PARTIAL PRESSURE ANALYSIS OF PLASMAS

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ABSTRACT

The application of partial pressure analysis for plasma diagnostic measurements is reviewed. A comparison is made between the techniques of plasma flux analysis and partial pressure analysis for mass spectrometry of plasmas. Emphasis is given to the application of quadrupole mass spectrometers (QMS). The interface problems associated with the coupling of a QMS to a plasma device are discussed including: differential-pumping requirements, electromagnetic interferences from the plasma environment, the detection of surface-active species, ion source interactions, and calibration procedures. Example measurements are presented from process monitoring of glow discharge plasmas which are useful for cleaning and conditioning vacuum vessels.

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I. Introduction

Mass spectrometric analysis is a routine vacuum measurement that has become increasingly useful as a plasma diagnostic technique for both high temperature fusion plasmas[1] and low temperature (process) plasmas.[2] Mass spectrometry is the primary diagnostic for optimizing and process monitoring plasma discharge cleaning techniques[3] which have been developed for conditioning the large vacuum vessels of fusion devices and particle accelerators. The relative simplicity of the mass spectra of the gaseous components in a plasma process, in comparison to the optical emission spectra,[4-6] makes mass spectrometry useful for quantifying plasma composition and impurity concentrations for the study of gas-phase plasma reactions and plasma-surface interactions.

One can distinguish two general schemes for mass spectrometric analysis of plasmas: flux analysis and partial pressure analysis. Flux analysis involves sampling the plasma directly by coupling the line-of-sight emission of plasma particles through a small aperture into the ion optics of the mass spectrometer. This method is best suited for plasma species and energy analysis. In comparison, partial pressure analysis allows simpler vacuum connections between the mass spectrometer and plasma chamber, and generally less stringent differential pumping requirements. It is the method of choice for monitoring glow discharge treatments of vacuum vessels or large area materials processing because the measured signals are representative of the integrated effects of the relevant plasma-surface interactions.

This paper will focus on the partial pressure analysis method as it is more widely applied in fusion plasma and process plasma research and development. Emphasis is given to the most widely used type of mass spectrometer for real-time monitoring of plasma conditions, the quadrupole

mass spectrometer (QMS). A number of important interface problems associated with the coupling of a QMS to a plasma device will be discussed in this paper, including: (1) the trade-off of pressure reduction with system dynamic range when differential-pumping is required; (2) electromagnetic interferences (ultraviolet, soft X-ray, and magnetic field effects) from the plasma environment; (3) the difficulty of detection of surface-active species; (4) the decrease in signal-to-noise due to ion source interactions; and (5) the need to characterize and standardize calibration procedures.

II. Plasma Flux Analysis

Mass spectrometry has for many years been an important diagnostic tool for basic studies of the atomic and chemical processes that occur in both low temperature and high temperature (fusion) plasmas. A review article by Drawin[7] (1967) discusses the experimental configurations which were used for mass spectrometric studies of flames, arcs, glow discharges, and the fusion devices of the 1950-60's. These studies typically employed instrumentation for flux analysis of plasma particles which streamed through a sampling aperture located at or near the plasma vessel wall. After extraction from the plasma, the particle flux was energy and/or mass analyzed. Figure 1a shows a schematic diagram of the typical apparatus required for plasma flux analysis. Of the four stages shown in Fig. 1a, (1) the particle extraction/collimation optics, (2) the energy analyzer, (3) the mass analyzer, and (4) the ion detector, the most complicated component in the system in terms of its design and effect on signal collection and interpretation, is the extraction optics. The simplest extraction system employs an aperture which separates the plasma vessel from a separately pumped chamber containing the

energy and mass spectrometers. The resulting flux of neutral particles from the plasma (background atomic and molecular neutrals and plasma charge-exchange neutrals) will be collimated by the aperture according to the kinetic-gas laws appropriate to the neutral pressure in the vicinity of the aperture. Once collimated by the sampling aperture, the flux of neutrals can be ionized to allow energy and mass analysis by electrostatic and/or magnetic deflection. For particles with thermal or near thermal energies (0.02 - 10 eV), the ionization is usually performed with an electron bombardment ionizer,[8] whereas stripping cells[9] are used for ionizing the higher energy charge-exchange neutrals (100 eV - 100 keV) that are present in contemporary fusion devices.[10] It is important to notice that these two conventional ionization techniques leave a gap in the energy spectrum (10-100 eV) which is where the peak charge-exchange fluxes occur in tokamak fusion devices.[11] To bridge this shortcoming, surface ionization detectors have been developed with time-of-flight analyzers,[12] which have extended the detected charge-exchange energy spectrum down to 5 eV for H_2.[13]

Flux analysis of plasma ions is not as straightforward as the analysis of neutrals because of the electrostatics of the interaction of the sampling aperture with the plasma. The nature of the charged particle flux through the aperture depends on the plasma conditions in the vicinity of the aperture, the geometry and electrostatic potential of the aperture. This problem is not solvable in general; however, a few special cases of practical importance are well treated in the literature. Drawin[7] discusses three configurations for extracting plasma ions from an electrostatic plasma (i.e., glow discharge), that depend on the relative magnitude of the ion mean-free-paths, λ_i, the Debye shielding length, λ_D, and the extraction aperture diameter, D. The simplest case is collisionless extraction through a plasma sheath which occurs

when $\lambda_i >> D_1, \lambda_D$. In this case, if the extraction aperture is biased negatively (or is simply an aperture in the cathode if the plasma is a dc glow discharge), the sampled flux is described by the ion saturation current, $I_s^+ \sim n_i(2kT_i/\pi m_i)^{1/2}$, derivable by electrostatic probe theory. The case of collision dominated extraction through a plasma sheath, $\lambda_i < \lambda_D$, D is more complicated. Differing collision processes between plasma particles of differing mass can lead to the development of an extracted ion flux with a very different ion population from the unperturbed plasma far from the vicinity of the aperture. The third configuration described by Drawin is extraction by ambipolar effusion which results when the extraction aperture is biased at the floating potential, allowing an ambipolar current $j \sim D_{amb} \nabla n$ to effuse through the aperture. In certain cases, such as the analysis of the effusive flux from the positive column of glow discharge,[14] this method can lead to a relationship between the extracted and plasma ion flux that is similar to the ion saturation method.

Flux analysis of magnetized plasmas complicates the analysis of the extraction optics and, in addition, can cause practical problems with the design of the downstream instrumentation because of the necessity to shield fringing or stray magnetic fields from the plasma magnetic system. For flux sampling of neutral emissions from magnetized plasmas, the usual experimental approach is to move the apparatus a sufficient distance from the plasma vessel that ensures that the effects of stray magnetic fields on the spectrometer are negligible or can be minimized by shielding. A concomitant loss in signal intensity and a more stringent differential-pumping requirement are necessary with this approach because of the longer neutral flight path. This approach is impractical for sampling ion fluxes from magnetized plasmas. Because of the confinement of ions along magnetic field lines, the ion flux effusing

through an aperture will vary with the geometry of the aperture relative to the magnetic field geometry. If the aperture axis is parallel to the magnetic field axis, the streaming flux will be sampled; whereas, if the aperture axis is perpendicular to the magnetic field axis, the cross-field diffusive flux will be sampled. Both fluxes can be related to the local plasma ion density, but the analysis is complicated. This problem has been considered in the design of probes for space plasma research,[15] and more recently by Staib[16] in the design of a minature ExB mass spectrometer for in situ measurements of ion fluxes in the edge-plasma of fusion devices. Note that by using in situ spectrometers that make use of the ambient magnetic fields, the problem of shielding a high magnetic field from a sensitive instrument is circumvented.

