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PLASMA PHYSICS LABORATORY

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OBSERVATIONS OF CHANGES IN RESIDUAL GAS AND
SURFACE COMPOSITION WITH DISCHARGE CLEANING IN PLT

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ABSTRACT

Hydrogen discharge cleaning of the PLT vacuum vessel has been studied by mass spectroscopy of desorbed gases and surface analysis of exposed samples. Several modes of vessel conditioning have been studied to date: (1) a high power discharge cleaning (PDC) mode, with a peak power density to the vessel wall $P_s \sim 0.6 \text{ w/cm}^2$ and a peak electron temperature $T_e \sim 100 \text{ eV}$; (2) low power (Taylor-type) discharge cleaning (TDC) with $P_s \sim 0.05 \text{ w/cm}^2$ and $T_e \leq 5 \text{ eV}$. The predominant residual gases produced during PDC are CH_4 ($1-5 \times 10^{-6}$ torr) and CO ($1-10 \times 10^{-7}$ torr), whereas TDC produced primarily H_2O ($1-2 \times 10^{-6}$ torr) and CH_4 ($1-10 \times 10^{-7}$ torr). The transient behavior of the residual gases is not simply related to pumping time constants, and provides evidence for the role of surface diffusion and surface chemical reactions on the formation of these gases. In situ surface analysis of hydrocarbon-covered stainless steel has shown significant decreases in carbon coverage occurring after 10^3 - 10^4 pulses of either cleaning mode. Observed changes in oxygen coverage are more difficult to interpret because of the presence of the nascent oxide layer on the stainless steel substrates.

I. INTRODUCTION

A number of conditioning techniques have been applied to the Princeton Large Torus (PLT) tokamak since the device began operation in December, 1975. Structural aspects of the PLT device precluded the possibility of in situ bakeout of the vacuum vessel. Therefore, other cleaning techniques had to be relied upon for all vessel conditioning. The usual conditioning procedure involves the application of one of several modes of hydrogen discharge cleaning.^{1,2} Recently titanium gettering^{3,4} of the vacuum vessel has supplemented the discharge cleaning. Brief descriptions of the vessel conditioning procedures and the resulting plasma conditions have been reported previously.⁴⁻⁶ This paper describes in more detail measurements of the residual gas behavior and in situ surface analysis of the vessel conditioning.

Routine in situ vacuum vessel conditioning must be employed in tokamak devices to bring about two beneficial effects:

- (1) The removal of loosely bound adsorbates (mainly residual gases). In an unconditioned vessel low-Z impurities desorbed from the vacuum vessel can seriously affect plasma behavior.⁷
- (2) The control of the re-emission of the fill gas (usually H_2 or D_2) during the duration of the discharge. The minimization of recycling allows much more effective control of the evolution of plasma density through programmed gas injection.⁸ (Careful programming of

the gas injection during the initial phase of the discharge can also minimize impurity influx.⁴⁾

II. APPARATUS

The construction details of the PLT vacuum vessel and the design and operation of the torus vacuum system have been previously described in this journal.^{9,10} Four turbomolecular pumps, with a net H_2 pumping speed of 3000 l/s, evacuate the 6,400 liter toroidal vacuum vessel to -5×10^{-7} torr prior to any vessel conditioning. After discharge conditioning the best base pressures are -1×10^{-8} torr. Additional pumping is presently provided by three retractable titanium sublimation sources (Varian "Ti-balls"), with nominal evaporation rates of 0.2-0.5 gm/hr per source. In the first experiments there was one set of four tungsten limiters installed to protect the vacuum vessel from plasma bombardment. Presently, there are three sets of limiters: (1) a movable pair of stainless steel rail limiters; (2) a poloidal ring limiter, constructed of Mo with a stainless steel jacket, located at the same toroidal position as (1); and (3) a movable pair of water-cooled ATJ-S graphite limiters located 40° from the stainless steel limiters. The movable limiters can be retracted to a radius of 46 cm, whereas the fixed ring limiter extends from 45-50 cm. (Structural components of the vacuum vessel extend from 49 cm to 51 cm.)

