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MEMORANDUM REPORT NO. 2497



## APPARATUS FOR DETECTING INTERIOR BALLISTIC COMBUSTION PRODUCTS

Kevin J. White  
Robert W. Reynolds

July 1975

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USA BALLISTIC RESEARCH LABORATORIES  
ABERDEEN PROVING GROUND, MARYLAND

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| 20. ABSTRACT (Continue on reverse side if necessary and identify by block number)<br>A molecular beam sampling system has been developed for the purpose of studying transient chemical species produced in the interior ballistic combustion process. The system consists of four differentially pumped vacuum stages with a time-of-flight mass spectrometer used as a detector. Maximum flexibility was considered in the design, especially with respect to the reactor operating pressure. It is believed the reactions can be studied in the pressure range of a few torr up to 5000 psi. A number of factors that went into the design will |                       |                                                                                         |

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20. Abstract.

be discussed such as pumping capacity requirements, boundary layer problems, minimum reaction time calculations, minimum sensitivity, mass separation problem, mass cracking pattern for radicals and excited species, non vibrational equilibrium problems, formation of initial jet, Reynolds number consideration, choice of skimmer cone angle, mass clustering problem, background penetration, beam quality analysis with such techniques as velocity profile, avoidance of shock formation at various stages, and finally the choice of a well known chemical system on which to test the apparatus.

Some initial studies will involve the decomposition flames of ethyl nitrate and n-propylnitrate.

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## I. INTRODUCTION

Numerous phenomena observed in the combustion and ignition processes related to guns and rockets have escaped a thorough explanation and, in some cases, are causing weapons performance difficulties. Some of the problems are directly related to the propellant function and formulation such as 1) energy lost in rocket propulsion due to frozen equilibrium; 2) plateau combustion and the effect of catalysts on combustion; 3) the aluminum cartridge case burn through problem; 4) gun barrel erosion; 5) high ignition temperature propellant; 6) noise generation in recoilless rifle systems which has been traced to the high ignition pressure required by some propellants; 7) gas leakage around in-bore projectiles and anomalously high ionization of these gases; and 8) ignition problems associated with fumer formulations. A solution to these problems will require a better understanding of the physical and chemical processes active in these phenomena. It is not clear exactly how chemistry affects all of these observations but it is believed to be important in at least some of them. An example of this is in the problem of gun barrel erosion where it was thought that temperature was the important parameter. A great deal of effort went in to formulating new propellant systems with lower flame temperatures. Subsequent tests have shown that the erosion problem was in some cases exacerbated by these new formulations. It is now thought that heterogeneous chemical reactions may also play an important part in this problem.

Determining the chemistry involved in reactions under gun and rocket conditions is very difficult experimentally because of the speed and complexity of the reaction, and the high pressures involved. Furthermore, under gun conditions, the entire process is nonsteady and involves almost exclusively transient reactions.

Several types of spectroscopy can be used to study molecular species under these conditions. At present optical and IR analytical methods are being employed in other subtasks in the Ignition and Combustion programs.

We are attempting to develop a mass spectrometer system as a detection device for analysis of chemical species generated during the course of a chemical reaction. However, since a mass spectrometer must operate under very low pressures ( $10^{-9}$  N/m<sup>2</sup> or less) and since the chemical reactions of interest are at high pressures (1-5000 atm; 0.1-500 MN/m<sup>2</sup>) an enormous problem is encountered in going from one pressure to the other without significantly perturbing the chemical make-up of the system. To accomplish this one could immerse a probe or some sort of surface with a hole in it into the reacting medium and allow the species to leak through into the low pressure region of the mass spectrometer. However such a procedure means that the sampled species will come from the boundary layer of the sampling orifice. This will certainly be true under effusive flow conditions (hole diameter < mean free path of molecule). This will yield a well characterized flow but the chemistry, particularly of reactive species, will be suspect. Moreover, with gases at

300 K and 1 atm ( $\lambda = 6 \times 10^{-6}$  cm) there is a great deal of difficulty in fabricating such a hole for effusive flow.

In 1951 Kantrowitz and Grey<sup>1\*</sup> suggested a molecular beam sampling technique for overcoming these problems. However, in spite of the fact that over 20 years of research have gone into characterizing this system, many questions about the beam remain unresolved and in other instances characterization of the beam is entirely empirical.

The Kantrowitz and Grey idea evolved from what had long been a well developed procedure in engineering, the use of a Laval supersonic nozzle as a sampling orifice. A converging-diverging nozzle of this kind has several characteristics which are very useful for such an application:

- 1) The flow through such a nozzle is well characterized.
- 2) Boundary layers on the axis of the nozzle can be avoided by proper choice of Reynolds number at the entrance

$$Re = \frac{\rho D v}{\mu}$$

Since the Reynolds number increases with hole diameter,  $D$ , and density,  $\rho$ , then a sampling hole considerably larger than the mean free path fulfills this requirement. It has been suggested that boundary layers can be avoided with Reynolds' numbers greater than 500.

- 3) The static temperature and density are greatly lowered in passing through a nozzle. This means that the pressure is now more consistent with the operating conditions of a mass spectrometer. In addition, the lowered static temperature that comes about because of the expansion process means that fewer chemical reactions will take place during sampling. As was mentioned the density is lowered through the nozzle and depends on the orifice size and initial pressure and temperature of the gas. However, a second chamber (skimmer section) is needed to extract the core of the flow so that this can be used for mass spectrometer detection. This core will have a smaller chance of being contaminated by species from the boundary layer. The orifice for this chamber will introduce a lower quantity of gas into a second chamber where practical pumping capacities can be realized.

Depending on the initial operating pressure, calculations show that a third pumping stage may be needed in order to reach mass spectrometer operating pressures.

This gives an overview of the system. We will now describe the phenomena and problems associated with each stage. A schematic of the system is given in Figure 1.

\* References are listed on page 30

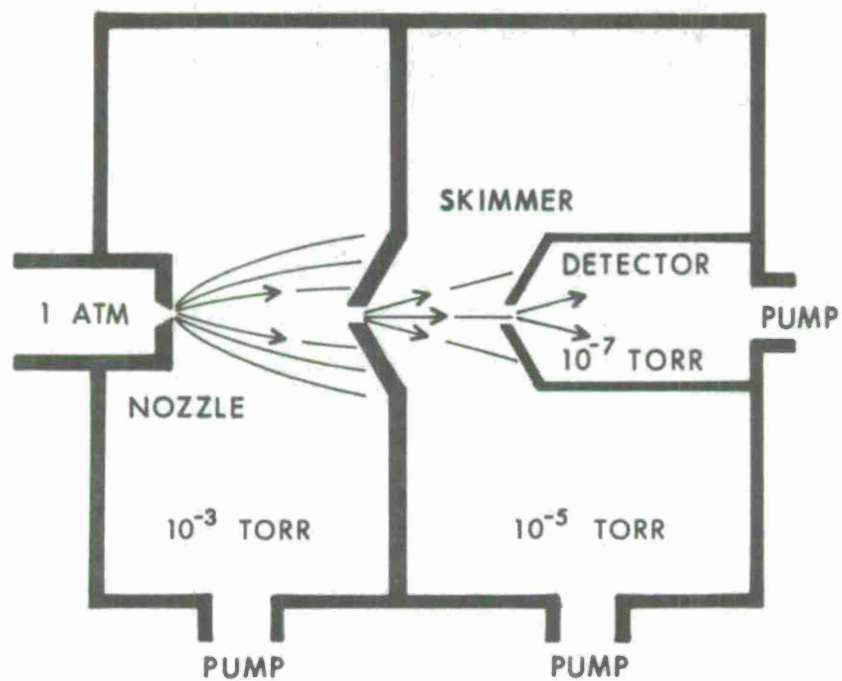


Figure 1- Block Diagram of Supersonic Molecular Beam System



## II. APPARATUS DESIGN

### A. Nozzle

Owen and Thornhill<sup>2</sup> used the method of characteristics on the conservation equations to characterize the flow through a nozzle where the downstream pressure was assumed to be zero. From this calculation one arrives at the velocity ( $v$ ) as a function of axial distance. Since this is an isentropic flow, all hydrodynamic calculations hold downstream, at least up to the formation of any shock waves. Very simply, the temperature along the axis can be found from the energy conservation equation:

$$h_o + \frac{1}{2} v_o^2 = h + \frac{1}{2} v^2. \quad (1)$$

If the initial gas has zero bulk velocity then

$$h_o - h = c_p(T_o - T) = \frac{\gamma}{\gamma-1} R(T_o - T) = \frac{v^2}{2}. \quad (2)$$

Physically this means that the enthalpy of the gas is converted into bulk velocity downstream. Here we see the conversion of random thermal energy ( $T$ ) to bulk kinetic energy ( $v$ ). This is an important process in quenching chemical reactions with positive activation energies. The sound speed is given by

$$c = (\gamma RT)^{1/2}. \quad (3)$$

Since we know  $T$  downstream we can calculate the Mach number,  $M = v/c$ . Physically, this is the ratio of directed bulk velocity to random velocity. Equation 2 can now be written in terms of  $T$  and  $M$  and we have  $T$  as a function of the calculated  $v$ .