In addition to the references already cited, there are many examples of plasma flux analysis in the literature.[7,17] Much of the early work (as cited by Drawin[7]) was concerned with analysis of dc and rf glow discharges. Often-cited examples are the measurements of the Vanderslice and Davis[18] and Houston and Uhl,[19] of the energy distributions of ions accelerated through the cathode-fall of argon glow discharges. Recently, similar measurements were made by Mathewson et al.[20] for the Ar/O_2 and H_2 glow discharge configurations which are useful for discharge cleaning of accelerator and fusion device vacuum vessels. Flux analysis has been applied to plasma sputtering and etching devices for plasma species analysis. The technique is capable of identifying ionized and neutral species in the vicinity of the substrate plane of sputter-deposition devices such that the chemical composition of the sputter deposited film can be monitored in real-time.[21] The line-of-sight sampling of plasma flux analysis is useful for analyzing reactive or transient species in plasma-etching systems,[22-25] and has proved helpful for identifying reaction mechanisms in such devices.[26,27] Similarly, flux analysis has been

used for identifying the ion species in rf[28] and high current plasma ion sources.[29,30]

These measurements illustrate the primary advantage of the flux analysis technique for mass spectrometry of plasmas. With proper design of the sampling aperture and extraction optics, plasma ions and neutrals that are representative of the plasma composition in the vicinity of the aperture, can be sampled such that the extracted species is unaffected by subsequent gas-phase and surface collisions. The primary disadvantages of the flux analysis techniques are basically practical concerns: (1) the technique requires careful design of the sampling and extraction optics, which cannot always be made nonperturbing to the plasma device. (2) The differential-pumping requirements to maintain collision-free conditions may be nontrivial especially for sampling the higher pressure process plasmas. (3) For fusion device applications, the magnetic shielding requirements for the spectrometers can be severe in terms of the design trade-off between the level of required shielding and the loss of signal intensity with remote separation of the spectrometer from the plasma device.

III. Partial Pressure Analysis

The alternative experimental scheme for mass spectrometry of plasmas, which employs the same or somewhat simpler instrumentation as required for flux analysis, is partial pressure analysis. Figure 1b shows a schematic diagram of the typical experimental arrangement for partial pressure analysis in which a quadrupole mass spectrometer (QMS) is indicated for the partial pressure measurement. Depending on the operating pressure regime of the plasma device, there is either a high conductance or low conductance

connection tube between the plasma device and the QMS chamber, which is usually differentially pumped.

The choice of a quadrupole mass spectrometer[8] as the commonly used instrument for this type of measurement is the result of the following: (1) the QMS is widely available as a commercial instrument; (2) its design and operation are simple; and (3) the QMS has high sensitivity at reasonable resolution with moderate mass discrimination over the essentially small mass range (0-200 amu) of interest to plasma processes and plasma chemistry.

The instrumentation for partial pressure analysis can be made simpler than that for flux analysis, because the extraction optics and energy analyzing stages are unnecessary. Usually, plasma particles are allowed to thermalize before reaching the QMS chamber by repeated collisions on the connection tube wall. Thus, for the typical electron-bombardment ionizer of a QMS the mass analyzed ion current, which is proportional to the local gas density, can be calibrated in terms of a partial pressure equilibrated to the temperature of the QMS chamber walls.

The primary experimental attribute of partial pressure analysis is the fact that the technique samples volatile reaction products resulting from the integral effects of plasma-wall interactions summed over the entire surface area of the plasma chamber. In contrast, the flux analysis technique samples only the reaction products that are representative of plasma-material interactions in the vicinity of the sampling aperture. This advantage of partial pressure analysis is particularly useful when mass spectrometry is used as a diagnostic or monitor of: (1) plasma-etching[26] of large area samples; (2) discharge cleaning[3] for large vacuum vessels; (3) plasma-wall interactions[31] in fusion devices, such as impurity generation from wall sputtering and hydrogen isotope replacement processes in wall materials.

The combination of the latter two applications, and the advantages of

simpler instrumentation with commercial availability, have resulted in the dominance of partial pressure analysis for mass spectrometric studies in fusion research of the past decade.[32-34] Partial pressure analysis has also been applied widely in the field of process plasmas, particularly as a process monitor and end-point detector for plasma sputtering and etching.[2,45-47] However, there are a number of problems concerned with interface of a partial pressure analyzer to a plasma device which have been poorly addressed in the literature and commercial documentation, that can lead to confusing and erroneous data interpretation. The following sections will address these interface problems in detail.

A. Pressure Reduction vs. Dynamic Range

Let us consider the operating specifications of the typical high performance QMS that would be used for partial pressure analysis. The operational pressure range with an electron multiplier ion detector is 10^{-11}-10^{-4} Torr. The lower limit is considerably above the minimum partial pressures which have been detected with a QMS (< 10^{-13} Torr), but does represent a practical detection limit defined as a signal-to-noise ratio of approximately unity after integration of the minimal output current (10^{-11}-10^{-12} A) of a 14 stage CuBe multiplier over a period of 1 sec. The high pressure limit (10^{-4} Torr) is usually defined as the pressure at which gas scattering becomes important in the ion source and the signals become non-linear with total pressure. It is a rare experimental situation in which one has the luxury of the full factor of 10^{7} in dynamic range that such a QMS affords, because of the presence of background signals at mass numbers of practical interest. Background levels are almost always a concern when sampling plasma systems because of the chemically active gases that are

involved, and the increased chemical reactivity of volatiles caused by dissociation, excitation, and ionization in the plasma.

To rescale the operational pressure range of the QMS so that the upper pressure limit encompasses the regime of glow discharge plasmas (10^{-3}-1 Torr), it is necessary to use a pressure reduction stage between the plasma chamber and QMS chamber. This pressure reduction can be accomplished with a low conductance connection tube with differential pumping of the QMS chamber as shown in Fig. 1b.

By such means, the upper pressure limit can be scaled arbitrarily, and the full dynamic range of measurement is available over the rescaled range. The minimum detectable pressure in the plasma chamber would increase in proportion to the rescaled upper pressure limit, assuming there were no increases in background levels. However, in practice, the pressure reduction cannot be accomplished without an incommensurate increase in background levels because of the high flow of the primary plasma gas species through the throttling conductance and the QMS. Figure 2 plots the trade-off between the pressure reduction factor and the minimum detectable partial pressure in the plasma chamber for a QMS with the specifications given above. Also shown is the decrease in system dynamic range as a function of the background partial pressure.

A practical example: The application of partial pressure analysis to hydrogen plasmas illustrates the possible severity in loss of dynamic range when sampling reactive gas systems. When molecular H_2 is introduced into the typical electron-bombardment ionizer, of the order of 0.1-0.5% of the H_2 will be converted to the residual gases H_2O, CO, and CH_4 because of the formation of atomic hydrogen on the ionizer filaments, and the subsequent reaction of H_2 with the electrode surfaces.