Two diagnostics are extensively used to monitor vessel conditioning. A quadrupole residual gas analyzer is interfaced to a pumping duct through a conductance with a vacuum time constant

of 30 ms for H_2 . The characteristic pumping time of H_2 for the torus is $(V_{TORUS}/S_{NET}) = 2.3$ s. The partial pressures of hydrogenic gases as measured by the RGA were found to be linear with absolute hydrogenic pressures up to 2×10^{-4} torr. However, for several of the heavier molecular weight gases of interest (CH_4 and CO) the effects of ion-molecular reactions in the ionizer become significant above hydrogen pressures of 1×10^{-4} torr. For the pressure range, $1 \times 10^{-7} < p < 2 \times 10^{-4}$ torr, the QMS was absolutely calibrated with a capacitance manometer for the following gases: H_2 , D_2 , He, N_2 , O_2 , H_2O , CH_4 , Ne, CO, Ar, and CO_2 .

A surface analysis probe (SAS) is located on the outer mid-plane of the torus $\sim 170^\circ$ from the stainless steel limiters. This diagnostic is capable of exposing stainless steel substrates or evaporated films of clean metals to the edge plasma region between the limiter inner radius and vacuum vessel outer radius. After exposure the sample can be retracted to an attached ultra-high vacuum system for Auger analysis. Previously published are details of the design and operation of the diagnostic, and the results of studies of high temperature plasma discharges.¹¹ In addition to this capability of in situ surface analysis, a number of sample exposure probes have been placed around the torus to monitor long-term changes in the vacuum vessel surface conditions. Samples are removed from these probes after various exposure times (ranging from less than one day to six months), and analyzed off-site by a variety of surface analysis techniques.¹²

III. CONDITIONING HISTORY

Two modes of discharge cleaning have been used to condition the PLT vacuum vessel. A comparison of the discharge parameters of the two modes is given in Table I. The first mode of discharge cleaning (PDC) in use on PLT was a standard hydrogen tokamak discharge run at lower discharge currents and higher repetition rates than the typical high-power PLT discharge. Such conditions resulted in a discharge with a central electron temperature and density of 100 eV and 10^{13} cm^{-3} respectively. After major vacuum openings, it was generally necessary to discharge clean in this mode for 5-10 days (approximately 10^4 discharges) before relatively disruption-free high power discharges could be maintained for several hundred milliseconds. This mode of discharge cleaning was used on PLT from December 1975 through February 1977, and in general it had the effect of reducing the carbon impurity content to an unimportant level, but left the oxygen impurity concentration near 5%. Z_{eff} values were not reduced below 4-6 for H_2 discharges.⁶

The second mode of discharge cleaning (TDC) is similar to a methodology described by Taylor² and has been in use on PLT since February 1977. This mode also involves producing a discharge in the usual manner; however, the hydrogen fill pressure ($\sim 1 \times 10^{-4}$ torr) was adjusted to yield a partially-ionized plasma with correspondingly low values of the plasma current (2-4 kA), electron density ($1-5 \times 10^{11} \text{ cm}^{-3}$), and electron temperature (2-5 eV). The low power in the resulting discharge enabled the

repetition rate to be increased without significantly increasing the vacuum vessel or ohmic-heating coil temperatures. Taylor demonstrated in studies on tokamaks at MIT¹³ and UCLA² the utility of such low temperature discharges for removal of oxygen from the vacuum vessel by maximizing the production of water vapor. Generally, the effect of extended use (10^4 pulses) of TDC on PLT⁵ was a reduction of the oxygen impurity level by a factor of 3-5 to about 1%, and a reduction in Z_{eff} to values in the range of 1.5-2.5.

PLT discharges with the lowest oxygen concentrations ($n_O = 10^{11} \text{ cm}^{-3}$), and Z_{eff} values equal to or near unity have been obtained when TDC conditioning was followed by titanium gettering of the vacuum vessel.⁴ Preliminary gettering experiments with one sublimation source were done in October and December 1977. This was followed by extensive gettering with up to three sources during the most recent operating period, May 1978 to present (September 1978). The gettering parameters for the sublimation sources are given in Table I.