Ashkenas and Sherman<sup>3</sup> have extended the Owen and Thornhill calculations and their results are shown in graphical form in Figure 2. Assuming an ideal gas we can also find  $\rho$  and  $p$ . The ratios  $T/T_o$  and  $\rho/\rho_o$  as a function of downstream distance in nozzle orifice diameters are given in Figure 3. Since the bulk velocity  $v$  is known, then the time to reach any temperature ratio  $T/T_o$  can be found. This is important in determining what reactions can take place during the sampling process. It has been found experimentally that a converging-diverging nozzle is not actually necessary but that a hole in a thin-walled plate gives essentially the same results as that of a nozzle. A schematic of the shock details which have been found experimentally for a thin walled orifice is given in Figure 4.

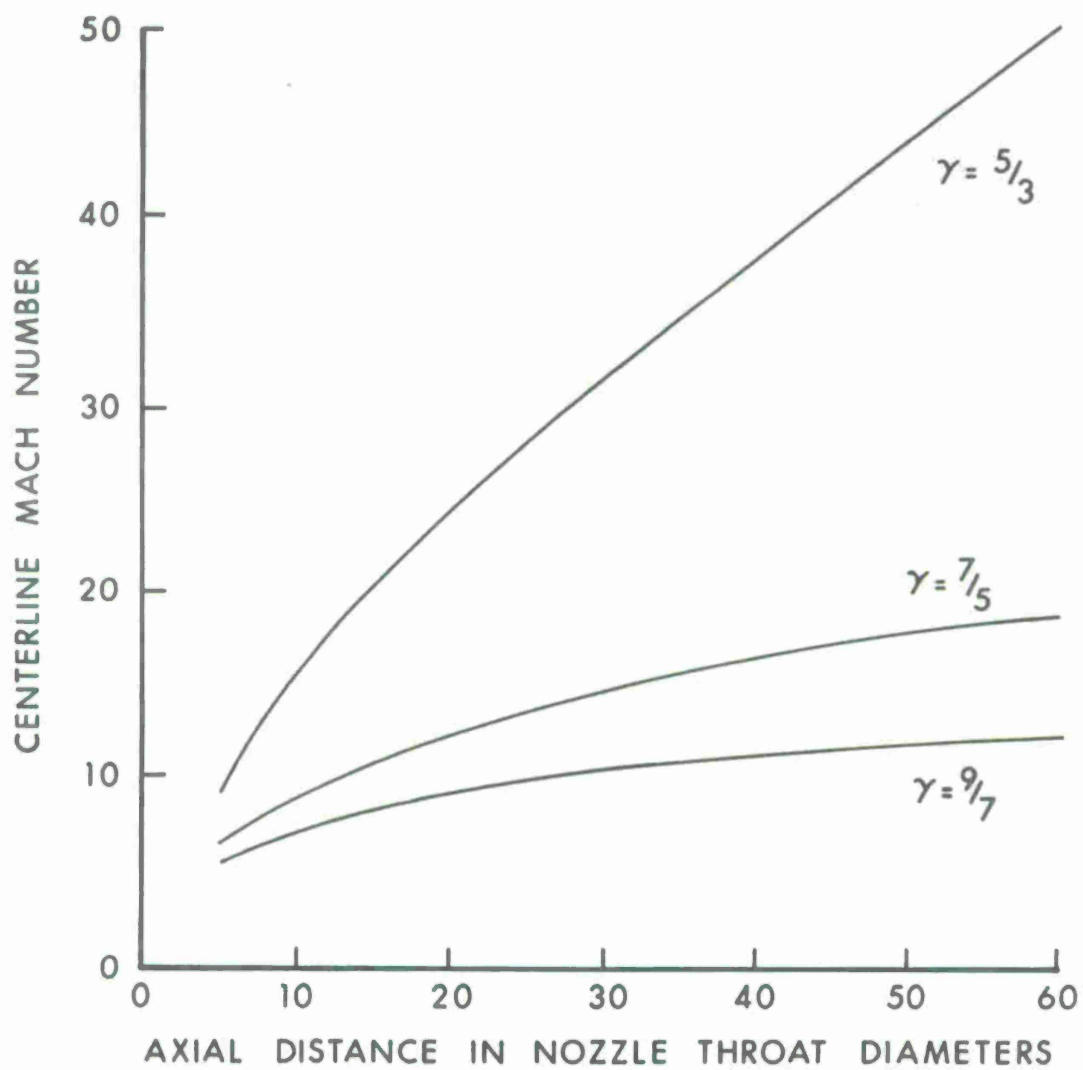


Figure 2 - Theoretical Axial Mach Number Distribution

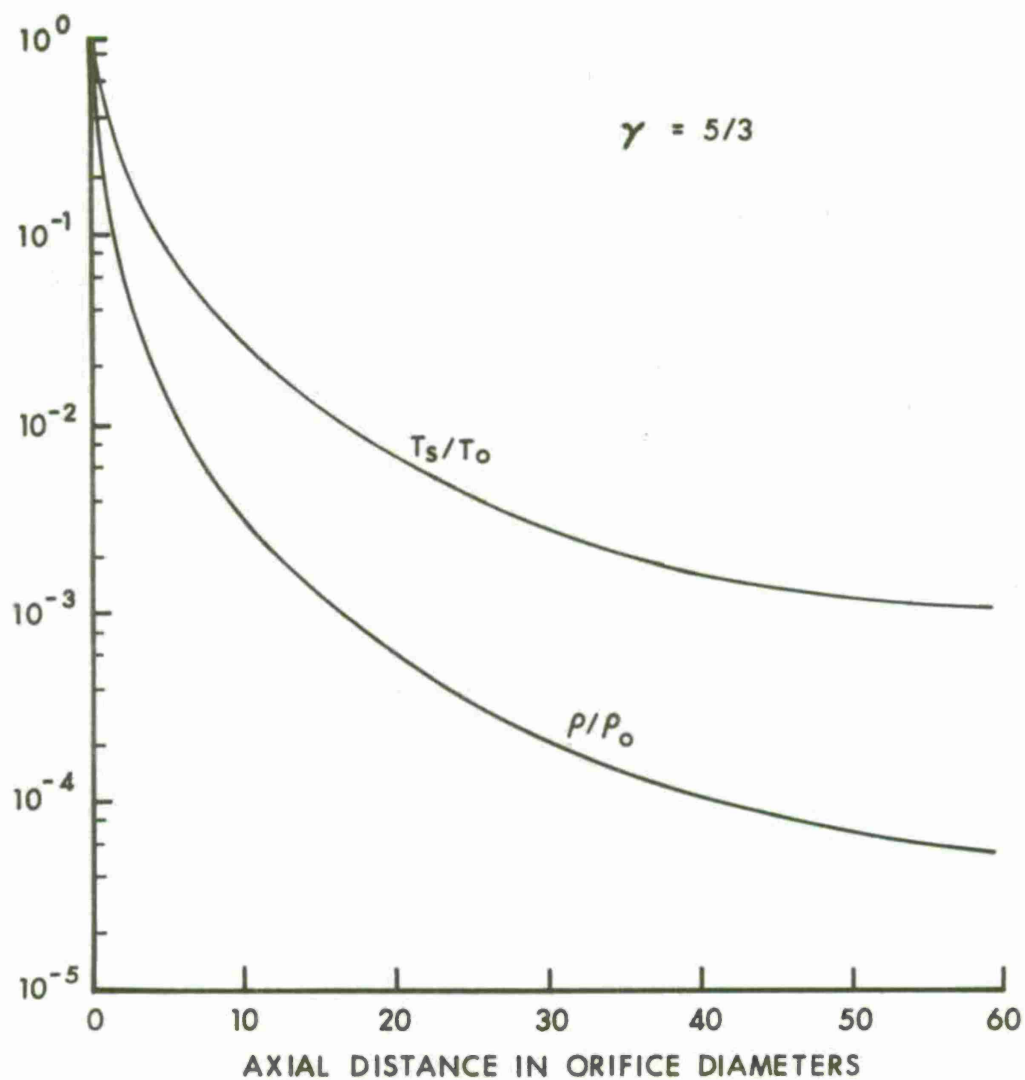


Figure 3- Isentropic Axial Density and Static Temperature Distribution

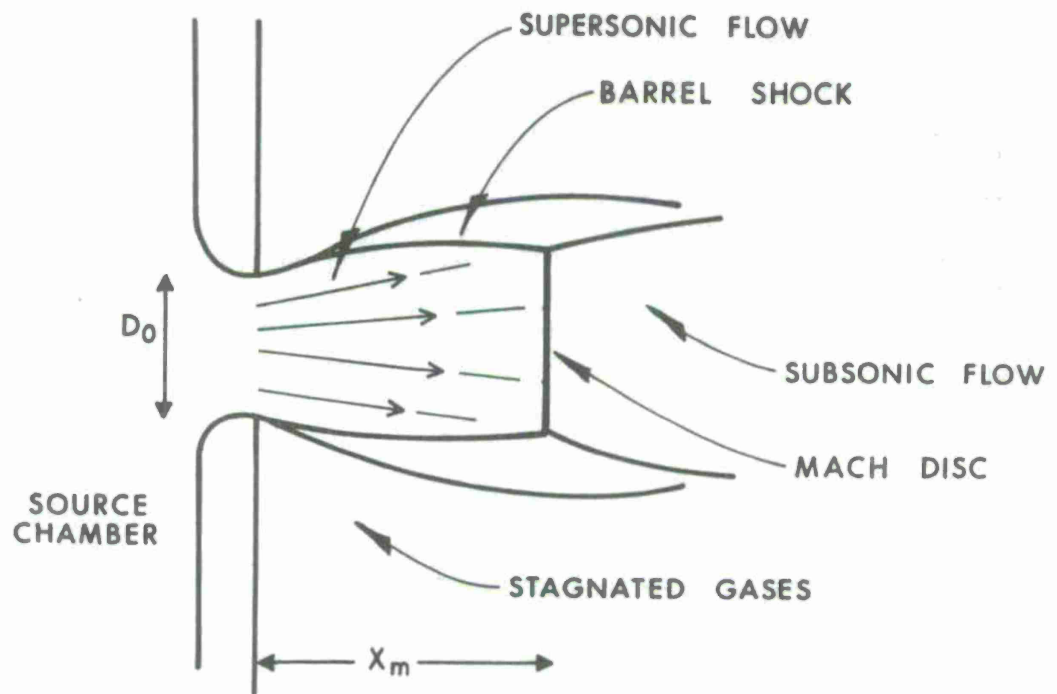


Figure 4- Formation of Free Jet

A number of things must be considered in the design of a nozzle and they are given below:

1) Shock Waves - As seen in Figure 4 a number of shock waves are generated in the formation of the jet expansion. These must be considered in the overall design.

2) Boundary Layer - Contamination of the centerline of the jet by boundary layers in the orifice can be avoided by orifice Reynolds' numbers greater than 500.

3) Mach Disc - As indicated in Figure 4 a shock wave is formed in the flow normal to the flow velocity. The location is given by the empirical formula

$$\frac{x_m}{D_o} = 0.67 \left( \frac{P_o}{P} \right)^{1/2} . \quad (4)$$

4) Terminal Mach Number,  $M_T$  - At some point in the flow the collision frequency becomes low enough that very little energy transfer takes place and there is virtually no more conversion of random kinetic energy into directed energy which translates into a constant Mach number. It can be shown that<sup>5</sup>

$$M_T = 1.17 Kn_o \frac{\gamma-1}{\gamma} \quad (5)$$

when  $Kn_o$  is the Knudsen number at the orifice.

5) Background Penetration - Work by Fenn and Anderson<sup>6</sup> has shown that penetration of background gas molecules into the molecular beam can be a significant problem. Scattering of the beam, contamination by other species, or further chemical reactions in the sampling process are not desirable. In surveying the literature and talking with investigators it is not clear at what point in the jet this becomes a problem. Knuth<sup>7</sup> has operated jets with high background pressures. He has deliberately introduced contaminants into his background, such as Argon, to see if he could pick up the signal in the mass spectrometer. He was not able to do this and concluded that when a high density jet is formed, the barrel shock and Mach disc protect the core from contamination. Others believe that there is no serious problem until one gets beyond the point of Mach number termination.

