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NF ($a^1\Delta$) PRODUCTION IN A SUPERSONIC FLOW USING $N_2F_4 + H_2$ IN A BCL-16 NOZZLE

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Final Report

AIR FORCE WEAPONS LABORATORY
Air Force Systems Command
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A supersonic HF laser nozzle, BCL-16, has been used to produce high densities of NF($a^1\Delta$) from the N_2F_4 + H_2 reaction scheme. Through parametric studies, the flow rates to obtain optimum NF($a^1\Delta$) production were determined. The NF($a^1\Delta$) molecule has been considered for use as an energy storage and energy transfer molecule. The NF($a^1\Delta$) molecule is also a precursor of the highly energetic N_2(A) molecule. The N_2(A) species has also been considered as an excellent energy transfer molecule. The NF($a^1\Delta$) and NF($b^1\Sigma$) density spatial profiles were determined as a function of flow rate. Yields were determined and mixing examined.

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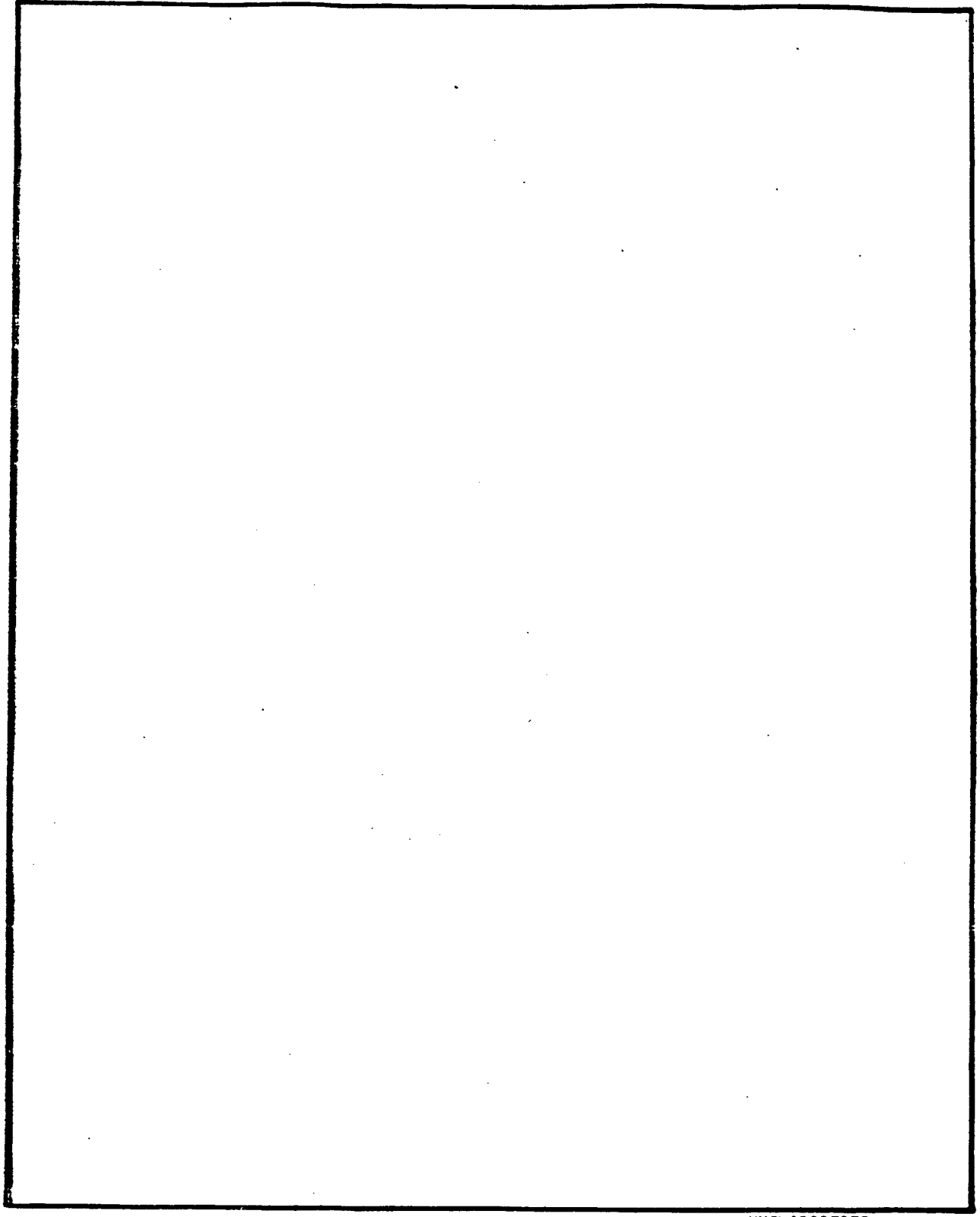

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ILLUSTRATIONS

INTRODUCTION

The $NF(a^1\Delta)$ has been of interest as an energy storage molecule for over 10 years. Work by Herbelin and Cohen in 1973 (Ref. 1) demonstrated that $NF(a^1\Delta)$ could be produced by chemical reaction. Malins and Setser (Ref. 2) as well as Clyne and coworkers (Refs. 3,4) produced $NF(a^1\Delta)$ efficiently (90%) over the ground state $NF(X^3\Sigma)$ in low pressure, low temperature flow systems.

The $N_2F_4 + H_2$ scheme for production of $NF(a^1\Delta)$ follows the general set of reactions as listed in Equations 1 through 5.

$$N_2F_4 \xrightarrow[800\text{-}1200K]{\Delta} 2NF_2 \qquad (1)$$

$$F + H_2 \longrightarrow HF + H \qquad (2)$$

$$H + NF_2 \longrightarrow HF(v' = 0\text{-}3) + NF(a^1\Delta) \qquad (3)$$

$$H + NF_2 \longrightarrow HF(v' = 0) + NF(b^1\Sigma) \qquad (4)$$

$$NF(a^1\Delta) + HF(v' = 2,3) \longrightarrow NF(b^1\Sigma) + HF(v = 0,1) \qquad (5)$$

The $NF(a^1\Delta)$ energy transfer has been investigated with potential visible chemical laser candidate molecules including BiF and Bi (Refs. 5,6). The $NF(a^1\Delta)$ is also of interest in terms of high production levels as a precursor to $N_2(A)$, another energy transfer molecule. This latter application was of interest for this work. The main objective was to determine maximum production levels of $NF(a^1\Delta)$ by parametric variation of reagent flows.

DEVICE DESCRIPTION

OVERVIEW

The overall experimental system consisted of a 316L stainless steel chamber with viewing ports on four sides. Figure 1 shows a top view of the chamber with positions shown for the gas input plumbing. The chamber was exhausted into a cooled diffuser in the transition section and two heat exchangers. The device was evacuated using two Kinney 850 cfm pumps with two M & D Pneumatic 2700 cfm blowers for a system total of 7,100 cfm. The BCL-16 nozzle (Fig. 2) was positioned on the chamber wall with the gas inputs.

GAS DELIVERY SYSTEMS

The flow systems were entirely stainless steel and nickel because of the corrosive nature of the gases being used such as F_2 or N_2F_4. A safe gas handling system was designed to accommodate the N_2F_4, which has been known to be shock sensitive. The remote operation design was then adapted to the F_2 system. Figure 3 shows the N_2F_4 cylinder storage or coffin. The bottles were opened by remote manual handles behind two $\frac{1}{4}$-in-thick steel barricades and the $\frac{1}{4}$-in-thick steel coffin. A heated charcoal barrel was designed especially for the flow requirements to scrub N_2F_4 from any emergency release of the gas. The barrel is on top of the coffin in Fig. 3. The N_2F_4 scrubber will be described in a separate article because of its unique design. The N_2F_4 coffin was separated from the F_2 coffin by two $\frac{1}{4}$-in-steel plates. The barrier is shown on the left-hand side of the photograph. Figures 4 and 5 are schematics of the F_2 and N_2F_4 flow system. Figure 6 illustrates the H_2/D_2 delivery system. All valves were remotely operated from a copper screened control room. The control room was equipped with its own air recirculating system and O_2 monitor. For safety the F_2 and N_2F_4 systems were Fleak-wired. The Fleak wire is wrapped around the gas lines and connected to a system shutdown interlock. If a small leak occurs, the wire is burnt through and the interlock activated. Hydrogen detectors were installed in the device area in case of leaks in the H_2/D_2 system. All flammable or hazardous flow systems