IV. RGA OBSERVATIONS

Observations of the changes in residual gas composition during and following the various vessel conditioning procedures are summarized in this section. The significant difference between the two modes of hydrogen cleaning (PDC and TDC) with regard to production of water vapor is shown in Figs. 1 and 2. Figure 1 shows typical RGA spectra (a) of the background; and (b) during the application of PDC and TDC. Approximately equal

CH_4 (16 amu) peak heights are noted for PDC and TDC, whereas only with TDC is there a noticeable increase in the H_2O (18 amu) peak. For the case of PDC, increases in the CO (28 amu) peak (to $\sim 10^{-6}$ torr) are observed; however the transient behavior of this peak following a PDC pulse (see Fig. 2d) shows that CO is rapidly pumped by the wall in time short ($\tau < 0.35$ s) compared to the vacuum pump-out time ($\tau \approx 5.0$ s for 28 amu).¹⁰ The effective sticking coefficient of the CH_4 molecules onto the wall is evidently small and the characteristic decay time for CH_4 (typically 9-10 s following either PDC or TDC pulses; see Fig. 2c), allows significant removal of carbon from the vessel via the vacuum pumps. Methane production was optimized in PDC by operating at low toroidal fields (~ 1 kG). Little difference was seen in TDC for either CH_4 or H_2O production for $2 \text{ kG} < B_T < 8 \text{ kG}$.

The temporal behavior of H_2O peak following each type of discharge is shown for TDC in Fig. 2a and PDC in Fig. 2b. The H_2O pressure transient after the TDC pulse decays with a time constant (~ 10 s) which is sufficiently long compared to the vacuum system time constant ($\tau = 4.6$ s for 18 amu),¹⁰ for the water (and therefore oxygen) to be pumped out. The transient behavior also shows that the H_2O production occurs after a TDC pulse, and that the wall has become either activated with respect to H_2O production or passivated with respect to H_2O adsorption. Injected pulses of O_2 show decay time constants < 0.3 s, and we would expect similarly short decay times for H_2O if the production were

confined to the pulse period and the wall not passivated. The transient behavior of H_2O following a PDC pulse (Fig. 2b) usually shows a large decrease relative to the ambient level, probably indicating that H_2O is dissociated or ionized by the dense PDC plasma, and there is no net H_2O production following recombination.

The time-averaged partial pressure of H_2O during an extended series of PDC pulses is shown in Fig. 3. After 6000 cleaning pulses 0.88 torr-liters of O_2 was injected into the torus to quantify the observed relation between O_2 pressure and oxygen coverage (following a method developed by Taylor²). The initial rapid drop in H_2O pressure is seen to correspond to a change in coverage of ~ 0.5 monolayer averaged over the torus area. Note that not all of the injected O_2 was removed by subsequent discharge cleaning, indicating that not all areas of the vacuum vessel accessible to the gaseous O_2 are effectively bombarded by the discharge.

Titanium gettering of the vacuum vessel has a significant effect both on the background residual gas spectra and on the production rate of H_2O during TDC. An increase in the effective pumping speed for active gases is measurable after any substantial gettering (>0.1 gm). Figure 1a shows the background residual gas spectrum before and after 45 minutes of evaporation from one source. The base pressure has been lowered by a factor of two, primarily due to decreases in the H_2 , H_2O and CO partial pressures. After gettering, decay times as short as 0.24 s have been measured

for a transient pulse of D_2 typical of the fill pressure ($\sim 1 \times 10^{-4}$ torr). This indicates an order of magnitude increase in the D_2 pumping speed (to 28,750 l/s) compared to the observed decay time (2.45 s) and resulting pumping speed (2800 l/s) characteristic of only the net turbomolecular pump speed for D_2 . An accurate estimate of the deposition area is difficult because of the vessel geometry; if, however, the coverage is estimated to be roughly 20% of the torus surface area per source, an average specific pumping speed of 0.3 liters/sec-cm² is obtained for thermal D_2 . This result is consistent with a sticking coefficient¹⁴ for thermal D_2 of $\sim 1\%$.

With freshly gettered surfaces in the torus, the partial pressure of H_2O decreases to background levels ($\sim 5 \times 10^{-9}$) and TDC has little subsequent effect.⁴ It is uncertain whether this behavior is due to the increased pumping area for H_2O (CH_4 is not pumped by titanium), or to the difference in oxygen binding to titanium relative to ungettered surfaces. After complete saturation or poisoning of the gettered surface by exposure to atmosphere, the behavior of H_2O during TDC again is similar to the behavior of ungettered surfaces.