6) Vibrational Freezing - During the expansion process the vibrational temperature may not drop as quickly as the translational temperature. This could result in a non-equilibrium situation in the ionization source of the mass spectrometer which would lead to unusual mass cracking patterns. As a consequence identification and concentration measurements may be difficult to make.



7) Mass Separation - This is a phenomenon in which the ratio of light to heavy masses does not remain constant as a function of axial distance in the molecular beam. There are three causes of this:

a) Pressure Diffusion: There is a high radial pressure gradient in the jet and, after a given period of time this will cause the lighter species to diffuse out more rapidly than heavier species from the central core of the jet. Elaborate derivations have been carried out, and the phenomenon can be calculated.<sup>8</sup> Experimental results<sup>9</sup> indicate the validity of the model. This phenomenon becomes insignificant with orifice Reynolds numbers greater than 5000.

b) Shock Waves: As was mentioned, an orifice or skimmer will be needed down stream in the jet in order to transmit the beam to a lower pressure region. This surface will be in a supersonic flow with the possibility of shock wave formation. Physically the mass separation phenomenon upon passing through the shock wave can be explained in the following way. The light species are more easily deflected than the heavier ones by a shock disturbance and this will result in a depletion of the lighter masses. This problem can be minimized by construction of a skimmer with very sharp edges.

c) Mach Number Separation: Consider a binary mixture of gases containing high ( $m_1$ ) and low ( $m_2$ ) mass species. In the expansion process, if translational equilibrium is maintained between the two, the bulk velocities and temperatures will be the same at a certain point down stream. However, since

$$M = \frac{v}{c} = \left( \frac{R}{\gamma_m T} \right)^{1/2}, \quad (6)$$

the Mach number will not be the same. We shall see in a later section that when we put another orifice (the skimmer) in this stream the intensity down stream will depend on  $M^2$ . As a consequence the intensity ratio will vary as the mass ratio. This can be interpreted physically in the following way. To achieve the same bulk velocities the transverse velocity of the heavier mass species must be reduced to a lower value than a light mass species. The bulk velocities will be the same but the energy of two species will be different. The smaller transverse velocity for the heavier species will result in a lower dispersion. Consequently the mass ratios will change as a function of axial distance. This mass separation is predictable and can be used in the reduction of raw mass data.