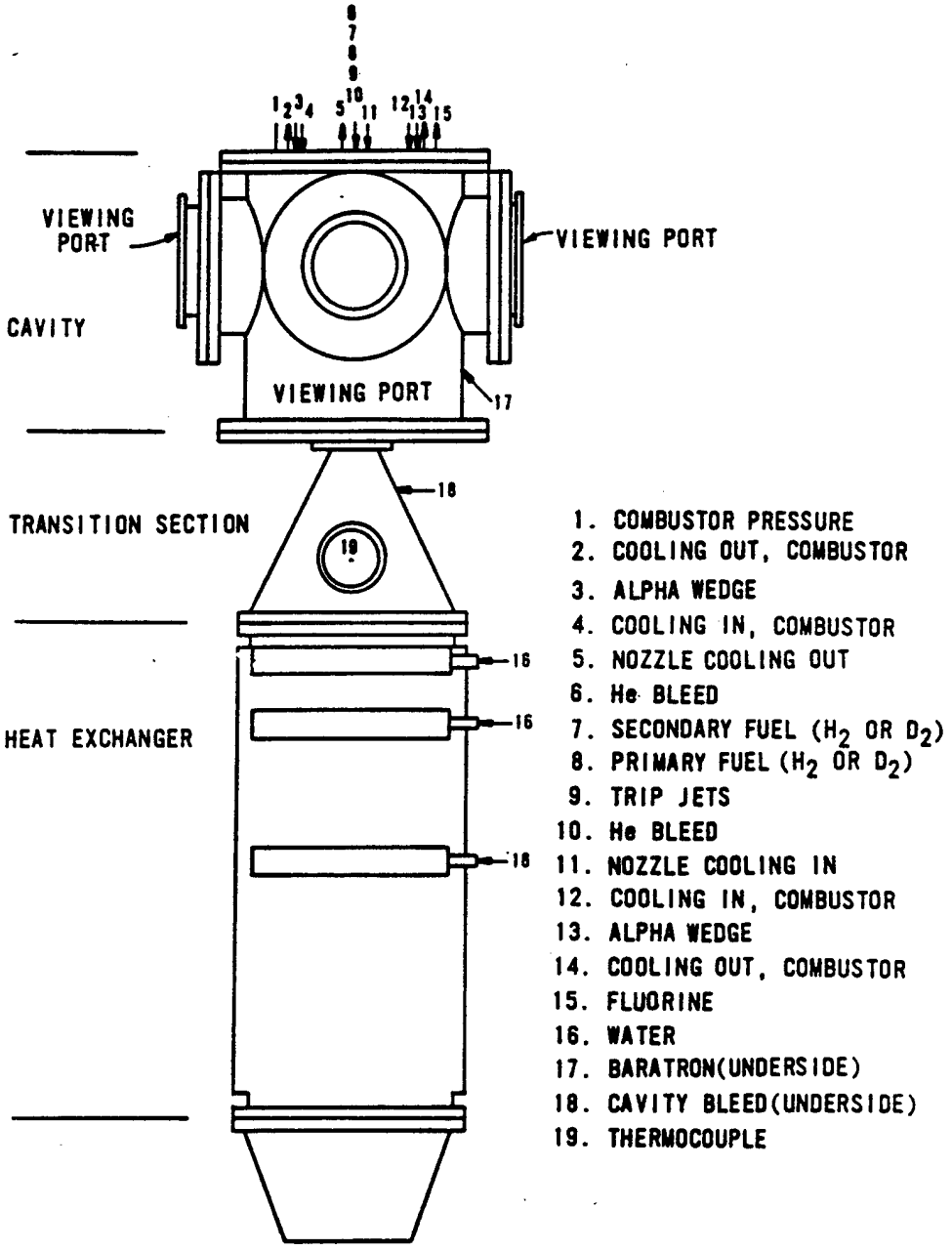

Figure 1. Device schematic.

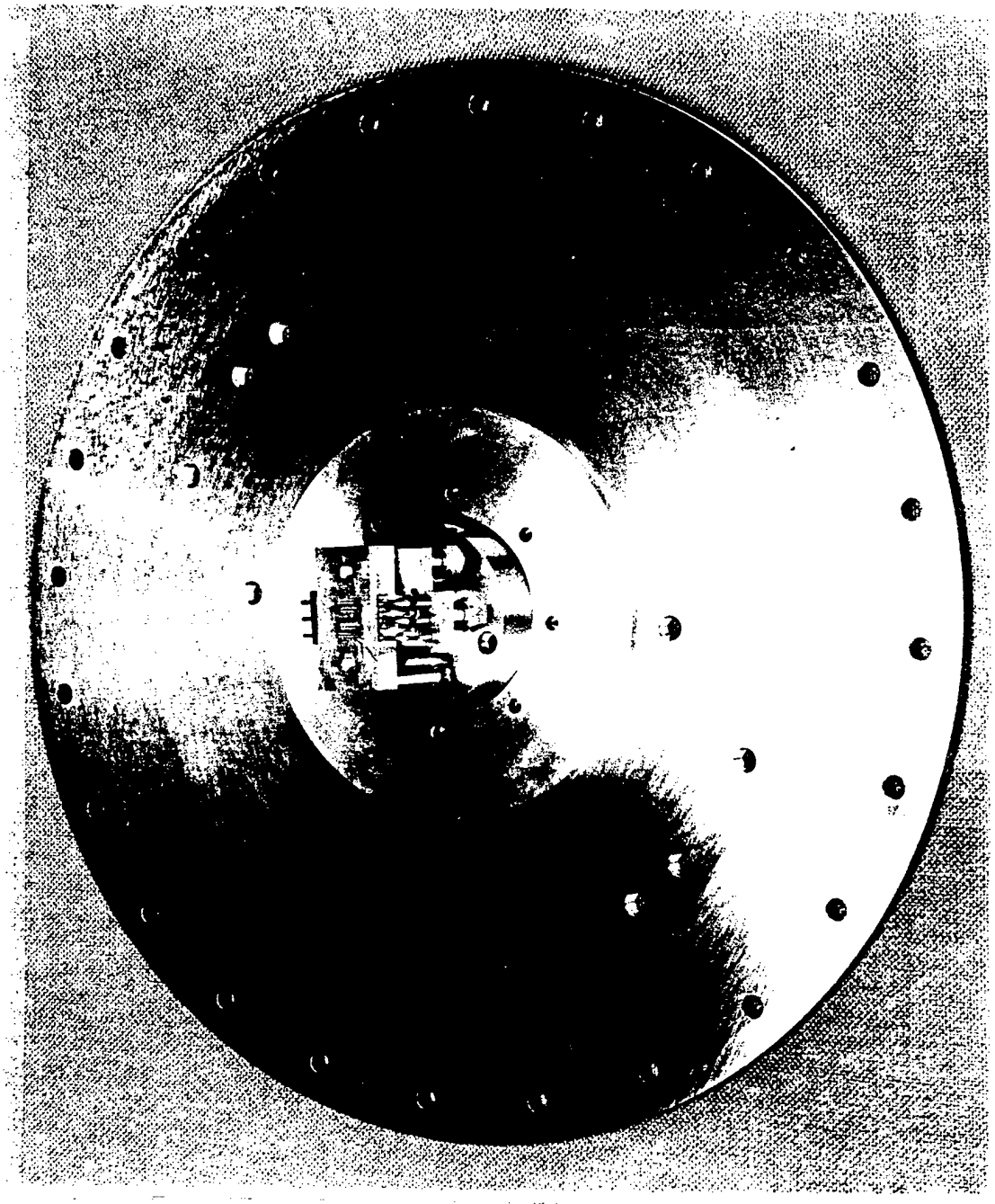

Figure 2. BCL-16 nozzle mounted on back plate of device.

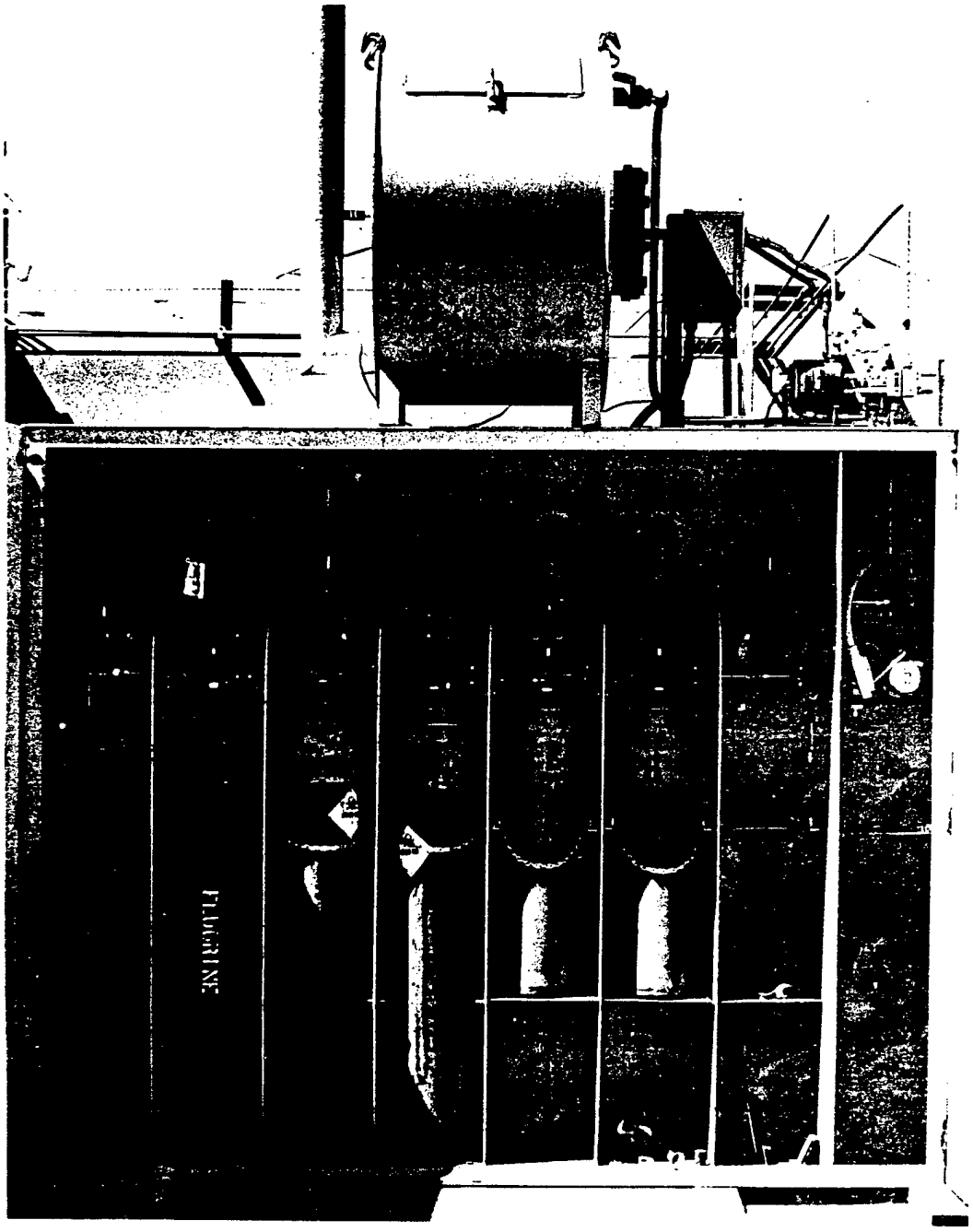

Figure 3. Photograph of the N_2F_4 coffin.

