperatures were measured with a pyrometer. The electron beam was focused into the hole of the ionizer cap by tuning the slit lenses and the electron gun lens voltage values while observing the beam with the mirror. The tungsten plate and ionizer cap temperatures, as functions of the power delivered to the ion source, are shown in Fig. 7. The ionizer plate and cap temperatures are very close when using filament heating. In sharp contrast, there is a temperature difference of 100 °C between the ionizer plate and the cap when using electron beam heating. The electron beam power required to heat the tungsten plate in the 1180 °C–1300 °C range is in the 12–20 W power range.

We believe that the 100 °C temperature gap between the ionizer plate and cap that we obtained is a minimum value that will increase after we will have improved the electron gun—slit-lenses—magnetic sector assembly. Indeed, we measured a much greater temperature difference between the ionizer plate and cap when we placed the electron gun directly in front of the ionizer. In the configuration used to obtain the results shown in Fig. 7, with the electron gun perpendicular to the cesium ion beam, a part of the electron beam is still directly heating the ionizer cap. We are in the process of optimizing the electron gun optical system to increase the temperature difference between ionizer plate and ionizer cap.

## C. Cesium ion current

Finally, we studied the cesium ion production as a function of the reservoir filament emission current when the ionizer plate was heated with the electron beam. The cesium ion current was measured in the Faraday cup FC2 [Fig. 1(b)]; the Einzel ionic lens was at a potential of 4 kV. The results are presented for a variety of tungsten plate temperatures in Fig. 8. We measured a maximum cesium ion current of 750 pA. The maximum ion current was independent of the ionizer plate temperature. Yet, the reservoir filament emission

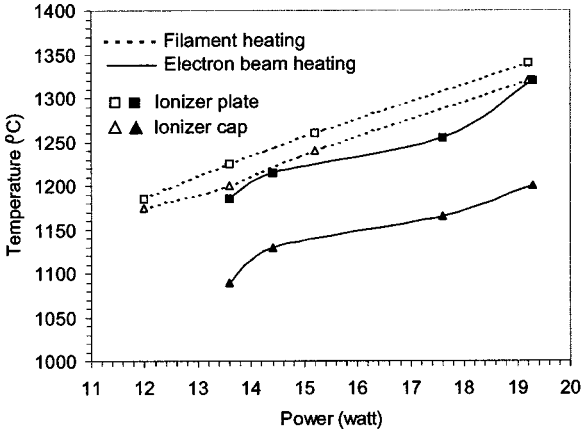

FIG. 7. Temperature of the ionizer cap and of the ionizer plate heated by filament emission current (dashed line) or electron beam (solid line). Temperature was measured for different heating powers of the ionizer assembly. The reservoir was not heated.

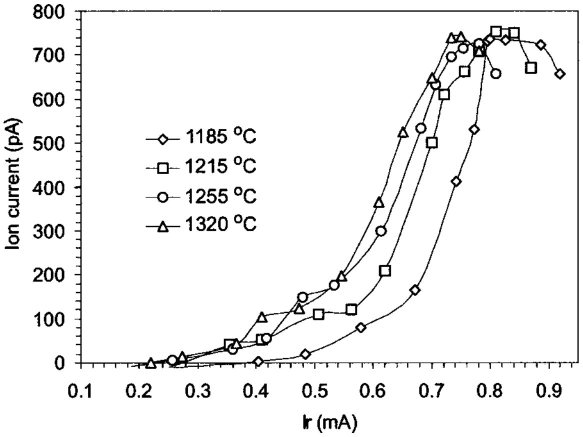

FIG. 8. Cesium ion current as a function of the reservoir heating for different ionizer plate temperatures, for the source ionizer heated with the electron beam.

current required to produce the maximum cesium ion current decreased with increasing temperature of the ionizer plate. These results indicate that we can use the electron gun to generate a cesium ion beam current. The brightness measurements will be performed after we have optimized the electron beam optical system.

## ACKNOWLEDGMENTS

This work was supported by Grant No. P41 RR 14579. The authors thank Professor G. Slodzian for many fruitful discussions.

[1] G. Slodzian, B. Daigne, F. Girard, and F. Boust, in *Secondary Ion Mass Spectrometry SIMS VI*, Proceedings of the Sixth International Conference on Secondary Ion Mass Spectrometry, Versailles, France, 1987, p. 189.
[2] G. Slodzian, B. Daigne, F. Girard, F. Boust, and F. Hillion, Biol. Cell. **74**, 43 (1992).
[3] MIMS is the prototype of the NanoSims50, Cameca.
[4] C. A. Andersen, Int. J. Mass Spectrom. Ion Phys. **3**, 413 (1970).
[5] G. Slodzian, B. Daigne, F. Girard, F. Boust, and F. Hillion, in *Secondary Ion Mass Spectrometry SIMS VIII*, Proceedings of the Eighth International Conference on Secondary Ion Mass Spectrometry, Amsterdam, Netherlands, 1991, edited by A. Benninghoven, K. T. Janssen, J. Tumpner, and H. W. Werner (Wiley, Chichester, 1992), p. 227.
[6] M. Von Ardenne, Technik. **11**, 65 (1956).
[7] G. Slodzian (private communication).
[8] J. Pelletier, C. Pomot, and J. Cocagne, J. Appl. Phys. **50**, 4517 (1979).
[9] J. Zhou, W. Ens, and K. G. Standing, Int. J. Mass Spectrom. Ion Processes **139**, 146 (1995).
[10] C. A. Andersen and H. J. Liebl, US Patent No. 3,508,045 (12 July 1970).
[11] G. Slodzian, M. Chaintreau, and R. Dennebouy, in *Secondary Ion Mass Spectrometry SIMS V*, Proceedings of the Fifth International Conference on Secondary Ion Mass Spectrometry, Washington, DC, 1985, edited by R. J. Colton, A. Benninghoven, D. S. Simon, and H. W. Werner (Springer, New York, 1986), p. 158.
[12] F. Hillion (private communication).