V. SURFACE ANALYSIS

Observations of changes in elemental surface composition of a movable surface analysis probe exposed to the vessel conditioning procedures are summarized in this section. A stainless steel tape welded into a continuous loop provides the sample substrate. Within the torus a cylindrical shield with a single

slit defines the exposed area of the tape. The axis of the probe intercepts the toroidal magnetic field (B_T) at an angle of $\sim 70^\circ$ from the normal. The position of observed surface changes on the substrate relative to the projection of the exposure slit along the direction of B_T can be used to separate the effects of charge particle from neutral particle impact,¹¹ (see Fig. 5c). For most of the data reported in this section the surface of the stainless steel tape was untreated before exposure. Therefore, prior to exposure Auger analysis shows the expected multilayer (hydro) carbon surface contamination on a 100-200 Å passivation oxide layer. For some exposures, 5000 Å films of In, (Al or Ag) were vacuum-evaporated or electrodeposited onto the tape to provide an Fe-free substrate.

Figure 4a shows the change in surface composition of such an uncoated stainless steel area after exposure to 5000 TDC pulses, at a radius of 50 cm (i.e. at the vacuum vessel wall). Significant removal of carbon is seen on the area below the exposure slit and not on the area of the projection of the slit along B_T . Concomitant with the carbon removal is the exposure of the underlying $\text{FeO-Cr}_2\text{O}_3$ oxide layer on the substrate. The increased concentration of S and Cl beneath the exposure slit can be the consequence of either the exposure of these impurities as intrinsic to the surface layer of the steel, or the deposition as impurities in the discharge. Exposure of clean elemental films (In or Al) to extended series of discharge cleaning pulses showed that both elements can be transported by the discharge.

These same clean film experiments were used to detect the transport of metals by the discharge. No metallic deposition (<0.5 at.%) was detected on clean In films exposed to 5000 TDC pulses; however occasional deposition of both wall and limiter metals was observed on films exposed to PDC pulses.

Figure 4b shows the change in elemental surface composition for the stainless steel tape as a function of exposure to TDC pulses. For exposure at the wall radius (50 cm), the rates of carbon removal, and iron and oxygen exposure, are linear up to an exposure of 5×10^3 pulses and then saturate. When the surface probe was exposed to radii smaller than the wall radius, saturation of the carbon removal (and iron exposure) occur sooner (see Fig. 5a). The radial dependence of the iron increase is plotted in Fig. 5a, showing a $30\times$ faster carbon removal rate for $r \leq 47$ cm than for $r \geq 50$ cm. Figure 5b shows an x-y scan of the Fe Auger signal on a substrate exposed to 450 TDC pulses at a radius of 46 cm. Longer exposures show broadening of the area depleted of carbon (to $6\times$ the slit width at an exposure of 15,000 pulses). The observed signals peak beneath the perpendicular projection of the exposure slit to the substrate surface, rather than beneath the projection along the B_T -field direction (Fig. 5c). Combining this observation with the strong radial dependence (Fig. 5a) of the carbon (iron) removal (exposure), and the high transparency of the low density plasma to neutrals, leads us to conclude that for $r \leq 47$ cm charged particles are responsible for the cleanup of the sample. Charged particles

will land directly under the slit if they initially have Larmor radii, $\ell \geq 1$ mm, or if there is a large accelerating potential difference between the plasma and the probe.¹¹ Electron bombardment of CO and CH_n covered surfaces usually results in carburization.¹⁵ Also electron energies greater than 2 keV are required for $\ell \sim 1$ mm. Moreover, the surface cleanup is not due to a heating effect since heat flow along the field lines is carried by the electrons. In contrast, H₂⁺ ions need only have energies of ~ 2 eV to have $\ell \sim 1$ mm. Thus we conclude that ions are responsible for the observed erosion of carbon for $r \leq 47$. The limiters are thus cleaned by ions, but we have not established that the vessel walls are also cleaned by ions.

In general, we observe on samples exposed to PDC similar changes in surface composition as observed on samples exposed to TDC. However, detailed analysis of PDC samples has shown erosion profiles that are neither as reproducible nor as symmetric as the profiles observed on TDC samples. In some instances carbon is seen to build up on areas of the tape adjacent to the slit projection (see Fig. 6). This effect has been reversed by changing the toroidal field direction. Generally for PDC the carbon removal rate (or the Fe or O exposure rate) was found to increase as the toroidal field was decreased. (In contrast, no field dependence was found for TDC.) The elemental profiles plotted in Fig. 6 show broad "shoulders" spanning distances equal to many times the slit width. Measurements after exposure of the sample to $\sim 10^3$ PDC pulses showed the oxygen coverage directly

under the slit decreased from a maximum of ~25 at.% (after ~500 pulses), to ~15 at.%, while the oxygen coverage at the peak of the "shoulder" increased to 40 at.%.