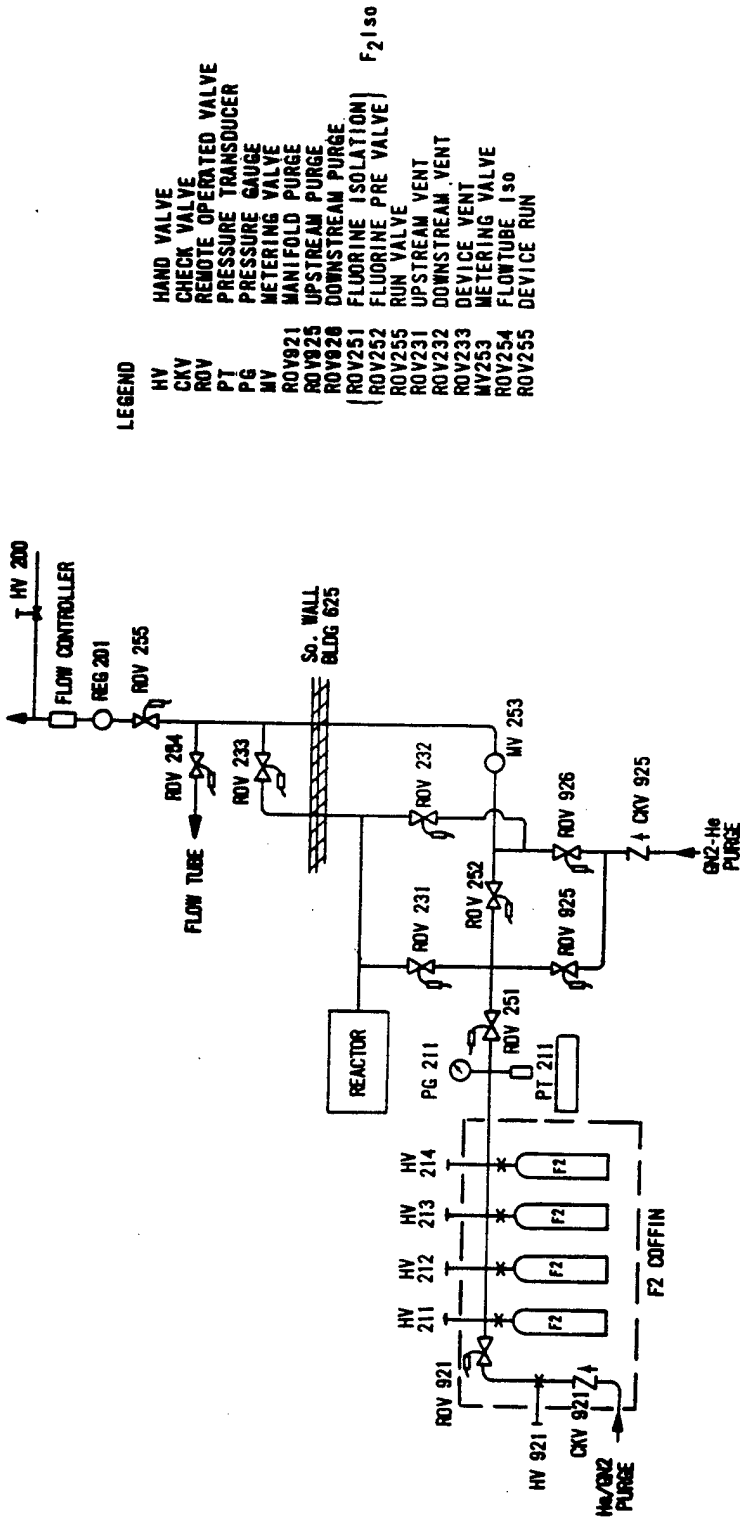

Figure 4. Schematic of the F_2 delivery system.

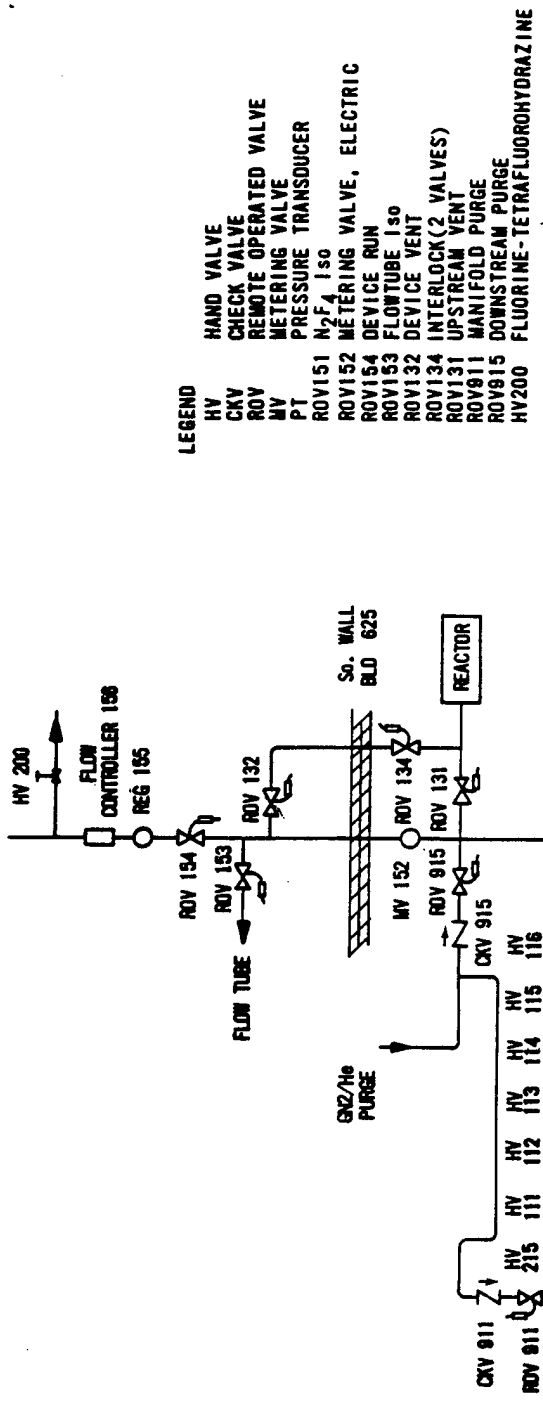

Figure 5. Schematic of the N_2F_4 delivery system.