This problem has been studied in detail by the author,[48] and Fig. 3 shows the results of background measurements as a function of ambient H_2 pressure for a commonly used QMS ionizer. Without special conditioning of this ion source, the background levels of H_2O, CO, and CH_4 are such that the dynamic range is reduced to 10^3 for H_2O, and 10^4 for CO/CH_4 for measurements of partial pressures at 18, 28, and 16 amu in H_2 plasmas. Pressure reduction would exacerbate this situation because the necessary throttling valve would preferentially conduct the lower mass H_2 in comparison to the higher mass residual gases, thus decreasing the effective signal-to-background level for these gases. This enhanced background problem is not limited to the case where the primary plasma species is a chemically active gas. The effect also has been documented in the sampling of inert gas (argon) plasmas.[49]

Standard UHV practice in the design and handling of the QMS components can reduce the background problem. In addition, there are several alternative schemes to minimize the problem: (1) the ionizer can be treated to minimize the offending gas-surface interactions. The interaction of H_2 with standard QMS ionizers was traced to the interaction of atomic hydrogen with iron oxides on the electrode surfaces. Figure 3 shows that a factor of five improvement in background generation was obtained when the electrode surfaces were Cr-plated and then conditioned by exposure to a H_2 glow discharge. (2) For background problems that can be traced to the presence of a hot filament in the ionizer, a low temperature emitter can be substituted, or an externally generated electron beam can be used as the ionization source allowing the removal of the filament from the QMS chamber.[50] A disadvantage of the common low temperature emitters (Th/W and LaB_6) is that their emission characteristics are unstable in active gases; and the primary disadvantage of the compound ionizer with a separated electron source is the considerable

complication in experimental design over the relatively simple arrangement shown schematically in Fig. 1b. (3) A last resort solution to an intolerable background problem in a partial pressure measurement is to revert to a flux-type measurement. By introducing an effusion aperture on the line-of-sight between the plasma chamber and QMS, and chopping the effusing beam with a mechanical chopper, the signal-to-background ratio of the QMS can be improved significantly using the well-known phase-sensitive detection technique[51] on the QMS output.

B. System Response

The experimental scheme for partial pressure analysis diagrammed in Fig. 1b, which shows the QMS separated from the plasma chamber by a differentially pumped connection tube, results in the dynamic response of a pressure change in the QMS chamber, $\Delta P_Q(t)$ being different from the pressure change in the plasma device, $\Delta P_D(t)$. Figure 4 shows the experimental scheme of Fig. 1b redrawn with the important kinetic factors in the system identified: the plasma chamber with volume V_D, is pumped with speed S_P, and may have one or more gas input sources Q_i; the QMS chamber with volume V_Q, is pumped with a separate pump with speed S_Q, and is connected to V_D through a tube with conductance C_T. The quantities S_P, S_Q, and C_T can be a function of the gas temperature T, and the pressure P, mass M, and viscosity of the gas depending on the flow regime and type of vacuum pump.

For proper calibration of a partial pressure analyzer, the relationship between $\Delta P_D(t)$ and $\Delta P_Q(t)$ needs to be evaluated. First, we consider the simplified case of nonreacting gases, i.e., gas-surface interactions within the connection tube and the QMS are ignored, so that only the kinetic relationship between $\Delta P_D(t)$ and $\Delta P_Q(t)$ need be considered. For static

measurements ΔP_D and ΔP_Q are related simply by a multiplicative throttling factor, ρ, which is the same number as the pressure reduction ratio used as a parameter in Fig. 2:

$$\Delta P_D = \rho \ (\Delta P_Q) \tag{1}$$

ρ is inversely proportional to the net series conductance (C_T) of the connection tube and any intervening apertures or flow restrictions, and proportional to the differential pumping speed, S_Q.

For dynamic measurements, a differential equation describing the time-response of the system must be solved[52]:

$$P_D(t) = \left(1 + \frac{S_Q}{C_T}\right) P_Q(t) + \frac{V_Q}{C_T} \frac{d[P_Q(t)]}{dt} - \frac{S_Q P_B}{C_T} \tag{2}$$

where the last term represents an explicit background pressure P_B correction in the QMS. Equation (2) usually has to be solved numerically. Solutions of interest to partial pressure analysis have been given by Poschenrieder et al.[1] and Banno et al.[52]

The KFA group[34] has considered the additional complication to system time response that results when the gas molecule of interest has a non-ignorable sticking time on the surface of the connection tube in comparison to transit times. They found for the case of the transmission of H_2O through a stainless steel connection tube that equilibration times between a step

function increase in P_D and a static value of P_Q were greater than an hour for T = 300 K and this declined to ~ 10 minutes for T = 430 K. Thus, absolute dynamic measurements for P_{H_2O} in such a system would be difficult on time scales shorter than the equilibration time because of the variable dependence of the equilibration rates on the surface conditions of the connection tube.

C. Electromagnetic Interferences

Interfacing a QMS to a plasma device requires a significant degree of effort to shield the instrumentation properly from electromagnetic noise sources generated by the plasma, or by the power sources which support the plasma. Generally, two types of interference problems are encountered: noise generated in the QMS ion detector by particles emitted from the plasma; and electromagnetic pickup in the QMS ion detector or detector electronics from EMI or RFI generated by plasma power supplies or magnetic systems.

For partial pressure analysis, it is not advisable to position the QMS components, particularly the ion detector, on a line-of-sight view of the plasma. Ultraviolet and soft-X-ray photons, and high energy neutrals which are emitted from the plasma can ionize residual gas in the vicinity of the ion detector or generate secondary electrons on the ion-to-electron conversion electrode. Either effect will cause a large increase in background signal that is mass independent. The practice of locating the ion detector off-axis, which is now standard in most commercial QMS instruments, will eliminate this problem in most process plasma applications. However, for a high brightness source such as fusion device, more drastic measures have to be taken. For example, the author observed that output signal of a QMS installed with a line-of-sight view of the plasma in the PLT tokamak was completely dominated by VUV emission during the duration of the plasma discharge. Apparently, the

reflectivity of VUV on the quadrupole mass filter rods was sufficient to cause the problem, even though the ion detector was located off the QMS axis. This particular problem was solved by adding an additional right-angle bend in the connection tube. An alternative solution would have been the use of a photon-absorbing coating within the connection tube and QMS chamber. This technique is often used for decreasing photon-induced background levels in charge-exchange neutral analyzers on fusion devices.[53]

To minimize problems with EMI/RFI, it is important to obey standard shielding and grounding practice for use of low level instrumentation in noisy environments, such as the single point grounding for the QMS and double electrostatic shielding of the ion detector-preamplier system. Here again, the problems are more severe with the interface of a QMS to a high power fusion plasma device. The most troublesome interference problem is concerned with the coupling of magnetic fields from the plasma device magnetic system. The mechanical problems associated with the use of the large, compound, ferromagnetic shields[54,55] which are required for high field areas, usually dictates the positioning of a QMS in a location where the stray or fringing fields are $\leq$ 1 kG. More modest shielding is still required to attenuate the local fields at the QMS to a level which does not affect operation. Typically, the field has to be less than 10 gauss at the ion source, quadrupole mass filter, and the electron multiplier (for the radial component of the field). However, most electron multipliers are more tolerant ($B < 100$ gauss) of axial magnetic fields.