8) Mass Clustering Or Condensation - The expansion process in the nozzle leads to a very low static temperature in the beam. In fact it is lower than the condensation point of most compounds. One would expect, and one does see, a certain amount of condensation or clustering. However, it is not as large as equilibrium values. This is because the rate of drop of temperature is much faster than the rate of cluster formation.<sup>10</sup> In fact as the orifice is made smaller the rate of temperature drop increases to a point where cluster concentration is the same as the equilibrium value

back in the reservoir calculated from  $T_0$ ,  $P_0$ . The result is that cluster concentration is a function of orifice diameter and initial pressure. This must be considered when making mass spectrometer measurements, i.e.,  $\text{Ar}_2$  or  $(\text{N}_2)_2$  should not be interpreted as another species at  $m/e$  80 or 56. Clustering can also be used to test the efficiency of the sampling system. The orifice size can be changed and the cluster concentration measured. Extrapolation to zero orifice size should lead to equilibrium concentration.<sup>11</sup>

9) Joule Thomson Effect - When a real gas drops in pressure, even under isenthalpic conditions, the temperature can increase or decrease depending on the initial temperature and pressure. Physically this can be interpreted in the following way. If the gases are in the attractive region of the intermolecular potential (low temperature region) then energy must be used in lowering the pressure. This will come from the kinetic energy of the gases and will be observed as a lowering of the temperature. If on the other hand, the gas is at a high temperature and consequently in the repulsive portion of the potential curve then energy will be released in going to lower pressure conditions. The so called inversion temperature separates these two regions and occurs for many gases in the region of 500 to 3000 K. Above this temperature the gas warms upon expansion. Calculations show however, that the temperature change is at most approximately 10 K for pressure changes up to 2000 psi ( $14 \text{ MN/m}^2$ ).<sup>12</sup>

## B. Skimmer

As can be seen from Figure 3, the densities down stream from the jet orifice are too large for operation of a mass spectrometer. Consequently another orifice and chamber are inserted in the molecular beam to "skim" out the central portion of the beam. This is called the skimmer. This surface, in the form of a cone with the orifice at the apex, is immersed in the supersonic flow down stream from the nozzle and consequently the formation of shock waves must be considered.

Kantrowitz, Grey,<sup>1</sup> Parker<sup>13</sup> and Anderson<sup>14</sup> have calculated the beam intensity down stream from the skimmer from kinetic theory assuming a) no skimmer interference with the beam and b) known conditions of density, Mach number, etc., at the entrance to the skimmer. These can be found from the nozzle continuum calculations. The results of the calculations are given in the following expressions and are shown in Figure 5.

$$\frac{dI_0}{dv} = A_s n_s \frac{1}{\pi \ell^2} \frac{1}{\pi^{1/2}} \frac{m}{2kT_s}^{3/2} v^3 \cdot \exp \left[ -\frac{m}{2kT_s} (v - v_s)^2 \right] \quad (7)$$

$$I_0 \approx \frac{A_s n_s v_s}{\pi \ell^2} \left( \frac{1}{2} \gamma M_s^2 + \frac{3}{2} \right) \quad (8)$$

From Figure 5 one can see the dramatic effect on the velocity distribution with increasing Mach number. It is seen from equation 8 that the intensity decreases as  $1/\lambda^2$  and is proportional to  $M_S^2$ , the Mach number at the skimmer. The mass separation problem discussed earlier can be predicted analytically from equation 8. The intensity  $I_0$  depends on  $M_S^2$  and consequently on  $m$ , the mass of the species.

Skimmer design considerations will now be outlined.

1) Shock Waves: Molecules passing through a shock wave will experience a number of collisions. This should be avoided to preserve the integrity of the beam. It can easily be shown<sup>15</sup> that, on the macroscopic scale, shock waves at the skimmer will be attached for flow Mach numbers greater than 2.5 if the skimmer cone angle is less than  $60^\circ$ . On a microscopic scale experience has shown that the cone lip should be as sharp as possible to avoid beam interference.

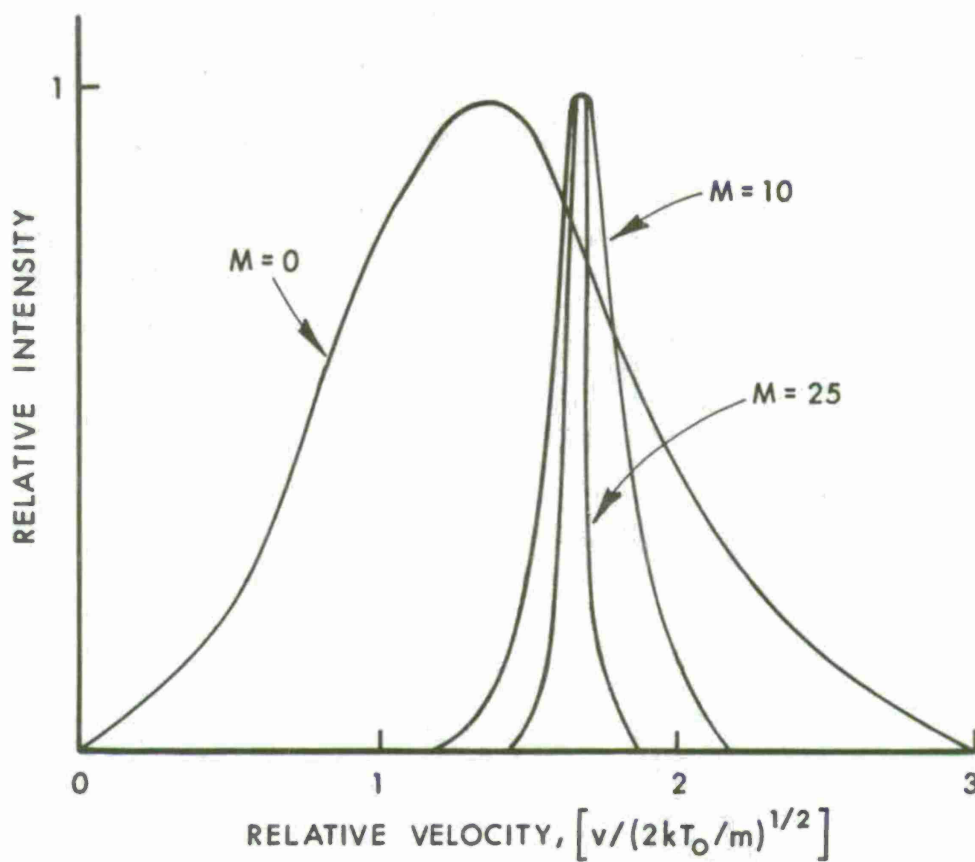
2) Optimum Beam Parameters: One of the measurements made by a number of investigators to test the efficiency of the sampling system is to measure the absolute values of the beam intensity and to compare this with the value calculated from expressions such as Equation 8. Experience has shown that calculated intensities cannot be realized. This is attributed to skimmer interference. However, it was found that for minimum beam attenuation,

$$Kn = \frac{\lambda}{D_S} = M_S \quad (9)$$

The ratio of the mean free path at the skimmer to the skimmer diameter should be as large as possible, or the ratio should be as close to the Mach number as possible. The physical reasoning behind this is not entirely clear and has been debated in the literature. However, some plausibility arguments offered are that a long mean free path is desirable since if a shock wave is formed the number of collisions before reaching the skimmer entrance will be smaller. The large Mach number will mean that the flow is more directed and hence more sensitive to perturbations. A skimmer orifice diameter smaller than the mean free path will insure that few collisions occur within the skimmer orifice.

