Mass analysis of CF_3I decomposition in a surface barrier discharge

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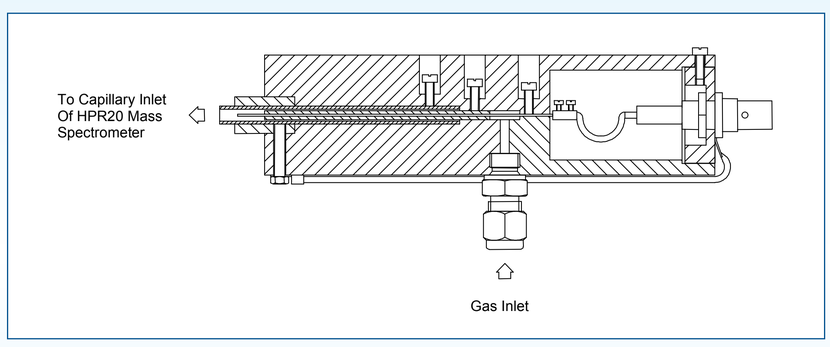


Abstract

Mass analysis of trifluoro-iodo-methane in a Surface Barrier Discharge. Surface barrier discharges operated at atmospheric pressure are effective chemical reactors. Mass analysis of the reaction products is possible using suitable high pressure mass spectrometer systems. As an example of the behaviour of simple surface barrier reactor (SBDs), experiments on the decomposition of CF_3I are described in which the output from the reactor is admitted via a capillary inlet system into a Hiden HPR20 mass spectrometer. The discharge was operated using helium as the carrier gas. The observed mass spectra are discussed in terms of the plasma dissociation and the subsequent ionisation of the dissociated products in the electron impact ionisation source of the mass spectrometer. When oxygen was added to the gas mixture in the SBD, CF_xO species were generated in the plasma. Their influence on the observed mass spectra is shown. The results demonstrate aspects of the capabilities of SBDs for dissociating halocarbon gases at atmospheric pressure and the possibilities of direct mass spectrometric monitoring of such processes.

Equipment

Figure 1



The surface barrier discharge (SBD) reactor is shown in figure 1. It consisted of a glass tube,. 6mm in internal diameter, with a central, 1mm diameter, stainless steel electrode loosely wrapped with several layers of aluminium foil to increase the surface area presented to the gas flowing through the reactor. A grounded tube was also fitted around the outer surface of the glass tube, to form the other SBD electrode. The discharge was operated using a 50kHz power supply similar to that described by Law et al.[1]. The power supplied was about 5 watts. The discharge was operated at atmospheric pressure using helium as the carrier gas to which small partial pressures of CF_3I were added. The output from the reactor was monitored using a Hiden HPR20 mass spectrometer system, by sampling through a capillary tube whose purpose was to reduce the pressure from the atmospheric pressure used in the reactor to around the 5.10^{-7} Torr required by the ionization source of the mass spectrometer.



HPR20 QIC Gas Analysis System



Heated 2 metre QIC capillary inlet

Experimental data

Mass spectra were obtained for two gas mixtures:

a) CF_3I (plus helium carrier),

b) CF_3I with added oxygen (plus helium carrier).

In both cases, the spectra obtained with the plasma operating were compared with those obtained with the plasma switched off.

Figure 2 shows a mass scan (m/e>5) for the CF_3I/helium mixture with no plasma operating. The dominant peaks include the expected fragments: CF_2I CF_3, CF, F and I. There was also a peak at m/e=63.5 corresponding to I^{++}

Figure 2

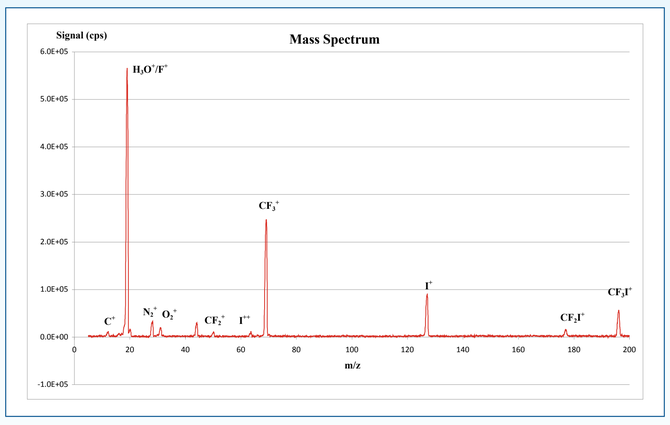


Figure 3 shows the intensities of the dissociation fragments CF_3^+ and I^+ for several on/off cycles of the plasma in the SBD reactor. The intensities of the fragments decreased while the plasma was on. The same behavior was observed for the CF^+ and CF_2^+ fragments.