D. Calibration Procedures

A common occurrence in the vacuum science literature is the presentation of partial pressure data in an uncalibrated format, i.e., the signal output of

a QMS is displayed as raw data (the electron multiplier or Faraday cup output current), or in arbitrary intensity units. Without, as a minimum, a calibration that encompasses the entire system as described in Sec. III B, the interpretation of QMS data can be very misleading. Reconsider the simplified vacuum system schematic shown in Fig. 4. A system calibration determines the functional relationship between P_D and P_Q for one or more gases over a pressure range of interest. In addition, the relationship between P_Q and the measured output current of the QMS must be determined.

In certain situations the calibration procedure can be quite complicated. For example, if the pumping speed of the plasma chamber or QMS chamber pump is not a well-defined function of gas species and pressure; or if the calibration must span a pressure regime which causes the conductance of the connection tube to overlap more than one kinetic flow regime; or if the gas species of interest has significant physical or chemical reactions with the vessel wall materials; then the calibration procedure is not straight forward.

There are several examples in the literature of experimental arrangements for partial pressure analysis that are difficult to calibrate, unstable with respect to maintaining a calibration, or capable of being calibrated only over a limited range of parameters. The use of an ion pump for the differential pump on the QMS chamber[47] is a bad choice because of the large variation of pumping speed with gas species, and the tendency of the pumping speed to decrease with time and total throughput. The closed-cycle cryopumps operating at 20 K have the same disadvantages and also should be avoided for this application.[56]

The optimum type of pump for this application is either a turbo-pump or a diffusion pump, because the pumping speed for both types of pumps can be

characterized as function of the mass of the gas and the gas pressure, and for all practical purposes, the speed does not vary with time or total throughput. This property greatly simplifies the calibration of a QMS system over a wide range in mass and pressure.

A second common pitfall which complicates calibration procedures is the use of needle-type throttling valves in the connection tube to accomplish the pressure reduction. The mechanical hysteresis that is inherent in the design of most valves of this type makes repeatability difficult. A better choice is the use of a series of well-defined apertures. There exists no optimal calibration procedure for the partial pressure analysis scheme shown in Fig. 4, nor have the standards organizations addressed this experimental situation. However, a number of relatively simple approaches can be taken which yield satisfactory results. If the process to be monitored is a static process or otherwise slowly varying with respect to the characteristic vacuum time constants, then a static calibration will suffice. In this case a predetermined gas mixture can be introduced to V_D, the amount of introduced gas is quantified either by means of a calibrated leak (Q_i) or by comparison with a calibrated total pressure gauge on the volume V_D, and finally the response of the QMS as a function of mass is recorded. For dynamic measurements, single component gases must be introduced individually, and the system differential equation, Eq. (2), can be obtained numerically from the measured pumping transients (V_D/S_D, V_Q/S_Q) and the form of $dP_Q(t)/dt$.

As a specific example, the calibration procedure that we have adopted for the partial pressure analysis on the Princeton tokamaks can be described. Since the details of any calibration procedure are dependent on the specific hardware involved, it is instructive to describe first the design of our partial pressure analyzers. Figure 5 shows the most recent version,

which is one of two instruments designed for use on the Tokamak Fusion Test Reactor (TFTR).[57,58] Two stages of differential pumping have been included with different-sized apertures that can be placed in front of each stage. A pressure reduction ratio of 70, 300, or 2 x 10^4, at m = 28 amu is accomplished by the system depending on whether the first, second, or both apertures are in place. For partial pressure measurements of tokamak-type discharges in TFTR, no pressure reduction is required; for sampling the hydrogen glow discharges that are applied to condition the vacuum vessel, a pressure reduction of 30 - 300 is used, since the glow discharge pressures are in the range 5 - 25 m Torr. (The highest pressure reduction ratio, 1000, was chosen for use when TFTR begins tritium operation, to minimize the exposure of the QMS electron multiplier to tritium).[59] Located in front of each pumping stage, and on the QMS chamber, are a gas inlet valve for introducing calibration gases, and a capacitance manometer and ion gauge which serve as secondary vacuum standards for the calibration.[55] The calibration of the capacitance manometers is periodically checked against a spinning rotor gauge[60] in a separate vacuum system.

The calibration of the TFTR partial pressure analyzer is performed in two parts. The first part involves measurement of the static pressure reduction ratios, and the dynamic response to transient pressure rises in the main plasma vessel for single gases of varying mass (m = 2 - 40 amu). This part of the calibration need be performed only once because it involves fixed conductances and pumping speeds. The result for static measurements is the pressure reduction ratio, ρ, (Eq. 1) which combines the effects of the connection tube and aperture conductances and the differential pumping speed. The second part of the calibration yields the relationship between an absolute partial pressure and the QMS output current for a particular gas

introduced into the QMS chamber. This calibration is done weekly for the TFTR instruments because they are generally in-service full-time, and the QMS sensitivity is dependent on the ionization and extraction efficiency of the ionizer, the transmission of the quadrupole mass filter, and the electron-multiplier gain, all of which can change with time or exposure history. Figure 6 shows an example of this part of the calibration. An equimolar gas mixture containing six components (H_2, He, CH_4, Ne, and Ar) is introduced into the unpumped QMS chamber through a leak valve. The calibration curves of Fig. 6 are generated by stepwise increasing the total pressure over the range 2 x 10^{-5}-2 x 10^{-4} Torr as measured on capacitance manometers. To increase the accuracy of the total pressure measurement in the low end of this range 2-5 x 10^{-5} Torr, an ion gauge is used which is scaled with a gauge factor for the gas mixture determined by comparison with the capacitance manometer over the range 5 x 10^{-5}-1 x 10^{-3} Torr. The partial pressure of each component gas is assumed to be equal to one-sixth of the total pressure. This assumption was checked by comparing calibration curves obtained with the gas mixture to curves obtained with a single component gas. The possibility of mass separation of the gas mixture during the inlet process was checked by comparing mass spectra of the gas mixture obtained from a fully pressurized gas bottle to that obtained from the same source when it was nearly exhausted.

Since the calibration is performed under static conditions, best results are obtained when the QMS and QMS chamber are thoroughly outgassed. Nevertheless, outgassing corrections are minimized by pumping the system to base pressure levels (~ 1 x 10^{-9} Torr) between each data point.

The accuracy of this calibration procedure is essentially limited by the accuracy of the secondary vacuum standards. The relative error in the calibration based on the systematic errors in the partial pressure measurement

are estimated to be ± 5%. The absolute error is estimated to be ± 10% over the pressure range of 10^{-5}-10^{-4} Torr. The accuracy of calibration could be improved by calibrating with individual gases against a spinning rotor gauge. For our purposes we have sacrificed some accuracy for the expediency of obtaining a relatively rapid calibration over the mass range of primary interest (2 - 40 amu). We attribute the primary cause of long-term drift in the calibration of a QMS to aging of the electron multiplier. Following the installation of a new (CuBe) multiplier the gain will fall by a factor of 5 after a short period of operation, but then will stabilize and exhibit a much slower decrease in gain with time (see Fig. 7).