3) Skimmer Location: Experimental results also show that increasing the nozzle-skimmer distance also increases beam intensity. This would indicate that the shock wave that is formed becomes weaker as the density decreases and consequently there is less intensity attenuation. There is a limit beyond which further nozzle-skimmer separation degrades the beam. At this distance, background penetration becomes more of a problem and this scattering lowers the beam intensity. When a skimmer is used with a high density jet it should be located inside of the Mach disc so that the species do not pass through this shock wave. It should probably be located in the flow approximately at the terminal Mach number region to avoid the background scatter that exists down stream from this point.





$$\frac{dl_0}{dv} = A_s n_s \frac{1}{\pi \ell^2} \frac{1}{\pi^{1/2}} \left( \frac{m}{2kT_s} \right)^{3/2} v^3 \cdot \exp \left[ -\frac{m}{2kT_s} (v - v_s)^2 \right]$$

Figure 5- Relative Beam Intensity after Skimmer

### C. Mass Spectrometer

Essentially three types of mass spectrometers were considered; magnetic, quadrupole and time-of-flight (TOF). Since transient as well as steady state phenomena were to be studied, consideration was given to the speed of the detecting system. The magnetic and quadrupole spectrometers are relatively slow. Since the magnetic spectrometer has the additional problem of physical bulkiness this system was rejected. The TOF spectrometer is very fast. The Bendix version can measure a mass range from 1-150 amu in 10  $\mu$ sec and at a frequency of 100 kHz. However, mass peaks for such a system are about 20  $\mu$ sec wide and recording techniques have not yet been developed which can digitize and store the large amount of data that would be generated in this short time frame. However, the TOF can be used in an averaging mode in which only one mass peak is observed during each sweep and, after a sufficient number of sweeps, moves on to the next peak. This however negates the prime advantage of the TOF, viz., that it looks at all masses with each 10  $\mu$ sec sweep. The quadrupole mass spectrometer has several advantages. The entire scanning process can be computer controlled with the data stored and analyzed. The quadrupole also has a higher resolution and, because of a higher duty cycle, has a greater sensitivity. However, the instrument is relatively slow with a sweep speed of about 1 amu/msec. We concluded from this analysis that a TOF was needed for transient studies in which 10 to 100 msec is the maximum sampling time. For steady state systems the quadrupole was the logical choice.

### D. Vacuum Pumps

Pumping speed requirements can be determined by the various beam intensity calculations shown in Figure 3 and equation 8. The requirements for each stage will be considered:

1) Mass Spectrometer Stage - Experience of other workers in this field indicates that residual mass spectra background is a persistent and annoying problem for performing good analyses. Two techniques can be used to reduce this problem. In the first instance pumps which do not use oil could be used such as turbo molecular pumps, magnetic ion pumps, and the cryo pumps. The turbo pumps proved to be expensive, noisy and to have a vibration problem, although they are very clean. The magnetic ion pumps are sometimes difficult to start and also fairly expensive. However, they are clean and perfectly quiet. The cryo pumps are also extremely clean but an article<sup>16</sup> on the subject indicated that there is a problem with long term pumping since insulating layers build up and render the pump inoperative. They are also somewhat inconvenient to use because of the coolants required such as liquid He. If oil pumps must be used the second method of avoiding background with a molecular beam is by intensity modulation using a beam chopper with mechanical modulation with such devices as tuning forks and rotating wheels. Phase sensitive detection could then be used to discriminate against background mass spectra. However, for transient studies of very

short duration, beam chopping frequencies may have to be too high to be achieved. Our conclusion from this analysis is that if transient studies are to be seriously considered then cryogenic pumps are probably the best since they would not have to pump continuous loads. Supplemental magnetic ion pumps could also be used, especially when steady state systems were to be studied.

2) Skimmer Stage - Cleanliness is not as critical at this stage consequently the cheapest and most reliable system is the oil diffusion pump of a size consistent with pumping needs.

3) Nozzle Stage - The system has been designed to operate at pressures as high as  $3 \times 10^7 \text{ N/m}^2$  (4500 psi) with temperatures up to 3000 K. As a consequence any hole or orifice will have a large mass flow rate. This will place extreme requirements on the pumping capacity of this first stage. Any one of these pumping systems can be used; a diffusion pump, a Roots blower or a very large cryo pump.

Figure 6 gives pumping requirements as a function of nozzle diameter. Figure 7 shows mass flow rates as a function of pumping speed for various pressures. The operating point will be where the curves intersect. AEDC<sup>17</sup> has a cryogenic pumping system where the speed is limited solely by the conductance of the pathway up to the pump. This requires large amounts of liquid He and is impractical in our case. A large 9m diameter ( $3.9 \times 10^2 \text{ m}^3$ ) sphere which can be evacuated down to 1/2 torr was also considered. This offered attractive possibilities for the high pressure transient situation but there were numerous practical problems (mounting pumps, etc. within the sphere's vacuum chamber) and it would not be very useful for low pressure experiments where nozzle pressures lower than 1/2 torr would be required.

We were left with the choice of either a diffusion pump or a mechanical pump. Since these operate over quite different pressure ranges an analysis was carried out to see which provides the most versatile pumping system. It must be remembered that we are interested in doing experiments in the region from 0.1 atm ( $10^4 \text{ N/m}^2$ ) and 3000 K up to 200 atm ( $20 \text{ MN/m}^2$ ) and 3000 K. Temperatures may also be as low as 400-500 K. Diffusion pump speeds fall off rapidly above a few millitorr whereas Roots blowers can operate up to a few torr, but speeds fall off rapidly below a milli torr. Using Figures 6 and 7 it is seen that nozzle orifice diameters for diffusion pumps must be around 0.1 mm and for Roots blowers approximately 1 mm. As a consequence the beam properties and other design considerations will vary considerably depending on the type of pump used in the first stage.

We will now examine in detail the differences between a system using a Roots blower and a system using a diffusion pump.



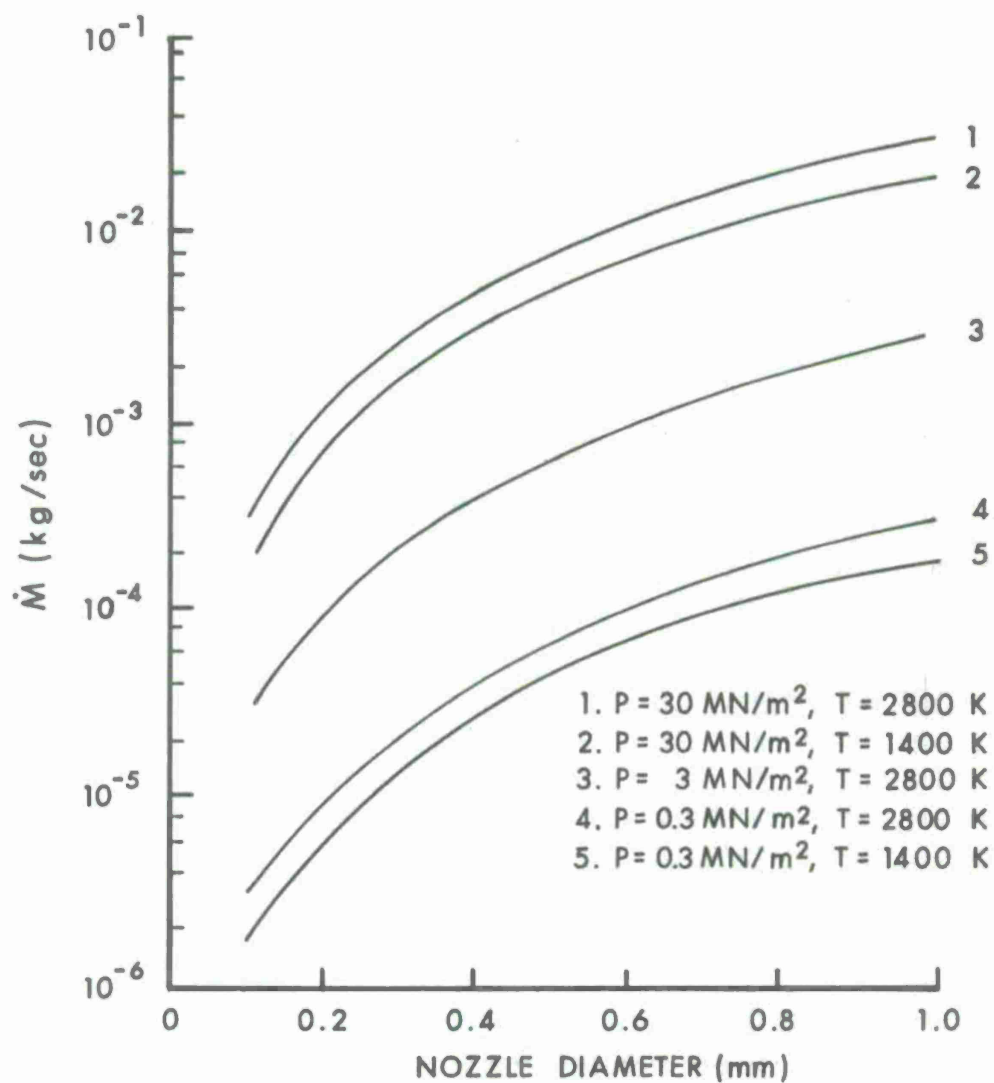


Figure 6 - Pumping Requirements: Mass Flow vs Nozzle Diameter (Parameters,  $T_0$  and  $P_0$ )