It is interesting to note that repeated lengthy (>24 hour) exposures of TDC- or PDC-cleaned samples to air did not result in appreciable recontamination.

VI. DISCUSSION

The SAS and RGA measurements show clear agreement in the removal of C from stainless steel and the resulting formation of CH_4 . The carbon removal rate, determined from the SAS data at $r \approx 50$ cm, and the CH_4 exhausted by the vacuum pumps, are roughly equivalent: approximately 2×10^{11} C atoms/cm² pulse. At $r \approx 46$ cm the removal rate is ~30× faster. At that smaller radius the expected combined flux of H^+ , H_2^+ and H_3^+ to the sample is about 10^{15} /cm² pulse, therefore making C erosion a very inefficient process.

In contrast, the SAS measurements have never shown a net decrease in O on stainless steel samples exposed to TDC or PDC, while the RGA data shows ~0.5 equivalent monolayers of O removed as H_2O during the TDC measurement period. Similar experiments by Clausung et al. on the ISX¹⁶ and T-12¹⁷ tokamaks have shown net decreases of surface oxygen on stainless steel samples exposed to H_2 discharge cleaning. (Although, the ISX exposures were generally $> 10^5$ pulses.)

A further complication to the question of oxygen and carbon removal is the fact that the wall has been found to be covered

with several hundred Å's of sprayed metal oxide and carbide.¹² So it is difficult to extrapolate wall clean-up rates using stainless steel samples when the actual wall is something quite different.

The mechanisms responsible for the removal of adsorbed carbon and oxygen atoms during discharge cleaning have not been established. However, we infer that chemical processes involving bond-breaking by incident atomic and molecular hydrogen ions and subsequent (atomic and molecular) hydrogen attachment are important. Examples of possible reactions of atomic hydrogen with adsorbed hydrocarbons yielding CH_4 have been given by Rye.¹⁸ The most likely reaction yielding H_2O based on equilibrium constants is empirically:

Less likely to be a source are the reduction of Cr_2O_3 and TiO . Both oxides are present on the wall but the respective equilibrium constants for the reduction reaction are 10^5 and 10^8 times smaller than the equilibrium constant for the above FeO reduction.

Though no measurements have been made of the abundances of H^+ , H_2^+ , H_3^+ and H^0 during discharge cleaning, simple comparisons of H_2 -e, H-e scattering processes indicate that the H^0/H_n^+ ratios should be large. But as previously noted, the transparency of the plasma combined with the strong radial dependence of the carbon removal indicate that ions are responsible for clean-up at $r \leq 47$ cm. (There are no measurements of n_e at the wall during

discharge cleaning, therefore we cannot state that ions are also responsible for the wall clean-up.) Since the H^0 Franck-Condon energy is comparable to the expected H^+ thermal energy we believe that the ions must be accelerated to ~ 10 - 20 eV by the plasma sheath, and thus their interaction with the adsorbed gases is greatly increased.

Two final questions remain: why does TDC allow cleaner plasmas than PDC; and can TDC be improved? An answer to the first, which is consistent with all the presented data, is that the low plasma temperature and density do not ionize H_2O formed by H_n^+ or H^0 impact on the walls.² In PDC any H_2O formed will be ionized to energetic and reactive radicals or ions, species which evidently have higher sticking coefficients than molecular H_2O . An additional difference between PDC and TDC is evident in the comparison of Fig. 4a with Fig. 6. Figure 6 shows that C and O moved from one area by PDC frequently land and stick on nearby (1-5 mm) areas. This behavior is indicative of ion motion in the 2 kG toroidal field. There is, however, no clear-cut difference in oxygen coverage directly under the slit when a sample is exposed to either TDC or PDC.

The second question has been investigated, in part, by: increasing the repetition rate of TDC; stretching the pulse; and changing the magnetic field, plasma current, and the applied loop voltage. Increasing the repetition rate did increase the H_2O pressure, presumably accelerating the clean-up. Power supply limitations did not allow the repetition rate to exceed 2 sec^{-1} .