The data of Fig. 6 were obtained with the Extranuclear 041-1 ionizer, which seems to be reasonably linear up to a pressure of 10^{-4} Torr. Alternatives to the use of pressure reduction methods for sampling pressures higher than 10^{-4} Torr are the use of optimized QMS components for high pressure operation with miniaturized electrodes, or the incorporation of an explicit nonlinear calibration at high pressures in the analysis of data.[46]

E. Glow Discharge Measurements

It is appropriate to conclude this review with an example application of partial pressure analysis. As previously stated, the technique is useful for monitoring the reaction products of plasmas with the plasma vessel wall or with large area samples in contact with the plasma. Partial pressure analysis has been the primary technique for monitoring and optimizing the hydrogen discharge cleaning techniques[3] which have been developed over the last decade to condition the large vacuum vessels of contemporary fusion devices. Figure 8 shows a recent example of this type of experiment: the time evolution of the primary impurity gases produced during the initial glow discharge cleaning

of the 85 m^3 stainless steel vacuum vessel of the TFTR device.[37] The measurements were made with the instrument shown in Fig. 5. Since this instrument was absolutely calibrated, the data of Fig. 8 were used to calculate the total amount of carbon (0.6 g) and oxygen (1.2 g) that was removed from the vessel as a result of the discharge cleaning.

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Figure Captions

Figure 1 (a) Schematic diagram of the typical apparatus required for plasma flux analysis. A sampling aperture (A) separates the plasma device from a vacuum enclosure housing energy and mass spectrometers. The aperture may include ion optical elements or be followed by additional ion lenses (L) for ion extraction, or by an ionizer (I) for ionization of neutral particles effusing through the aperture. The ion detector (shown here as an electron multiplier) should be positioned off the axis of the aperture to prevent background problems caused by high energy neutrals (n^*) and photons (ν) emitted by the plasma.
(b) Schematic diagram of the typical apparatus required for partial pressure analysis of plasma devices. The mass spectrometer measures the neutral density (n^o) in the analyzer chamber which can have a high conductance (V_1) or low conductance (V_2) connection to the plasma device. The indicated mass spectrometer components: a hot-filament (F) electron-bombardment ionizer (I), ion lens (L), quadrupole mass filter, and deflection electrode (D) for an off-axis (electron multiplier) ion detector are typical for commercially available quadrupole mass spectrometers.

Figure 2 Trade-off of the pressure reduction ratio (the ratio of static pressure in the plasma chamber to QMS chamber) versus the minimum detectable partial pressure in the plasma chamber. The QMS is assumed to have a minimum detectable partial pressure of 10^{-11} Torr and a dynamic range of 10^7. Also shown (with the dotted lines) is the loss in dynamic range (R) with increasing background pressure (P_B) in the QMS.

Figure 3 Change in partial pressure of the dominant residual gases produced in the Extranuclear 041 ion source as a function of the H_2 pressure for the standard ion source (open points), and after Cr plating and H_2 glow discharge conditioning (filled points). (From Ref. 48).

Figure 4 Simplified vacuum schematic for the partial pressure analyzer shown in Fig. 1b.

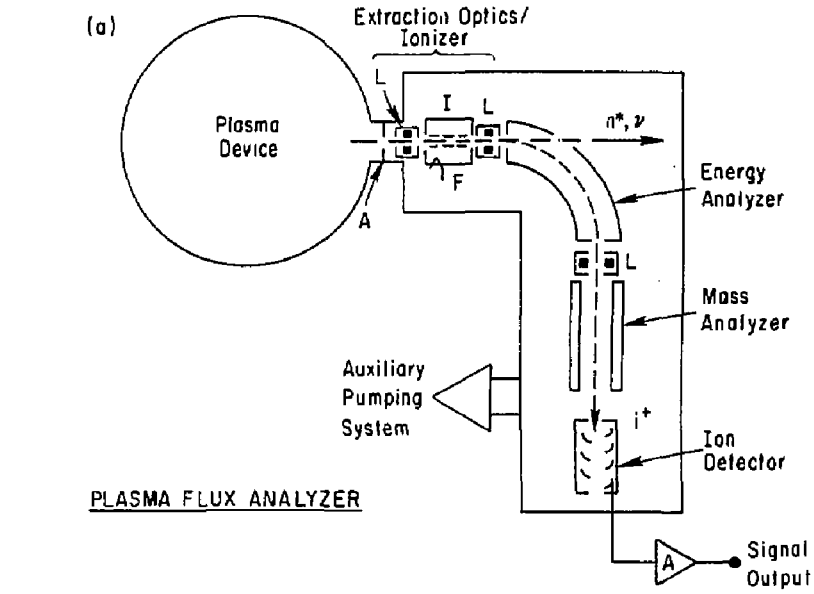
Figure 5 Schematic diagram of one of the two partial pressure analyzers on the Tokamak Fusion Test Reactor (TFTR). The analyzers are located on the torus vacuum pumping ducts shown in the inset figure.

Figure 6 Typical calibration curves for a QMS obtained by the procedure described in the text. These data were obtained with an Extranuclear 041-1 ionizer operated at 1 mA emission, an Extranuclear 324-9 quadrupole mass filter, and a 14 stage CuBe electron multiplier operated at 2.0 kV.

Figure 7 Observed change in QMS sensitivity for N_2 as a function of operating time in a partial pressure analyzer on the PDX tokamak. The configuration of this QMS was similar to the description given in Fig. 6.

Figure 8 Observed time dependence of the partial pressures of the predominant impurity gases produced during the first glow discharge cleaning of the TFTR vacuum vessel (From Ref. 37).