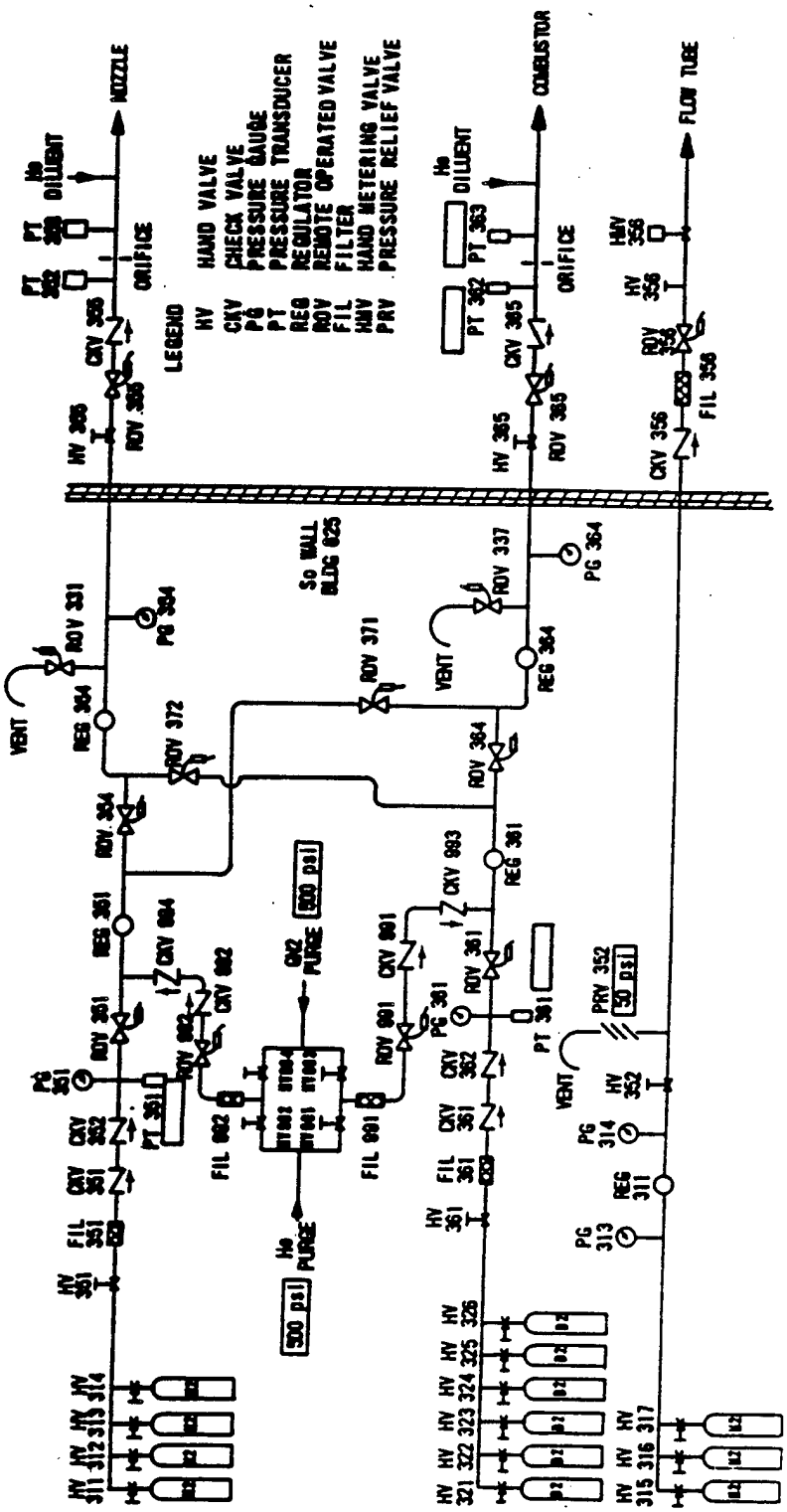

Figure 6. Schematic of the H_2/D_2 delivery system.

were equipped with N_2 and He purge systems. All exhaust from the pumps was passed through a packed quenching tower of 25 m through which water was sprayed. The tower removed any halogenated products remaining in the exhaust.

BCL-16 NOZZLE

The BCL-16 nozzle was developed for HF/DF laser applications (Ref. 7) and studied for those same systems (Ref. 8). For the $N_2F_4 + H_2$ system the combustor portion of the assembly nozzle was operated at design conditions to produce F atoms. The hydrogen or deuterium and fluorine were injected into the combustor along with He diluent at a molar ratio of F_2: D_2: He of approximately 1:2:50.

A one-half cross section of the nozzle is shown in Fig. 7. The nozzle is symmetric in the X-Y plane about the indicated X-axis. The He purge flow, as indicated in Fig. 7, represents the He bleed plate which was an annular injector positioned on the gas input wall of the device. The bleed plate injection was used to confine the nozzle flame and isolate the observation windows from the flame. The BCL-16 contained three secondary nozzles through which either H_2 or D_2 could be mixed with the F atoms arriving through the two primary nozzles. The trip jets were used to mix in the NF_2. The N_2F_4 was thermally dissociated to NF_2 prior to injection via feed lines heated to 200°C.

The combustor portion of the nozzle was N_2-cooled by an external copper collar around the body of the combustor. The internal temperature of the combustor was maintained by using a Ni liner with an air gap between the outer diameter of the liner and inner diameter of the combustor wall. This configuration was determined by extensive testing with LaB_6 and alumina liners. The LaB_6 and alumina liners failed after minimal usage. The result was that debris from the liners plugged the primary nozzles preventing further operation. The Ni Liner lasted over ten test sequences.

A study of the mixing performance of the BCL-16 nozzle was performed under the reactive conditions.

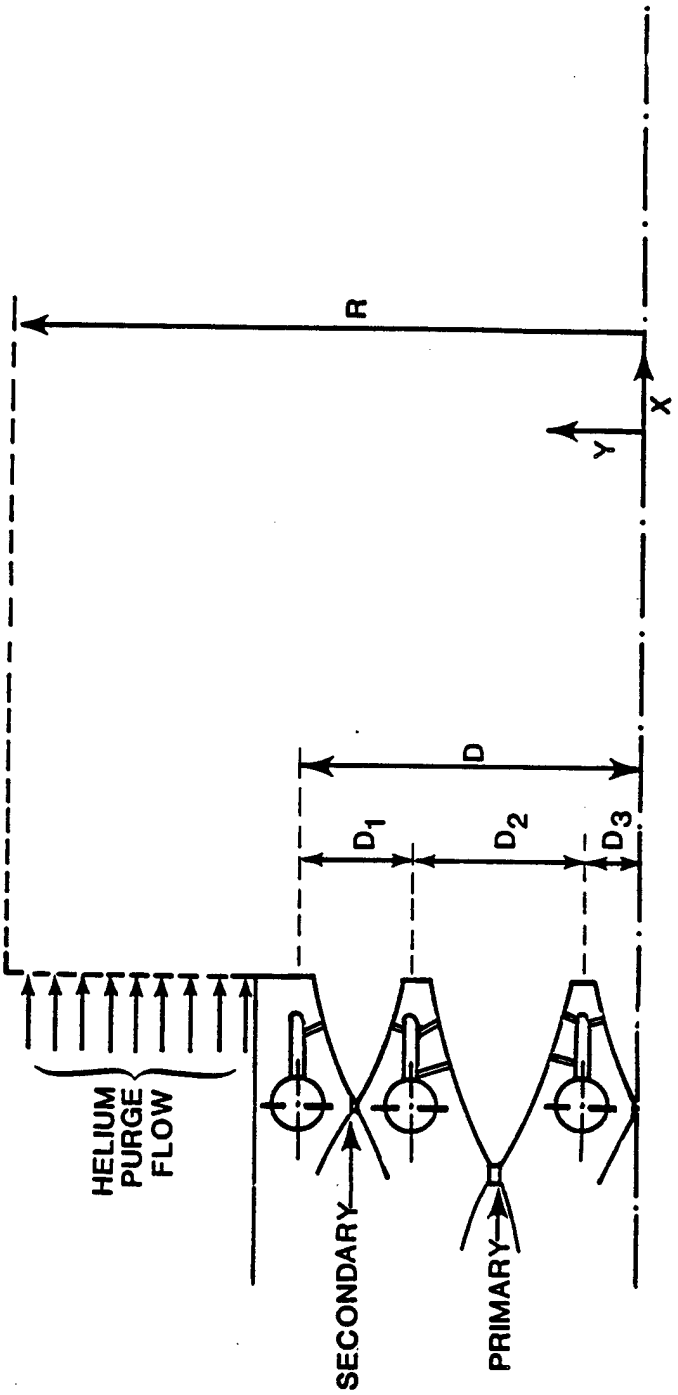

Figure 7. One-half of the BCL-16 nozzle cross-section.

DIAGNOSTICS

$NF(a^1\Delta)$ AND $NF(b^1\Sigma)$ DIAGNOSTICS

The $NF(a^1\Delta)$ diagnostic was an extremely important part of the device performance analysis. The overall arrangement of the NF(a) and NF(b) diagnostics is shown in Fig. 8. The 874.2 nm emission from the NF(a-X) transition was detected via a 38.1-cm-long spatial filter with 0.17-cm-dia orifices coupled to a fused silica fiber optic. The fiber optic was bifurcated so that one end was fed into the NF(b) diagnostic. This allowed simultaneous detection of NF(a) and NF(b) within the same viewing volume. The NF(a) emission was filtered using an extremely narrow bandpass filter (FWHM 0.98 nm) centered at 874.29 nm which essentially eliminated interferences from the close-lying $N_2(B)$ and HF(v=3) emissions. The amount of interference was determined by careful spectroscopic examination of the system under flow conditions using a 0.3 m monochromator with a 1200 l/mm grating blazed at 1.0 μm and examination with an EG&G PAR OMA III system incorporating a 0.25 m polychromator with a 1200 l/mm grating. Overall interferences were determined at $\leq$ 10% at normal flow conditions. Because of the immense amount of emission in the $N_2F_4 + H_2$ system, all bandpass filters were examined for transmission from approximately 250 nm to 1.5 μm. The NF(a) diagnostic used a thermoelectrically cooled RCA C31034A photomultiplier tube (PMT) for detection of the 874.2 nm photons. The diagnostic was calibrated by absolute radiometric methods using a FEL-type quartz halogen tungsten standard lamp (Eppley Laboratory, Inc.).

The NF(b) diagnostic used the same bifurcated fiber optic with the output of the other portion of the cable going to a narrow bandpass filter centered at 531.4 nm and a FWHM of 9.8 nm. The 528.8 nm emission of the $NF(b^1\Sigma - X^3\Sigma)$ transition did not suffer from any interference by close-lying emissions. The 528.8 nm photons passed through the filter to a thermoelectrically-cooled RCA 4837 PMT. Both PMTs were optimized for response by varying the applied voltage and cooling temperature. Optimum voltages are indicated in Fig. 8. Optimum temperature for both units was $-10^{\circ}C$.