Increasing the pulse duration to ~80 ms did not increase the H_2O pressure. This is consistent with the H_2O dissociation (ionization) argument. The mean free time for H_2O dissociation (ionization) in a 5 eV, $5 \times 10^{11}/cm^3$ electron plasma is about 0.02 (0.1) sec. Hence, an increase in the duration of the pulse will cause more H_2O breakup. Thus there is a peak in TDC efficiency between a continuous discharge and a pulsed mode. As noted previously, changing B_T from ~1 to 8 kG did not appreciably alter the H_2O production. Attempts to vary the plasma current showed an upper limit of ~15 kA beyond which the T_e increase resulted in such accelerated ionization that TDC conditions degenerated into PDC conditions. Increasing the applied loop voltage has allowed breakdown at higher filling pressures with greater TDC water production. Further increases in voltage may be possible. One way to improve TDC seems evident - withdraw the limiters to larger radii allowing more plasma contact with the wall. We are unable to test this on PLT due to the vacuum vessel construction.

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FIGURE CAPTIONS

Figure 1. (a) Background residual gas spectra before and after Ti gettering within the vacuum vessel. (b) Residual gas spectra during PDC and TDC conditioning.

Figure 2. Transient behavior of the primary residual gas components during TDC and PDC conditioning. The discharge pulse is indicated by the dotted lines.

Figure 3. Time-averaged partial pressure of H_2O during TDC (P_{H_2O} is averaged over the TDC pulse period). After 6000 pulses 0.88 torr-liters of O_2 was injected into the torus to quantify the observed H_2O pressure drop with oxygen coverage.

Figure 4. Observed changes in elemental surface composition of a hydrocarbon-covered 304 stainless steel substrate exposed to 5000 TDC pulses at a radius of 50 cm: (a) spatial dependence; (b) temporal dependence.

Figure 5. (a) Radial dependence of the change in surface composition of stainless steel exposed to varying total numbers of TDC pulses. (b) x-y scan of Fe Auger signal on the substrate surface after exposure to 450 TDC pulses. (c) Schematic diagram of the surface analysis probe showing Fe exposure on the substrate directly beneath the exposure slit rather than beneath the area of the slit projected along B_T . $H^+(D^+)$ ion impact can explain the observed C removal (Fe exposure).

Figure 6. Effect of PDC on a stainless steel substrate. Note the broad shoulders, peak-valley, and peak-to-peak features in each elemental spectral profile. This is a clear indication

of ionization of the eroded particles and subsequent ion gyromotion in the (2 kG) toroidal field.

TABLE I

A. DISCHARGE CLEANING PARAMETERS

	<u>PDC</u>	<u>TDC</u>
Discharge Duration (s)	.04	.04 (a)
Repetition Rate (s^{-1})	1/12	1/2
Magnetic Field (kG)	2-5	2-5
Plasma Current (kA)	50-100	2
Peak Power (kW)	300 kW	24 kW
Peak Power Density (W/cm^2)	.6	.5
Central Electron Density (cm^{-3})	2×10^{13}	10^{11}
Central Neutral Gas Density (cm^{-3})	$< 10^{11}$	$10^{13} \pm .5$
Central Electron Temperature (eV)	100	(2-5) (b)
Major Residual Gas Components	CH ₄ , CO	H ₂ O, CH ₄
Z _{eff} of Subsequent High Power Discharges	4-10	1.5-2.5

(a) A D.C. mode consists of a single pulse and an A.C. mode consists of 12 pulses at 280 Hz.

(b) An estimate based on spectroscopic measurements which show no CIII or OIII emission and minimal CII and OII emission.

B. GETTERING PARAMETERS

Sublimation Sources	(3) 35 gm Varian "Ti-balls"
Sublimation Rate/Source	0.2 - 0.5 gm/hr
Sublimation Period	~ 0.5 hr before start of run; 3-5 min. between discharges
Approximate Area of Coverage/Source	~ 2×10^4 cm ²
Z _{eff}	1 - 1.5

Fig. 1. 786079

Fig. 2. 786074

Fig. 2. 77347

Fig. 4. 786078

Fig. 5. 786083

Fig. 6. 773069