#83X0738



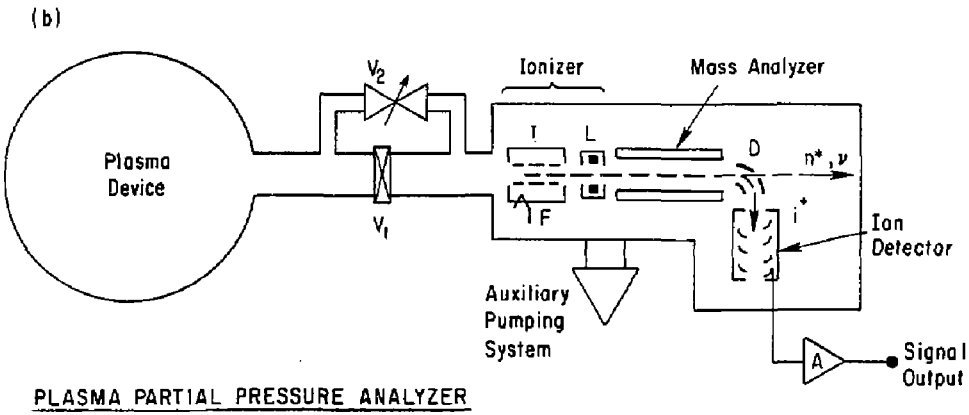
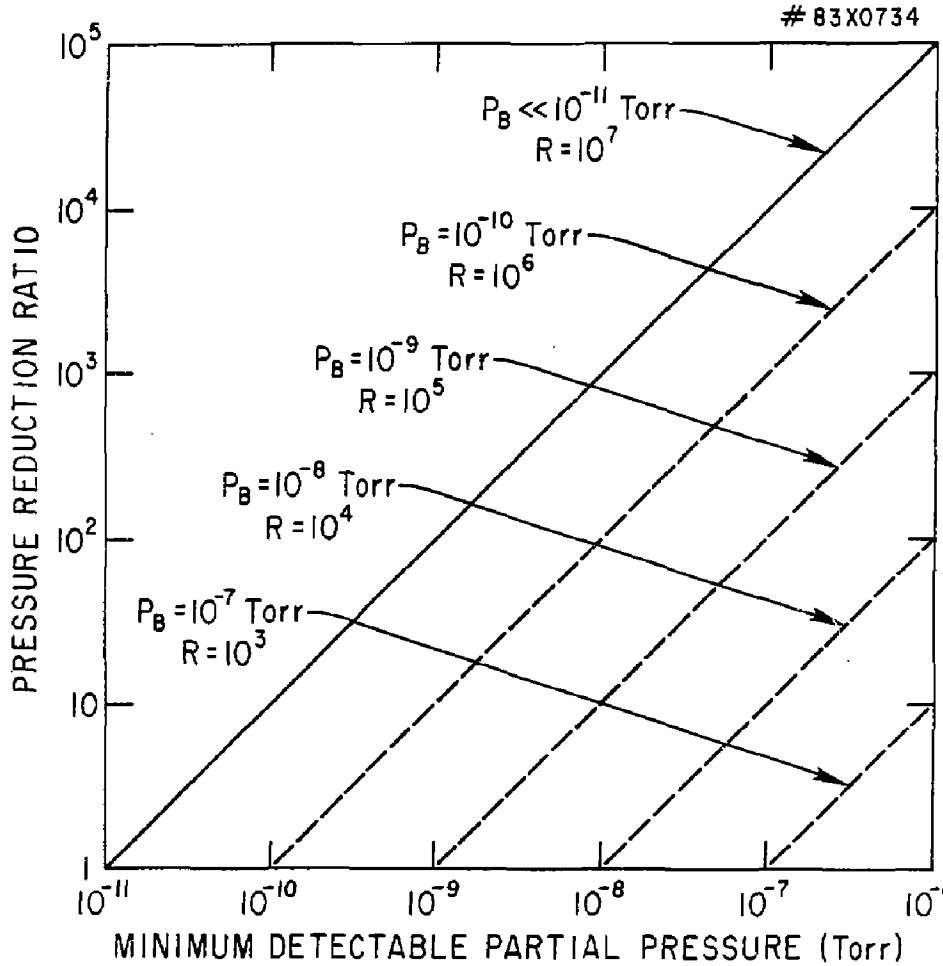


Fig. 1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PRESSURE REDUCTION RATIO
MINIMUM DETECTABLE PARTIAL PRESSURE (Torr)
$P_B \ll 10^{-11}$ Torr
R = 10^7
$P_B = 10^{-10}$ Torr
R = 10^6
$P_B = 10^{-9}$ Torr
R = 10^5
$P_B = 10^{-8}$ Torr
R = 10^4
$P_B = 10^{-7}$ Torr
R = 10^3
10^5
10^4
10^3
10^2
10
1
10^{-11}
10^{-10}
10^{-9}
10^{-8}
10^{-7}
10^{-6}

Fig. 2

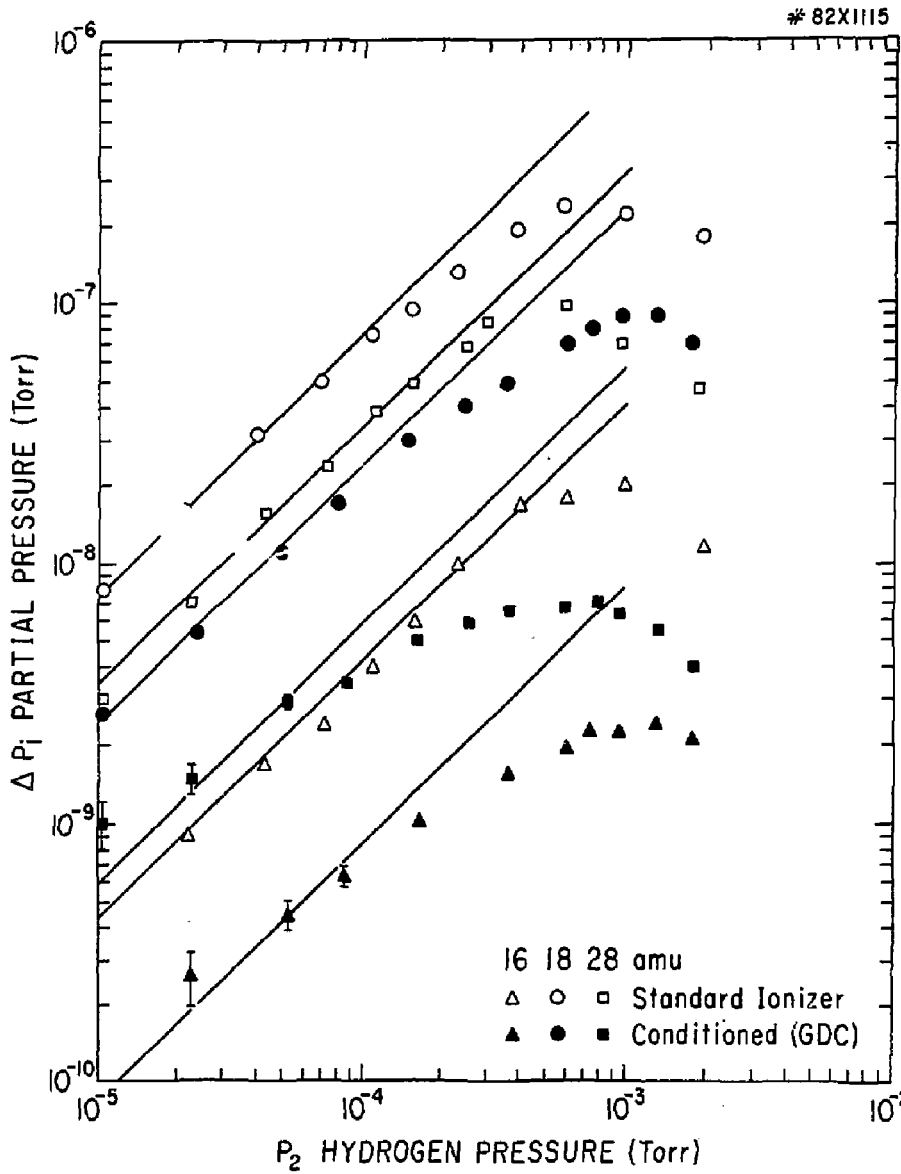

#82X1115
10^{-6}
10^{-7}
10^{-8}
10^{-9}
10^{-10}
ΔP_i PARTIAL PRESSURE (Torr)
10^{-5}
10^{-4}
10^{-3}
10^{-2}
P_2 HYDROGEN PRESSURE (Torr)
16 18 28 amu
Standard Ionizer
Conditioned (GDC)