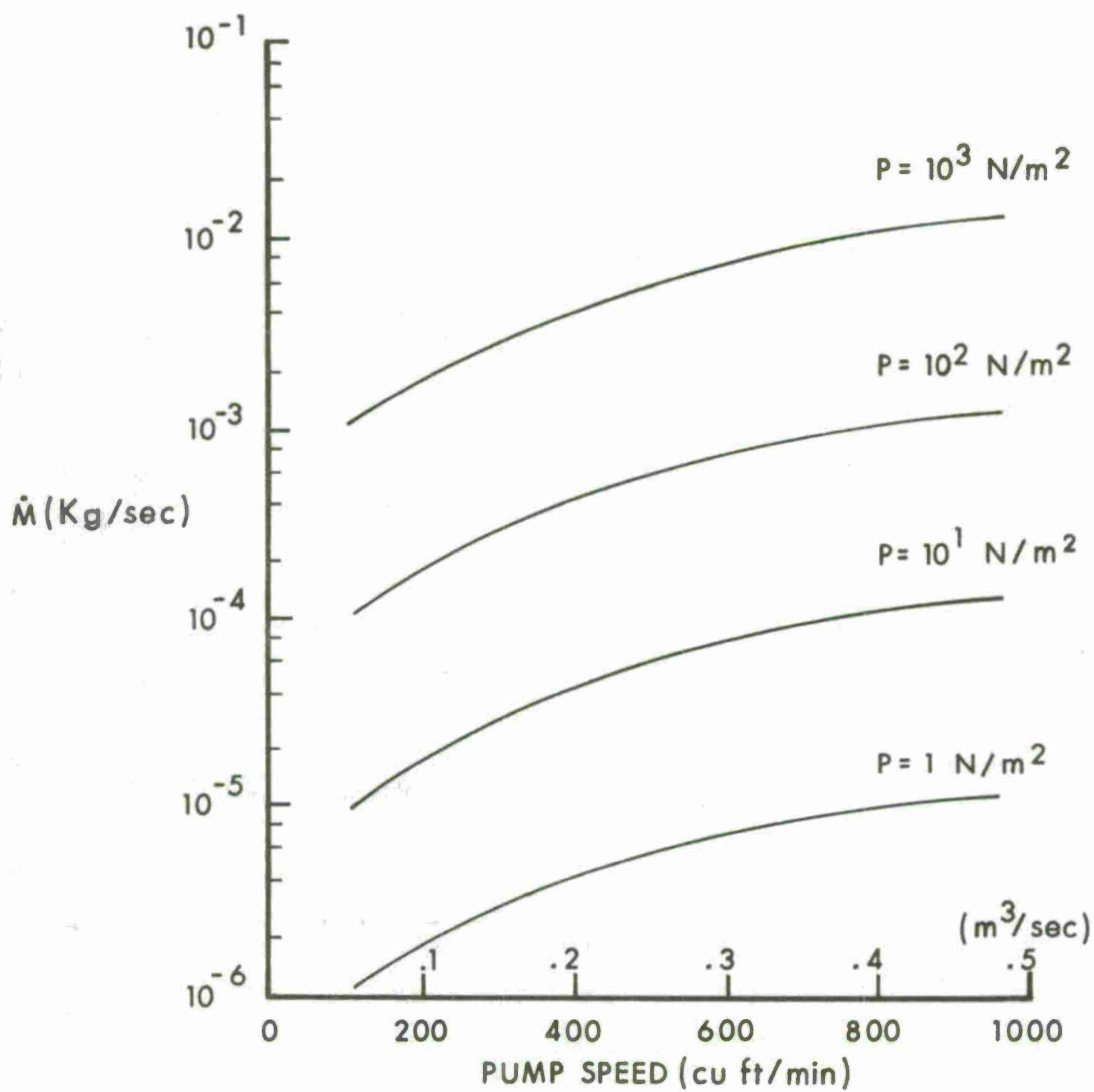


Figure 7 - Pump Speeds: Mass Flow vs Pump Speed  
(AVG MOL WT = 0.05 Kg)

a. Mach Disc Location - The actual physical location of the Mach disc will be the same in both instances. This means the nozzle to skimmer distance will be the same for both systems.

b. Reynolds Number - The criterion is, that to avoid boundary layer effects and pressure induced mass separation, the Reynolds number should be greater than 5000. This will be true for either system except in the low pressure studies (Table 1). Here the Reynolds number will be too small in the diffusion pump system. However, under these conditions the hole can be increased in size and this problem will be avoided. Consequently, this will not be a problem for either system.

c. Terminal Mach Number - Calculations show that this will be much smaller for the diffusion pump system in the intermediate pressure experiments. This will mean higher down stream temperatures with possibly more reactions and a loss of intensity.

d. Shock Formation at Skimmer - The density will be lower for the diffusion pump and hence the shock will be weaker and probably less of a problem than for the Roots blower system.

e. Rate of Temperature Drop - This will be larger for the diffusion pump system and hence it will be easier to observe shorter lived species. Order of magnitude calculations indicate that the temperature will drop a factor of 10 in 1  $\mu$ sec for the diffusion pump system and in 10  $\mu$ sec for the blower system.

f. Clustering - This will be less troublesome for the diffusion pump system because the ratio of temperature drop will be faster than the clustering rate.

g. Vibrational Relaxation - The opposite will be true here. The slower rate of temperature drop will induce less of a non-equilibrium problem in vibrational distribution with the Roots blower system. Consequently, the cracking patterns will be more "normal."

h. Nozzle Clogging - If the reacting system has any particulate matter, then the diffusion pump system will have more problems simply because of the smaller diameter hole.

i. Fabrication - Ultimately these nozzles will be subjected to high pressures and temperatures and will require a wall thickness adequate to withstand these conditions. This could result in an excessively large length to diameter ratio for the small hole used in the diffusion pump system which would lead to boundary layer build up and contamination of the central core of the beam.

Table 1. Reynolds Number as a Function of Orifice Diameter  $D(\text{cm})$ ,  $T_0(\text{K})$  and  $P_0(\text{MN/m}^2)$ . Viscosity ( $\mu$ ) =  $50\mu\text{NS/m}^2$  (500  $\mu\text{poise}$ ).