Errors for the diagnostics were based upon the extent of interferences from other emissions and calibration errors. The error for the $NF(b^1\Sigma)$

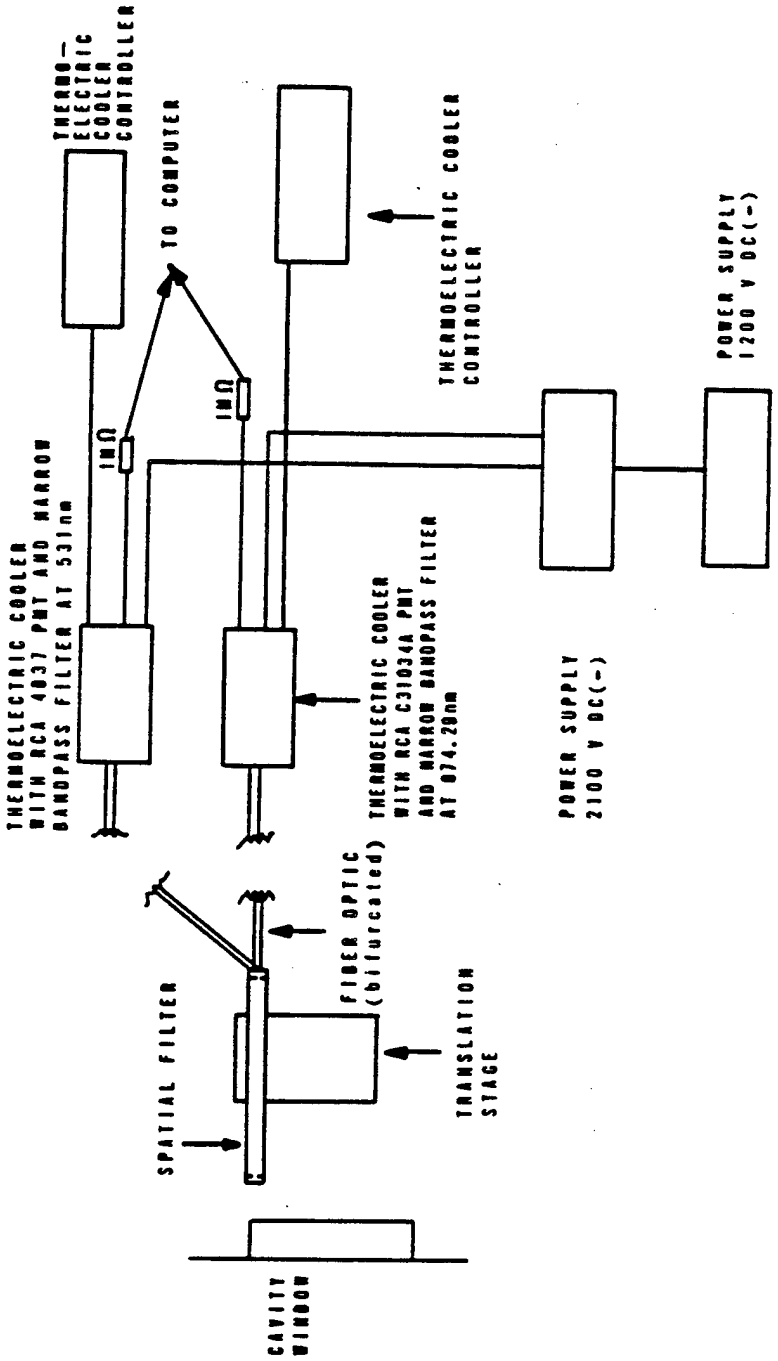

Figure 8. Schematic of the $NF(a^1\Delta)$ and $NF(b^1\Sigma^+)$ diagnostics.

diagnostic was determined to be ±10% with a range of 10^{11} to 10^{13} molecules/cm^3. For the NF($a^1\Delta$) diagnostic, the error was larger due to the interferences from other emissions in the system and was estimated at ± 20% with a range of 10^{14} to 10^{16} molecules/cm^3. Additional error in the NF($a^1\Delta$) may be present due to uncertainty in the lifetime. Current efforts are directed towards remeasuring the branching ration between Eqs. 3 and 4 and determining the NF($a^1\Delta$) lifetime without a branching ratio dependency.

The spatial filter was mounted on a remotely operated translation stage with a linear voltage displacement transducer (LVDT) to accomplish scans across the centerline of the flow field of the device with a known position. Sample scans of the NF($a^1\Delta$) and NF($b^1\Sigma$) emissions are shown in Figs. 9 and 10. The NF($a^1\Delta$) and NF($b^1\Sigma$) diagnostics are described in more detail in Reference 9.

OPTICAL MUTICHANNEL ANALYZER (OMA)

The OMA III 1460R system (EG&G PAR) was used to monitor the change in emission with respect to reagent flow rate over a wide wavelength range (usually 300 - 900 nm) at a fixed point within the device. The OMA III system consisted of a nonintensified diode array head (Model 1412) coupled to a Model 1233 polychromator. The triple grating polychromator was usually operated using the 150 l/mm or 600 l/mm grating. The emission from the device was delivered to the polychromator via a fused silica fiber optic matched to the entrance slit. The system using the 150 l/mm grating had a resolution of 0.6 nm/channel. Using the fiber optic with a spatial filter, the spatial resolution was about 4 cm. This diagnostic was used only to determine volume averaged changes in the excited state production in the device with respect to flow rate changes.

The OMA III was calibrated using the same quartz halogen tungsten standard lamp. Calibrations were performed for each combination of grating and center wavelength selection which were used. Bandpass filters were used for the ultraviolet (UV) and near-UV region calibrations. A sample scan of the operating device is shown in Fig. 11.

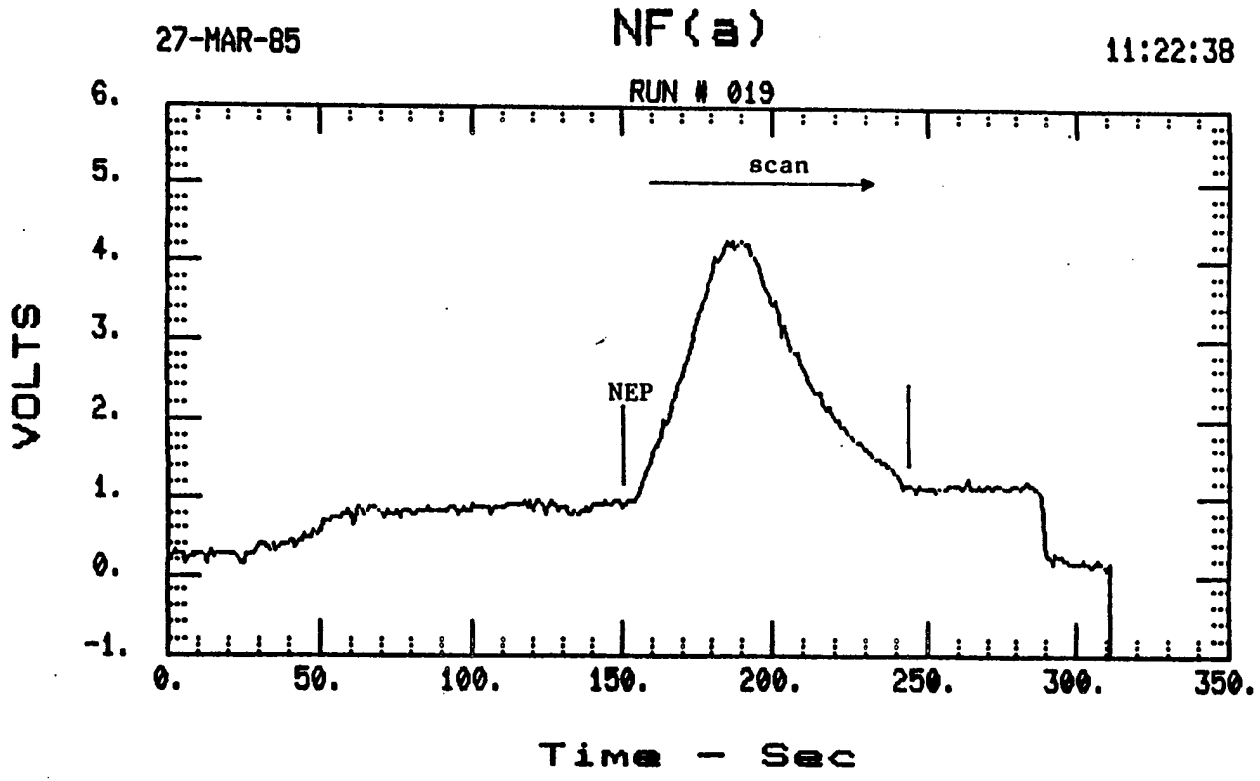

Figure 9. Sample NF($a^1\Delta$) scan.