Fig. 3

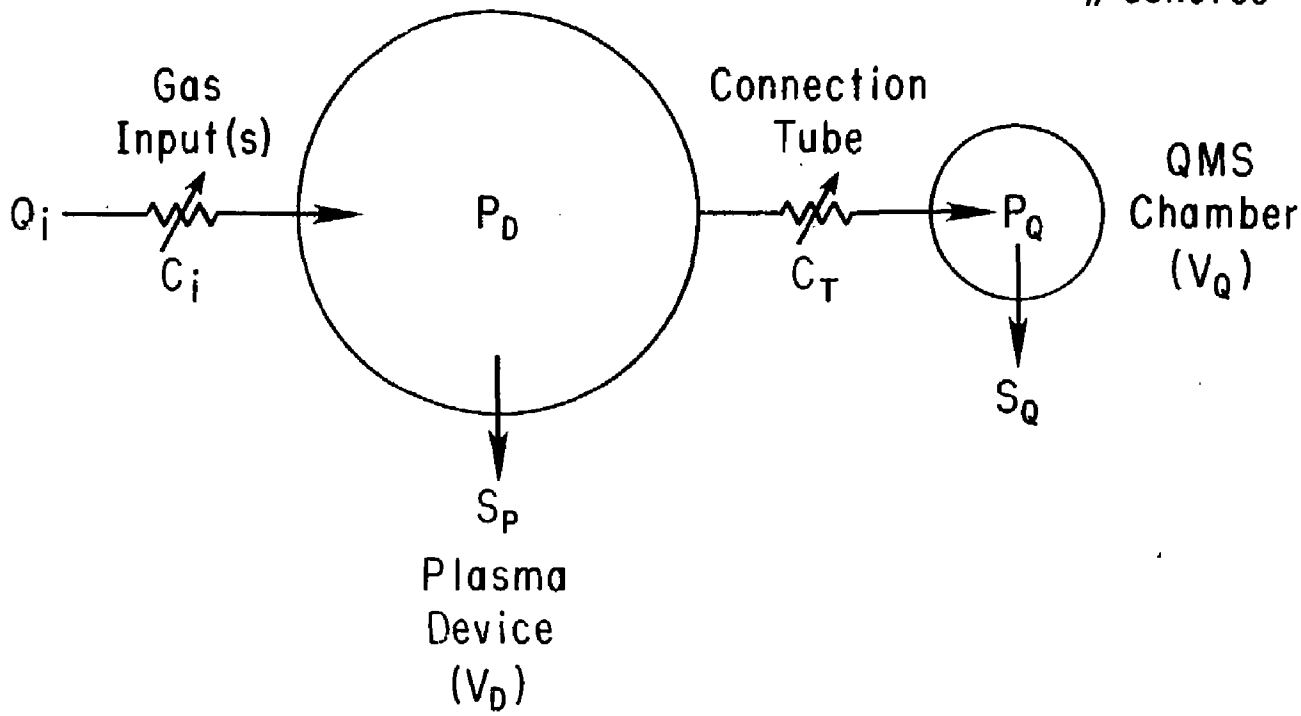
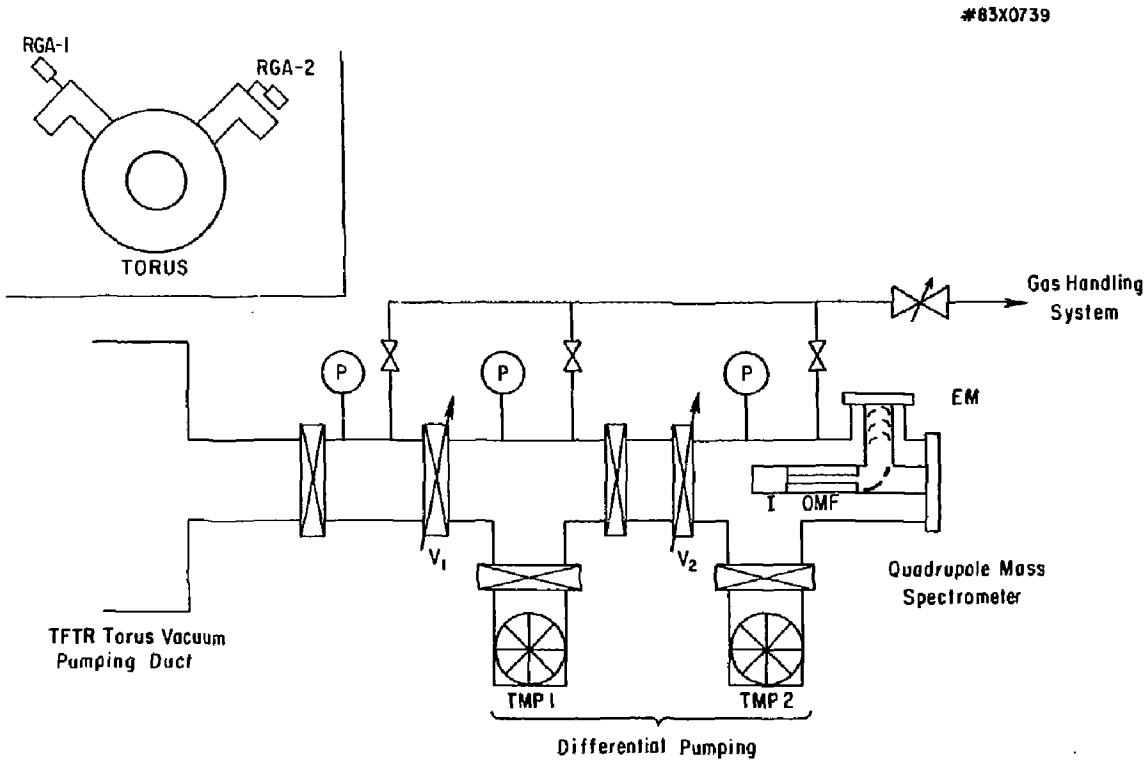

#83X0733
Gas
Input(s)
Q_i
C_i
P_D
S_P
Plasma
Device
(V_D)
Connection
Tube
C_T
P_Q
S_Q
QMS
Chamber
(V_Q)

Fig. 4



TFTR RESIDUAL GAS ANALYZER (RGA)