| $D(\text{cm})$ | $T(\text{K})$ | $P \frac{\text{MN}}{\text{m}^2}$ | Re      | 1 atm = $0.101 \frac{\text{MN}}{\text{m}^2}$ |
|----------------|---------------|----------------------------------|---------|----------------------------------------------|
| 0.01           | 300           | 0.01                             | 64      |                                              |
| 0.01           | 300           | 0.1                              | 640     |                                              |
| 0.01           | 300           | 1.0                              | 6,400   |                                              |
| 0.01           | 300           | 10.0                             | 64,000  |                                              |
| 0.01           | 3,000         | 0.01                             | 20      |                                              |
| 0.01           | 3,000         | 0.1                              | 200     |                                              |
| 0.01           | 3,000         | 1.0                              | 2,000   |                                              |
| 0.01           | 3,000         | 10.0                             | 20,000  |                                              |
| 0.05           | 300           | 0.01                             | 320     |                                              |
| 0.05           | 300           | 0.1                              | 3,200   |                                              |
| 0.05           | 300           | 1.0                              | 32,000  |                                              |
| 0.05           | 300           | 10.0                             | 320,000 |                                              |
| 0.05           | 3,000         | 0.01                             | 100     |                                              |
| 0.05           | 3,000         | 0.1                              | 1,000   |                                              |
| 0.05           | 3,000         | 1.0                              | 10,000  |                                              |
| 0.05           | 3,000         | 10.0                             | 100,000 |                                              |
| 0.1            | 300           | 0.01                             | 640     |                                              |
| 0.1            | 300           | 0.1                              | 6,400   |                                              |
| 0.1            | 300           | 1.0                              | 64,000  |                                              |
| 0.1            | 300           | 10.0                             | 640,000 |                                              |
| 0.1            | 3,000         | 0.01                             | 200     |                                              |
| 0.1            | 3,000         | 0.1                              | 2,000   |                                              |
| 0.1            | 3,000         | 1.0                              | 20,000  |                                              |
| 0.1            | 3,000         | 10.0                             | 200,000 |                                              |

j. Background Penetration - Experiments by Knuth have shown that this is not a problem when high jet densities are used as was discussed earlier. The shock waves "protect" the jet from invasion by background gases.

Investigators working with low density jets have experienced considerable problems with background penetration. In this instance the Roots blower system is less bothered by background penetration.

k. Pumping Speed - Although much higher pumping speeds can be achieved by diffusion pumps as compared with blowers, the actual limitation comes about from geometry effects, i.e., the available space between the skimmer and nozzle. As a consequence the advantage of a diffusion pump may be lost due to geometrical effects.

One of the most important considerations was the fabrication of the nozzle. The scale of this nozzle will be much larger for the Roots blower system and, hence, will be much easier to machine.

### III. MOLECULAR BEAM ANALYSIS

There are several types of analysis that can be carried out to determine the efficiency and performance of the system. These can aid in determining how much perturbation of the chemical species there is during the sampling process.

a) Electron Gun: The molecular beam is subjected to bombardment by an electron beam. The molecular species are electronically excited and emit visible radiation. The intensity of the radiation is proportional to the density. This technique can be used to determine qualitative features of the beam such as shock wave formation and structure. Such studies have been carried out by Jakus.<sup>18</sup>

b) Velocity Analysis: A time-of-flight velocity profile is probably the most useful indicator of the quality of the beam. Any unwanted collisions will show up as a broadening of the velocity profile and a lowering of the Mach number as indicated in Figure 5. A great deal of work has been done in this area. To perform this analysis, a mechanical molecular beam chopper is used. A detector is placed downstream and records the time profile of the beam.

c) Intensity Analysis: Actual beam intensities can be measured and compared with calculations to determine the efficiency of the system (Equation 8). Deviations from theory have been studied extensively.<sup>19</sup>

d) Argon Dimer Concentration: Milne and Greene<sup>13</sup> have found Ar concentrations as a function of nozzle diameter. These should lead to upstream equilibrium values as the diameter goes to zero. Deviations from this result would indicate poor beam quality.



e) Chemical System: The ultimate test will be in measurements made on a well-characterized chemically reactive system. This in itself is a difficult requirement, especially at the higher pressures where good chemical data are not available. For low pressure flames a number of investigators have used  $\text{CH}_4 + \text{O}_2$  as a system.<sup>20</sup>

#### IV. APPARATUS

A schematic of the apparatus is shown in Figure 8. In this setup we are studying flames in the range of one atmosphere and below. Consequently, the 2600 cfm ( $1.2\text{M}^3/\text{s}$ ) Roots blower, instead of being used in the nozzle region (as outlined in the previous section), is used only for evacuating the burner chamber. The nozzle region is evacuated by a 10" diffusion pump, the skimmer region by a 4" diffusion pump and the TOF mass spectrometer by a 300  $\ell/\text{s}$  ( $0.3\text{M}^3/\text{s}$ ) mag-ion pump along with a 2000  $\ell/\text{s}$  ( $2\text{M}^3/\text{s}$ ) helium cryopump.

The apparatus dimensions are given as follows:

Nozzle orifice diameter - 0.27 up to 1.4mm

Nozzle cone angle - external  $120^\circ$ ; internal  $103^\circ$

Skimmer cone angle - external  $60^\circ$ ; internal  $50^\circ$

Nozzle-skimmer distance - variable from 1mm to 20mm

Skimmer to mass spectrometer - 142mm

Typical operating pressures are: reaction chamber, 100 torr ( $1.3 \times 10^4 \text{N/m}^2$ ); nozzle region,  $10^{-3}$  torr ( $0.13 \text{N/m}^2$ ); skimmer region,  $10^{-5}$  torr ( $1.3 \times 10^{-3} \text{N/m}^2$ ); mass spectrometer region,  $10^{-8}$  torr ( $1.3 \times 10^{-6} \text{N/m}^2$ ).

When the system is to be used for high pressure studies the burner will be replaced by a combustion bomb with an orifice, or nozzle, on the end plate. The reaction chamber will then be the nozzle stage and will be evacuated by the Roots blower. The system can then be used as a three or four stage molecular beam apparatus. If it is to be used as a three stage system the skimmer would be removed and the skimmer and nozzle regions would be combined. This will depend on the combustion chamber operating pressure. A picture of the apparatus is shown in Figure 9 and a further description is given in reference 21. Originally this apparatus was used at the MIT Lincoln Laboratories for analyzing wakes behind projectiles fired from a light gas gun.