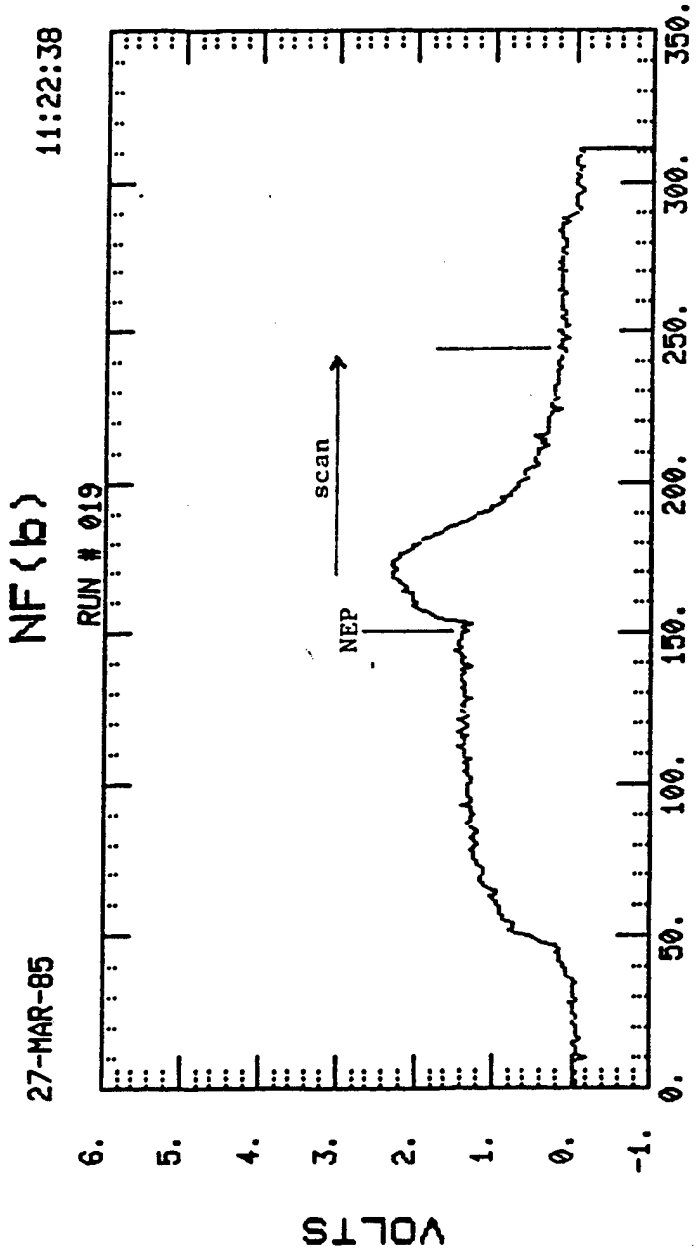

Figure 10. Sample NF($b^1\Sigma^+$) scan.

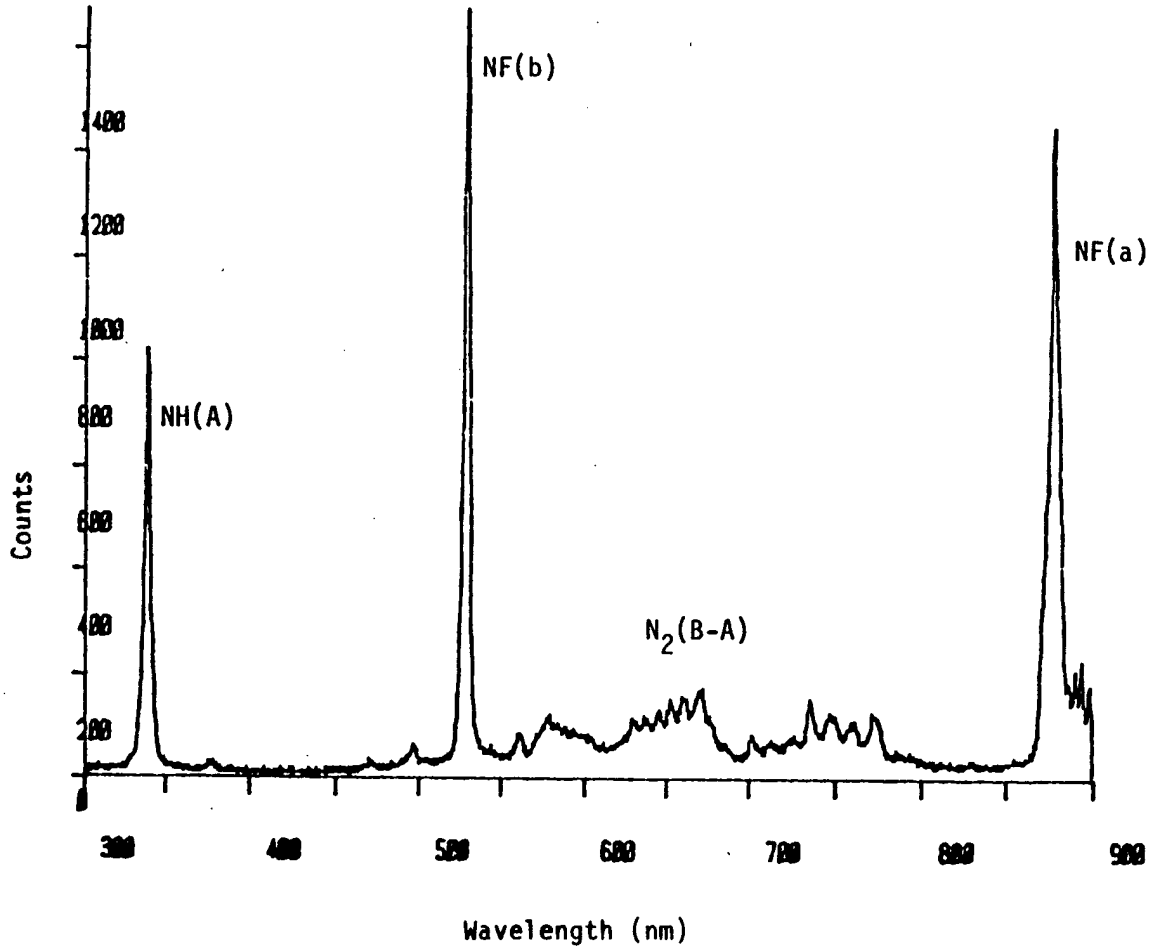

Figure 11. Sample OMA III scan.

N_2F_4 THERMAL DISSOCIATION

A deuterium lamp (Opthos Instruments, Inc.) was used to monitor the NF_2 absorption centered at around 260 nm with a FWHM of about 20 nm (Ref. 10). The absorption cross section was measured using a 25 cm low pressure cell filled with 10 torr* of N_2F_4 at 300 K. The cell was heater-taped and the temperature range monitored from 25 to 200^0 C. Knowing the equilibrium constant for the N_2F_4 dissociation (Refs. 11-13).

$$K_{eq}(T) = 6.3 \times 10^{15} e(-14,129 cal/RT) \text{ cm}^3/\text{mole} \quad (6)$$

and the temperature, the $[NF_2]$ was determined. The cross section was determined to be approximately 10^{-18} cm^2. This compared favorably with the estimate of another group.**

The diagnostic incorporated the D_2 lamp and a fiber optic coupled 0.3 m monochromator (Acton) on opposite sides of the device. The monochromator used a grating blazed at 300 nm with 600 l/mm under a vacuum of 10^{-4} torr. Detection was via a R166 PMT (Hamamatsu). Initial absorption measurements showed that the N_2F_4 was fully dissociated after traversing the heated lines and nozzle within the 0.12 to 0.25 g/s flow regime which was used. This was anticipated from the equilibrium as given in Eq. 6. Further use of this diagnostic was not required once the dissociation had been confirmed.

HNO* DIAGNOSTIC

In an effort to approximately determine the H atom production in the device, NO was injected through the trip jets in place of the N_2F_4. The H atom reaction with NO produces excited state HNO indicated by red emission at

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* 1 torr = 1.33 x 10^2 pascals

** Private communication - R. F. Heidner, The Aerospace Corp., Los Angeles, CA, Dec 1984.

760 nm (Ref. 14). The emission was detected via the arrangement shown in Fig. 12. The diagnostic was similar to the $NF(a^1\Delta)$ diagnostic. The HNO^* diagnostic incorporated a narrow bandpass filter centered at 748.5 nm, FWHM 95 nm and peak transmission of 0.63. The diagnostic was calibrated using the FEL-type lamp as described in the $NF(a^1\Delta)$ diagnostic section with an error of $\pm$15%. Using a titration method in a flow system involves several other sources of error. Chiefly, the extent of mixing of the reagent streams is critical.

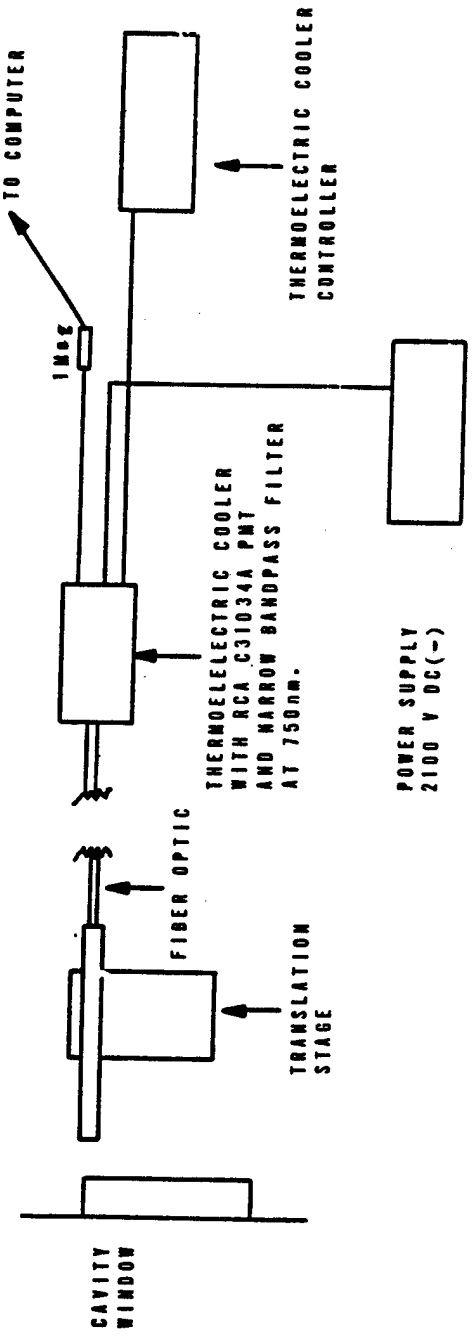


Figure 12. Schematic of the HNO* diagnostic.