Fig. 5

#83X0755

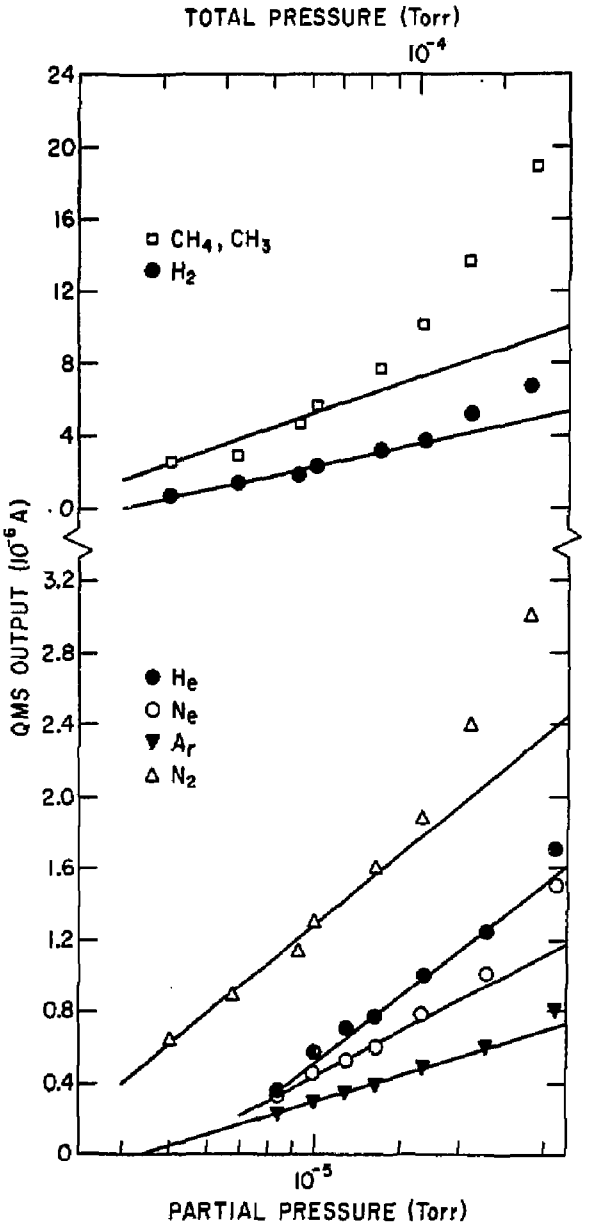


Fig. 6

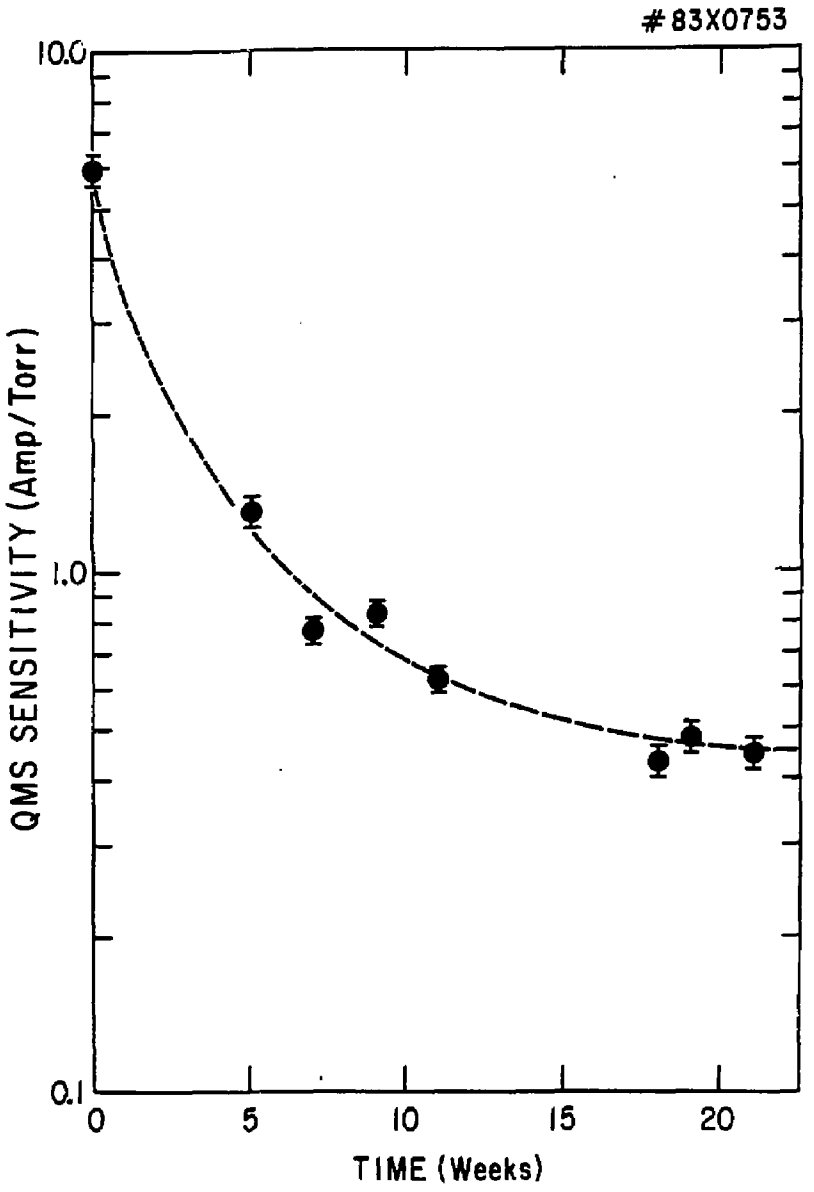

#83X0753
10.0
1.0
0.1
QMS SENSITIVITY (Amp/Torr)
0
5
10
15
20
TIME (Weeks)

Fig. 7

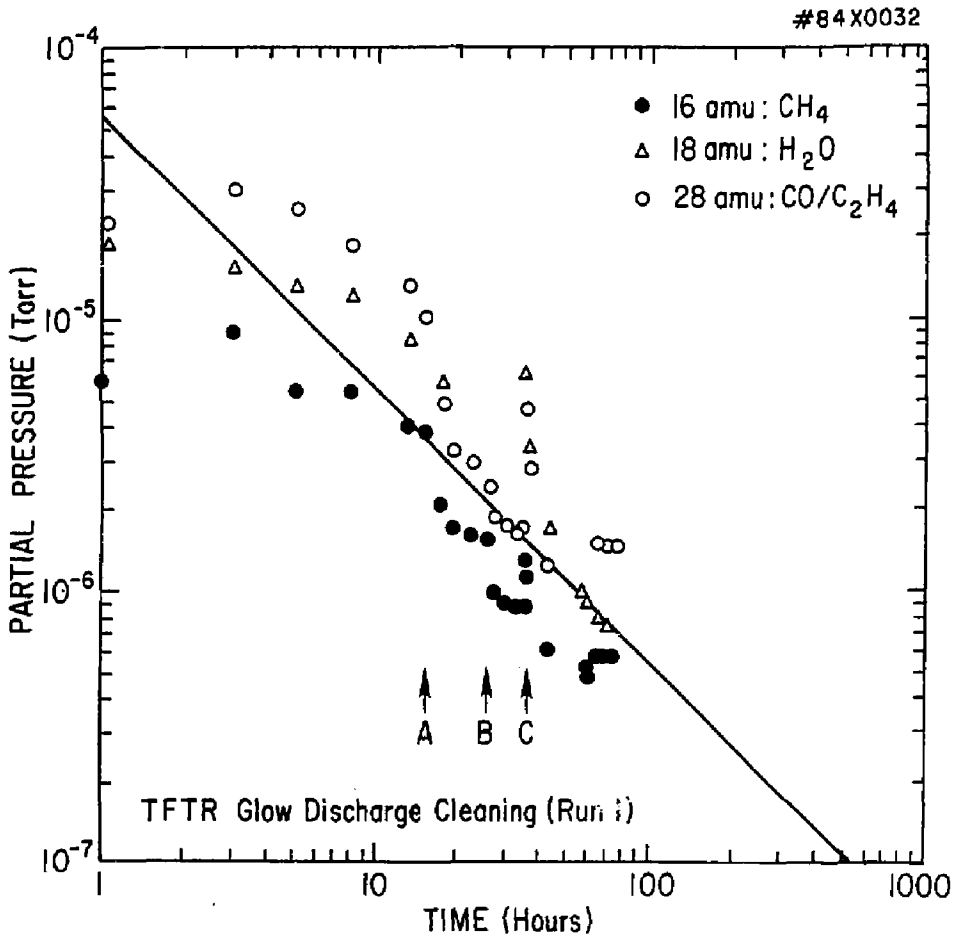

#84X0032
16 amu : CH_4
18 amu : H_2O
28 amu : CO/C_2H_4
A
B
C
TFTR Glow Discharge Cleaning (Run 1)
PARTIAL PRESSURE (Torr)
TIME (Hours)
10^{-4}
10^{-5}
10^{-6}
10^{-7}
1
10
100
1000

Fig. 8

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