Figure 3

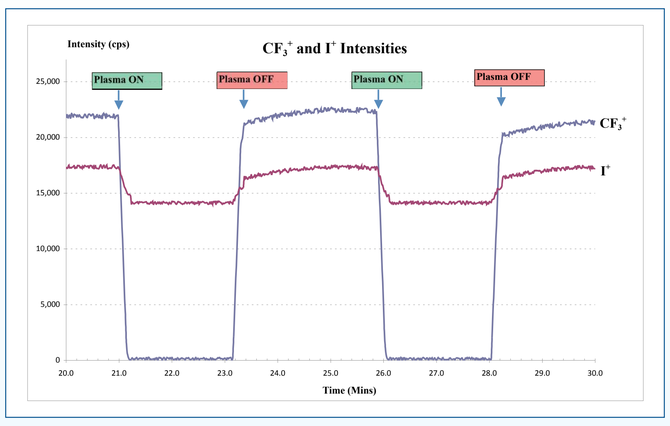
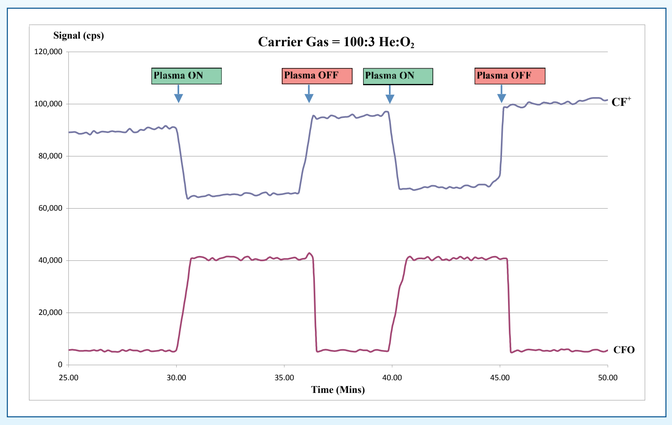


Figure 4 shows the corresponding data obtained with 3% of oxygen added to the gas mixture. The data are presented as a pair of scans for CF^+ and CFO^+. Similar scans to those of figure 4 were obtained for the corresponding pair: CF_2^+ and CF_2O^+.

Figure 4



Discussion

The decrease in intensity of the signals for CF_3 and the other dissociation products when the plasma was on is at first sight unexpected, since the plasma causes dissociation of the CF_3I. However, the main contribution to the peaks for these species comes from dissociative ionisation in the source of the mass spectrometer. The energy of these electrons was 50eV which is well above the onset energy for production of, for example, CF_3+ which is around 13eV, as is seen from figure 5. In figure 6, when the plasma was turned on, small CF_3+ signals were recorded for source energies of less than 13eV, down to about 8.5eV, which is the ionization energy for the CF_3 radical. More intense plasmas would be expected to increase the dissociation and give larger signals for energies between 9 and 13eV. Similar data was obtained for the other dissociation fragments.

With oxygen added to the gas mixture, masses corresponding to CF_xO were observed, and as seen in figure 4 the intensities of these mass peaks increased when the plasma was turned on. The behavior of the m/e = 47 and 66 peaks (taken to be CFO and CF_2O) is consistent with the view that they originate from CFx generated in the plasma.

The reduction in m/e =31, 50, and 69 when the plasma was turned on was not equal to the increase in the corresponding m/e = 47, 66 and 85 oxides of these fragments, but the sum of the observed intensities of the m/e =31,47,50,66, 69 and 85 signals with the plasma on was equal to that with the plasma off.

We take the view that the reduction in the combined intensities of the CF, CF_2, and CF_3 signals due to the decomposition of CF_3I in the plasma is balanced by the abundances of the CFO and CF_2O created in the plasma from the decomposition products.

Figure 5

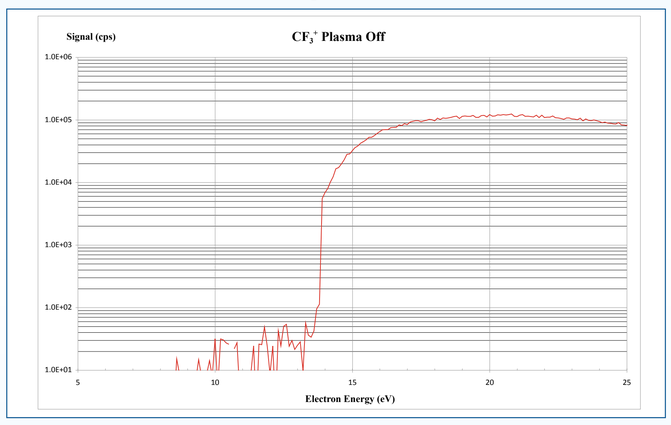
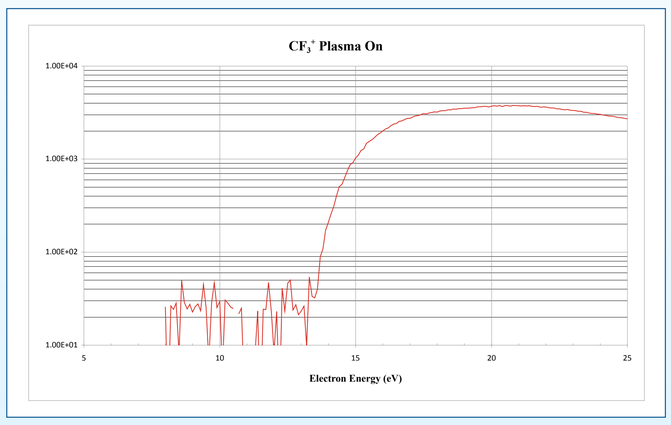


Figure 6



Conclusions

The present work has demonstrated the capabilities of SBD sources for the dissociation of halocarbon gases at atmospheric pressure and the possibility of directly monitoring such processes using a Hiden HPR20 mass spectrometer equipped with a capillary inlet system. The introduction of small quantities of oxygen into the gas mixture was significant.

References

[1] V.J.Law et.al presented at Frontiers in Low Temperature Plasma Diagnostics, Beverley UK, April 2007.

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