PARAMETRIC STUDIES

The purpose of the device study was to determine the optimum flow conditions for $NF(a^1\Delta)$ production and minimized $NF(b^1\Sigma)$. The first portion of the optimization was to determine the operating conditions for maximum F atom production which, in turn, would yield the maximum H atom production. Since F atom determination has been difficult, the approximate H atom concentration was approached via gas phase titration. The NO was injected through the trip jets at increasing flow rate while the HNO^* emission was monitored. A sample scan of the HNO^* diagnostic is shown in Fig. 13. A clear end point was not determined because of the supersonic flow and mixing regime. A minimum H atom production was estimated at 10^{15} molecules/cm^3 due to the mixing concerns. This achieved the goal of determining if efficient H atom production was occurring; therefore, N_2F_4 injection was then tried. Based upon the minimum H atom concentration and Eq. 3, it was anticipated that at least 10^{15} molecules/cm^3 of $NF(a^1\Delta)$ could be produced. Parametric studies involved first setting the combustor at conditions for F_2 production as determined by computer modeling using an HF/DF laser combustion code and then varying flows around the set point. The H_2 and N_2F_4 flow rates were initially set by ALPHA computer code predictions and varied about the set point. Little effect was seen in $NF(a^1\Delta)$ production when combustor flows (D_2,F_2 and diluent He) were varied. To summarize the results of several sets of tests, Figures 14-19 show the correlation of $NF(a^1\Delta)$ and $NF(b^1\Sigma)$ densities to flow rates of the various gases. The variation of $NF(a^1\Delta)$ concentration with NF_2 flow rate was the most marked. The He bleed plate variation simply had the effect of compressing the reactive flow into a smaller volume thus increasing the local number density but not the yield.

Through multiple tests the combustor, then the secondary H_2 (or D_2) and NF_2 flow rates were optimized. Table 1 gives a summary of several sets of tests using H_2 as the secondary gas. The maximum $NF(a^1\Delta)$ concentration obtained with H_2 was $\sim 5 \times 10^{15}$ molecules/cm^3. Some testing was performed using D_2 as the secondary gas. The major effect of using secondary D_2 was the decrease in $NF(b^1\Sigma)$ production. The maximum $NF(a^1\Delta)$ concentration with D_2 was $\sim 7 \times 10^{16}$ molecules/cm^3. Yields based upon the initial NF_2 revealed that

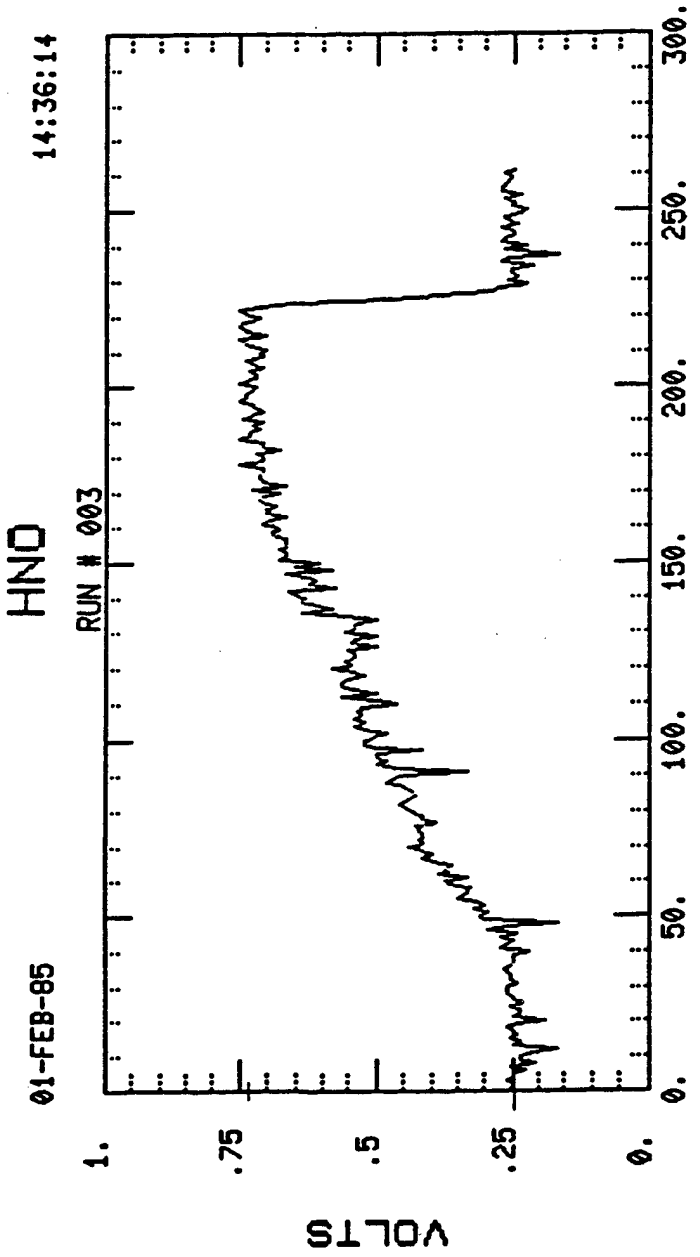


Figure 13. Sample HNO* scan.

TABLE 1. SAMPLE TEST DATA USING H_2 IN SECONDARY NOZZLES

TEST No.	F_2,25% in He (g/s)	Combustor D_2 (g/s)	Secondary H_2 (g/s)	Secondary diluent He (g/s)	Trip Jet N_2F_4 (g/s)	Pcombustor (PSIA)	Pcavity (Torr)	NF(a) (molecules/ cm) Peak	NF(b) (molecules) cm) Peak
18-1	0.10	0.002	0.0028	0.01	0.27	8.0	10.0	5.2×10^{14}	1.9×10^{11}
18-2	0.15	0.002	0.0024	0.01	0.26	10.0	10.0	1.41×10^{15}	6.1×10^{12}
18-4	0.20	0.005	0.0072	0.02	0.25	15.0	12.0	2.8×10^{15}	1.5×10^{12}
23-1	0.14	0.006	0.03	none	0.16	9.0	10.0	4.2×10^{13}	6.0×10^{11}
24-7	0.16	0.0063	0.01	none	0.25	9.2	9.38	7.6×10^{15}	4.7×10^{12}