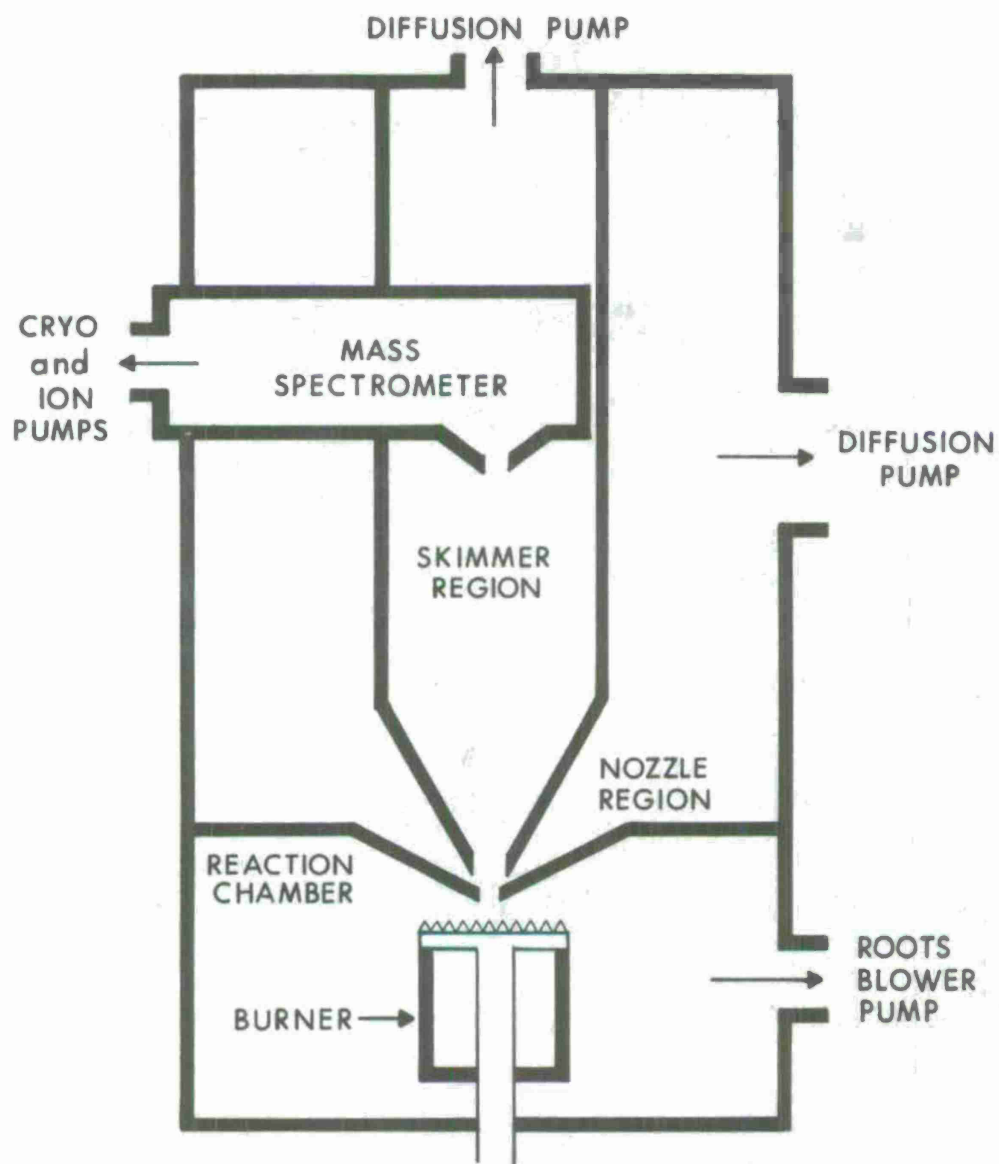


Figure 8 - Schematic of Supersonic Molecular Beam Sampling System

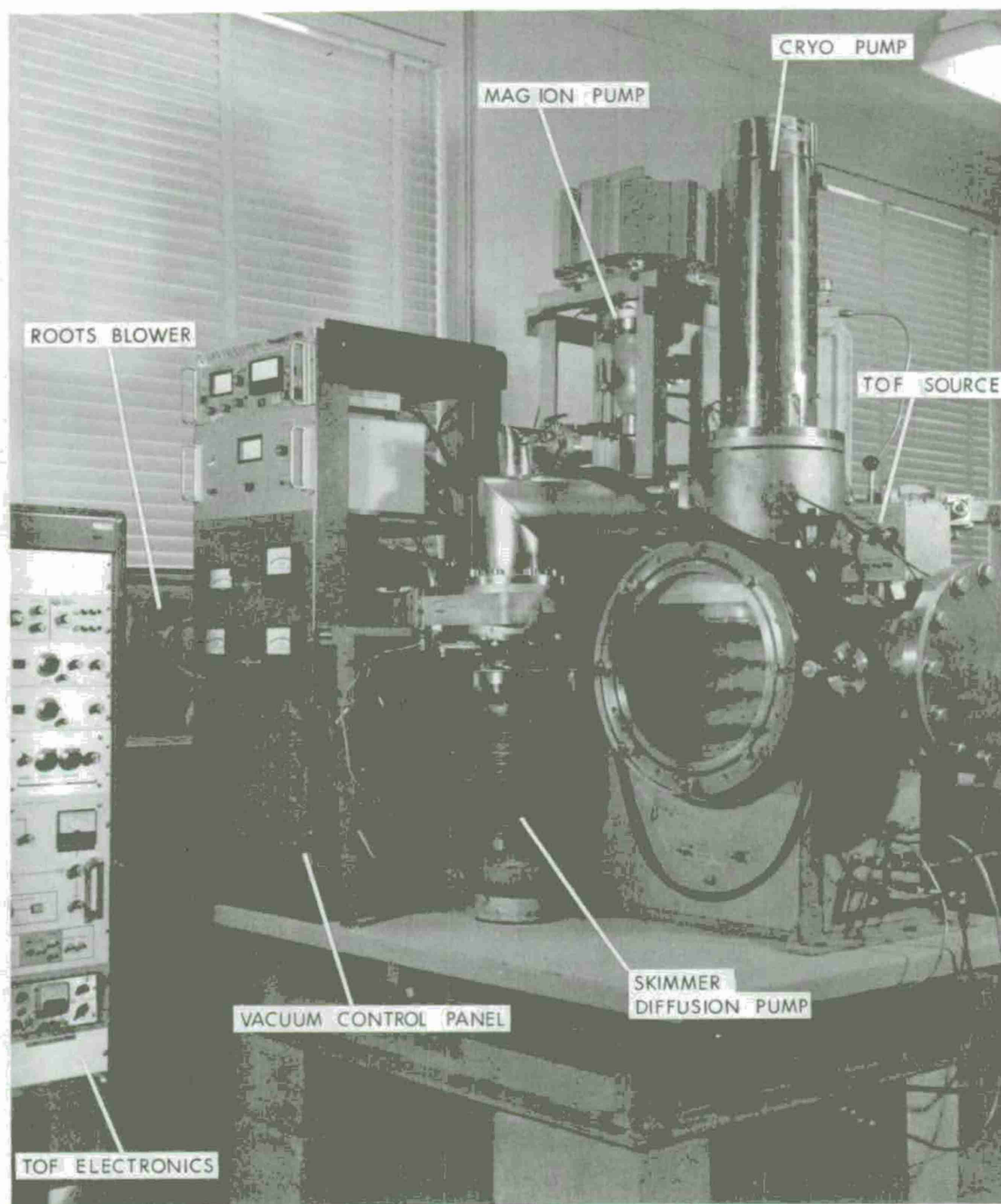


Figure 9 - Molecular Beam Sampling Apparatus

## V. CONCLUSION

The motivation for building this apparatus for studying the chemistry of combustion was outlined in the "Introduction" section. Some of the projects to be carried out using this experimental system will be outlined and related to the ballistic problems.

The mechanisms involved in the decomposition and pyrolysis of nitrate esters will be studied. Low pressure flames of ethylnitrate and n-propylnitrate will be studied in the range of a few torr up to ambient pressure. This will complement the work being carried out in the Ignition Program, "Radical Mechanisms in Ignition of Propellants." The effect of lead additives on flame properties will also be studied.

It is well known that these compounds have dramatic effects on the combustion properties of nitrate esters but the mechanism is not well understood.

Another problem in sub-atmospheric combustion that will be studied is the chemistry of ignition of fumer formulations. Base projectile ignition involves sub-atmospheric conditions and ignition criteria need to be clarified if new formulations are to be used in fumer applications. Some compounds show excellent characteristics for assisting in base drag reduction but frequently cannot be used because of ignition problems. A better understanding of the ignition chemistry may help in overcoming this problem.

Vulnerability of caseless ammunition continues to be an important obstacle in the development and application of this technology. The ignition and combustion of these compounds under ambient conditions with the effect of additives needs to be carefully studied in order to rationalize the choice of inhibiting compounds that can be used to reduce this vulnerability problem. Molecular beam sampling of these compounds ignited under ambient conditions will help in sorting out the chemistry of this problem.

High pressure studies (up to 4500 psi, 30 MN/m<sup>2</sup>) of the chemistry of the combustion of ammonium perchlorate are needed to help understand the dramatic effect of small quantities of additives on the combustion of this compound.<sup>22</sup> A combustion bomb will be constructed to operate in this pressure region, and will have an orifice in the end plate for extraction of chemical species. This bomb will also be used to study the combustion of single base propellants with and without the lead additives in order to understand the effect that these compounds have on the propellant combustion.



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## LIST OF SYMBOLS

|           |                             |
|-----------|-----------------------------|
| A         | area                        |
| c         | speed of sound              |
| $c_p$     | specific heat               |
| D         | diameter                    |
| h         | enthalpy                    |
| I         | intensity                   |
| k         | Boltzmann constant          |
| Kn        | Knudsen number              |
|           | axial distance from skimmer |
| M         | Mach number                 |
| m         | molecular mass              |
| n         | number density              |
| P         | pressure                    |
| R         | gas constant                |
| Re        | Reynolds number             |
| T         | absolute temperature        |
| v         | velocity                    |
| $x_m$     | axial distance from orifice |
| $\gamma$  | specific heat ratio         |
| $\mu$     | viscosity                   |
| $\lambda$ | mean free path              |
| $\rho$    | mass density                |

## Subscripts

|   |           |
|---|-----------|
| o | orifice   |
| s | skimmer   |
| m | Mach disc |

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