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OMS Letters

Dear Sir

A Device For Sensing Ion Source Conditions In FAB MS

We recently reported the hybridization of electrochemistry and fast atom bombardment (FAB) mass spectrometry (MS) to create a new "hyphenated" technique, referred to as "electrochemically assisted fast atom bombardment (EFAB) mass spectrometry [1]". This technique improves the applicability of FAB MS in selectivity and the range of compounds that it can analyze, and also reduces mass spectral complications due to matrix-related artifacts [2]. Samples are activated by electrolysis, carried out directly in the sample matrix. A modified FAB sample probe was constructed containing a small electrolytic cell on the tip (Figure 1) [3]. In operation, one electrode is held at the normal sample-probe/ion-source voltage, while the other electrode can be continuously varied ±15 volts, with respect to the ion-source voltage, to create electrochemical potentials. We have also found this device useful in determining ion source conditions, however.

In particular, this new sample probe can be utilized in optimizing 1) operation of the FAB particle gun, 2) position of the sample probe, and, among other possibilities, 3) determination of the temperature of the sample matrix under bombardment conditions. Noteworthy of the first two proposed uses is the use of the EFAB probe as a sample-less instrument monitor, thus sparing the mass spectrometer from unnecessary contamination.

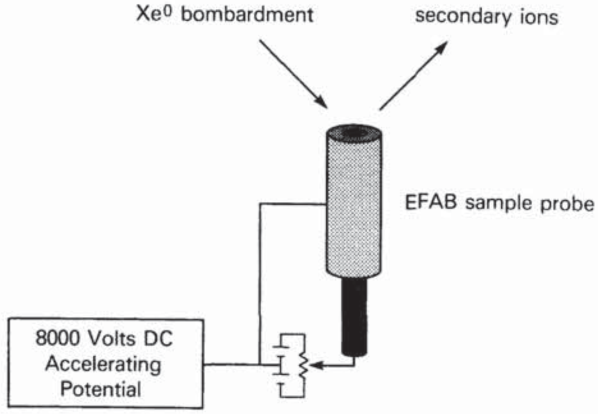

Figure 1. Diagrammatic representation of the EFAB sample probe (see text).

Although the FAB particle gun may be used with almost any combination of particle density and energy, usually there is an optimum combination of these parameters resulting in minimal scan-to-scan data variation. This condition may be found by trial and error, but use of the EFAB sample probe provides a direct approach to the problem. If the EFAB sample probe is introduced into the instrument empty with the battery-potentiometer circuit shown in Figure 1 replaced by a voltmeter and the resulting voltage between the two electrodes of the cell (open cell voltage) noted under various bombardment conditions, that combination of particle density and energy which gives the highest open cell voltage will, under normal FAB operation, produce data that varies least over the time of the experiment. Optimum input for our fast atom gun is 0.4 mA at 8500 volts (using xenon gas) and 0.35 mA at 4500 volts (using argon gas). As a rule, the fast atom gun requires maintenance if these settings don't give the desired results.

The axial and rotational positions of the probe in the ion source may also be investigated by bombarding the empty cell of the EFAB sample probe and observing the resulting open cell voltage. Maximum exposure of the empty cell to the particle beam results in an open cell voltage maximum. This allows verification of the probe positioning and the alignment of the

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particle beam with the sample target. These simple tests involving the particle gun and sample probe permit the instrument operator to verify some of the conditions found in the bombardment experiment. Note that these measurements are performed without a need for ion sorting or detection by the mass spectrometer, and without the need for any chemical contamination of the vacuum system.

Other applications of the EFAB sample probe can be envisioned. For example, it is known that glycerol gives optimum sensitivity in FAB at a temperature of 25°C [4]. Although the experimental details of the temperature measurement of this matrix are not given in that work, the adaptability of the EFAB device to make such a measurement is now shown. If the EFAB sample probe is loaded with a sample of glycerol and placed in an oven, the electrical conductivity of the glycerol sample under conditions of constant voltage (ion-source voltage is not used here, only the probe's ±15 V power supply) can then be monitored as a function of temperature. It is found that conductivity increases linearly with temperature. The resulting "electrical current *vs.* temperature" plot allows for utilization of the EFAB sample probe as an "electrical conductivity thermometer," so that one can measure the temperature of the sample matrix under bombardment conditions. It is interesting to note, for example, that the electrical conductivity of glycerol subjected to 10 minutes of particle bombardment is, at any time during the bombardment, no different than that measured for a glycerol sample maintained at a temperature of about 25°C in an oven. One may then conclude that fast atom bombardment does not significantly contribute to heating the matrix bulk, and therefore the FAB experiment is performed macroscopically at ambient temperature [5].

This present report involves our experiences with a Kratos MS-50 mass spectrometer equipped with a Kratos DS-55 data system (Data General Nova 4X minicomputer, employing the RDOS operating system) and a Saddle-Field neutral-beam fast atom gun manufactured by Ion Tech, Ltd. The EFAB sample probe is a non-commercial prototype.

We are continuing our investigations into the design, chemistry, and applications of electrochemically assisted fast atom bombardment mass spectrometry, the results of which will be reported elsewhere.

Yours

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