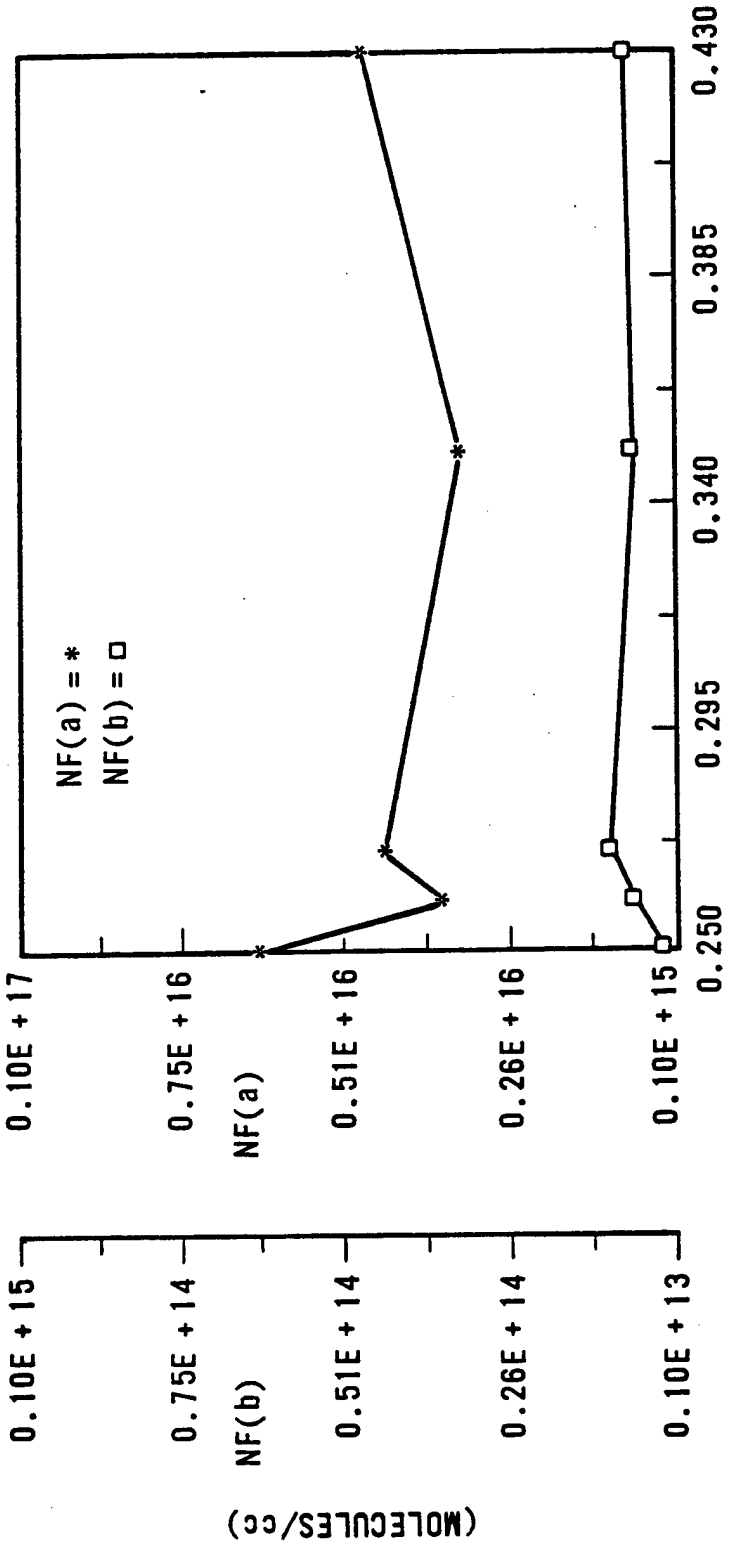


Figure 14. Variation of NF(a) and NF(b) with D_2 combustor flow.

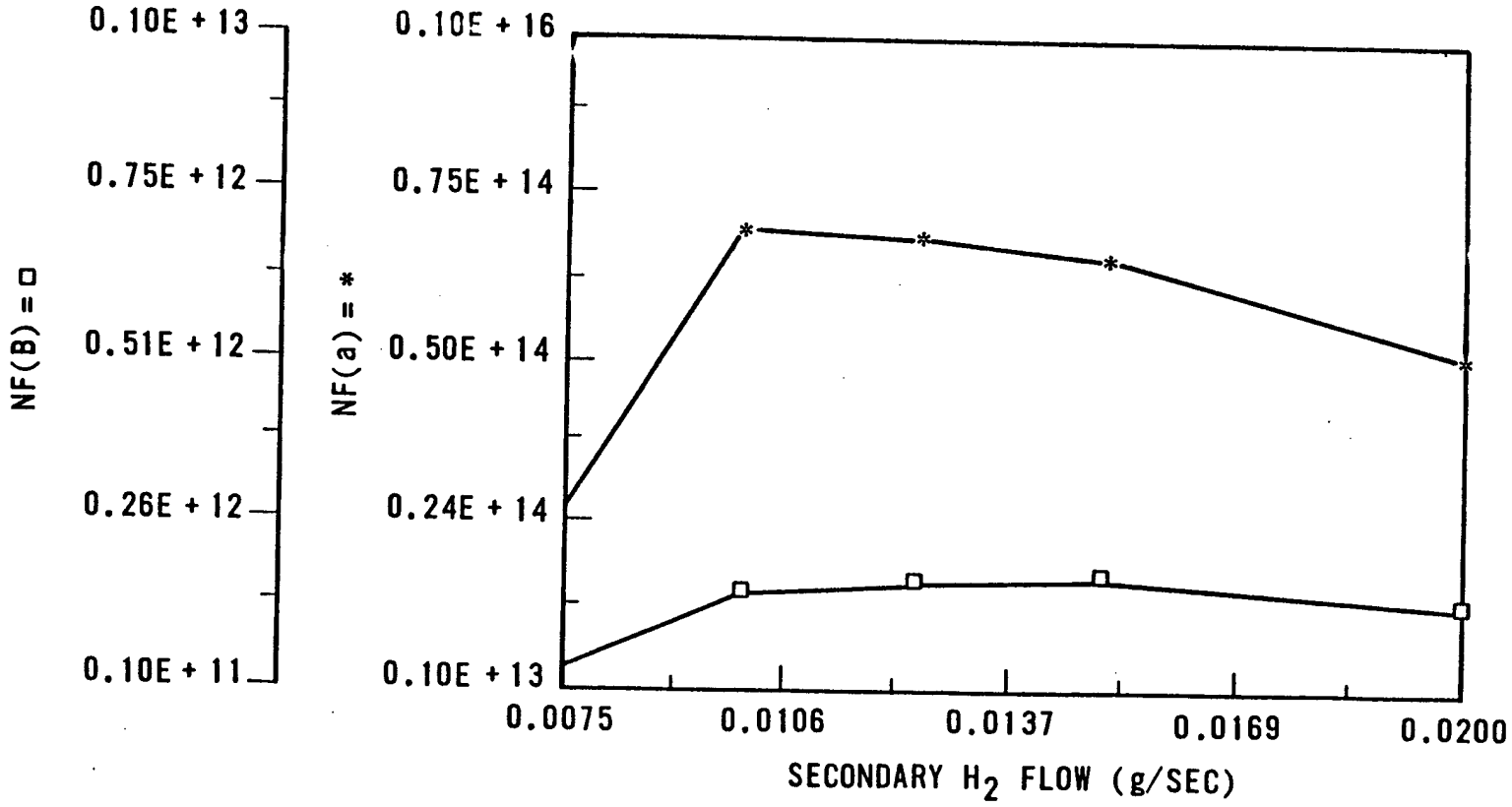


Figure 15. Variation of NF(a) and NF(b) with secondary H_2 flow rate.

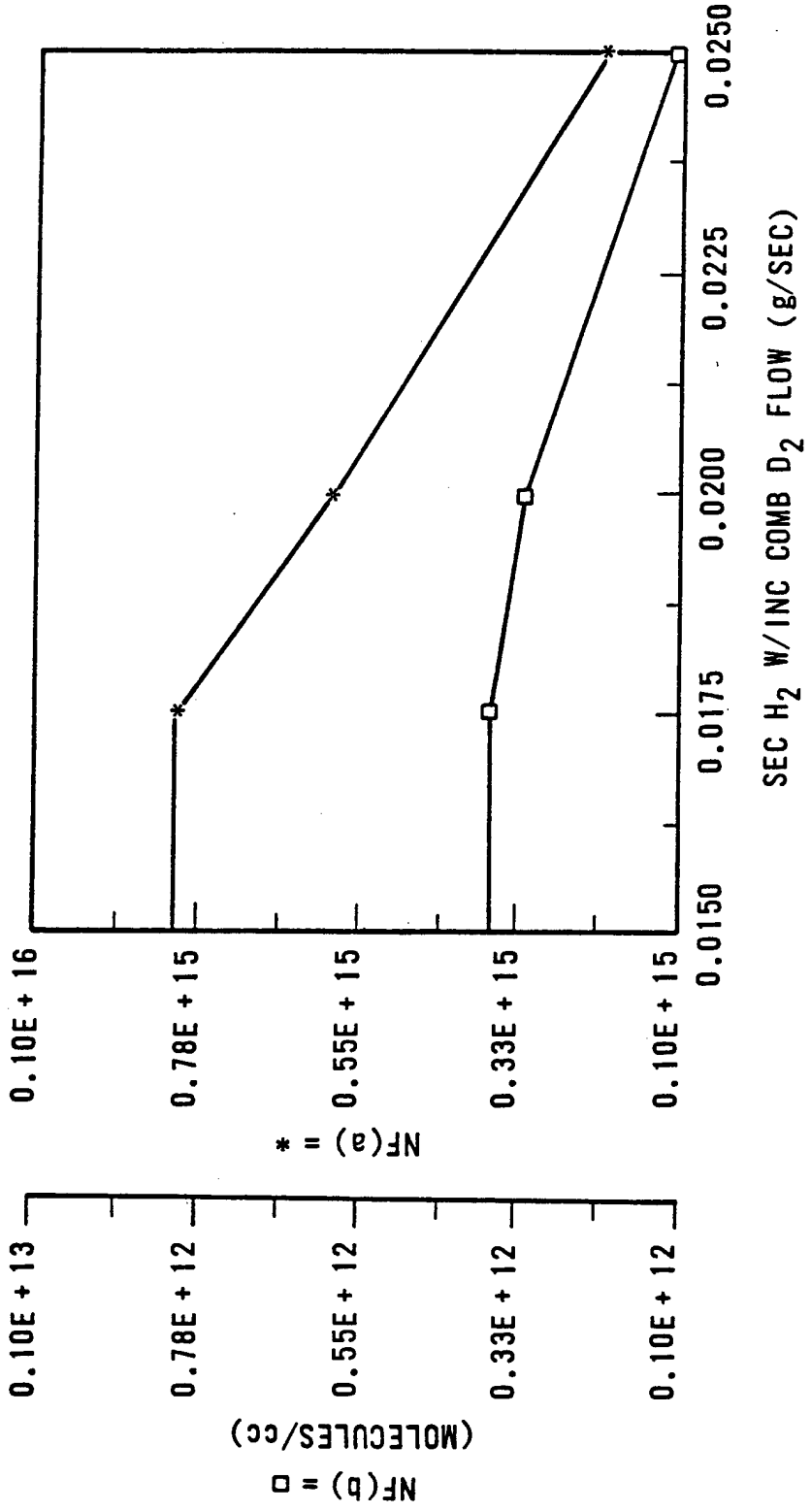


Figure 16. Variation of NF(a) and NF(b) with secondary H_2 at higher combustor D_2 flows.

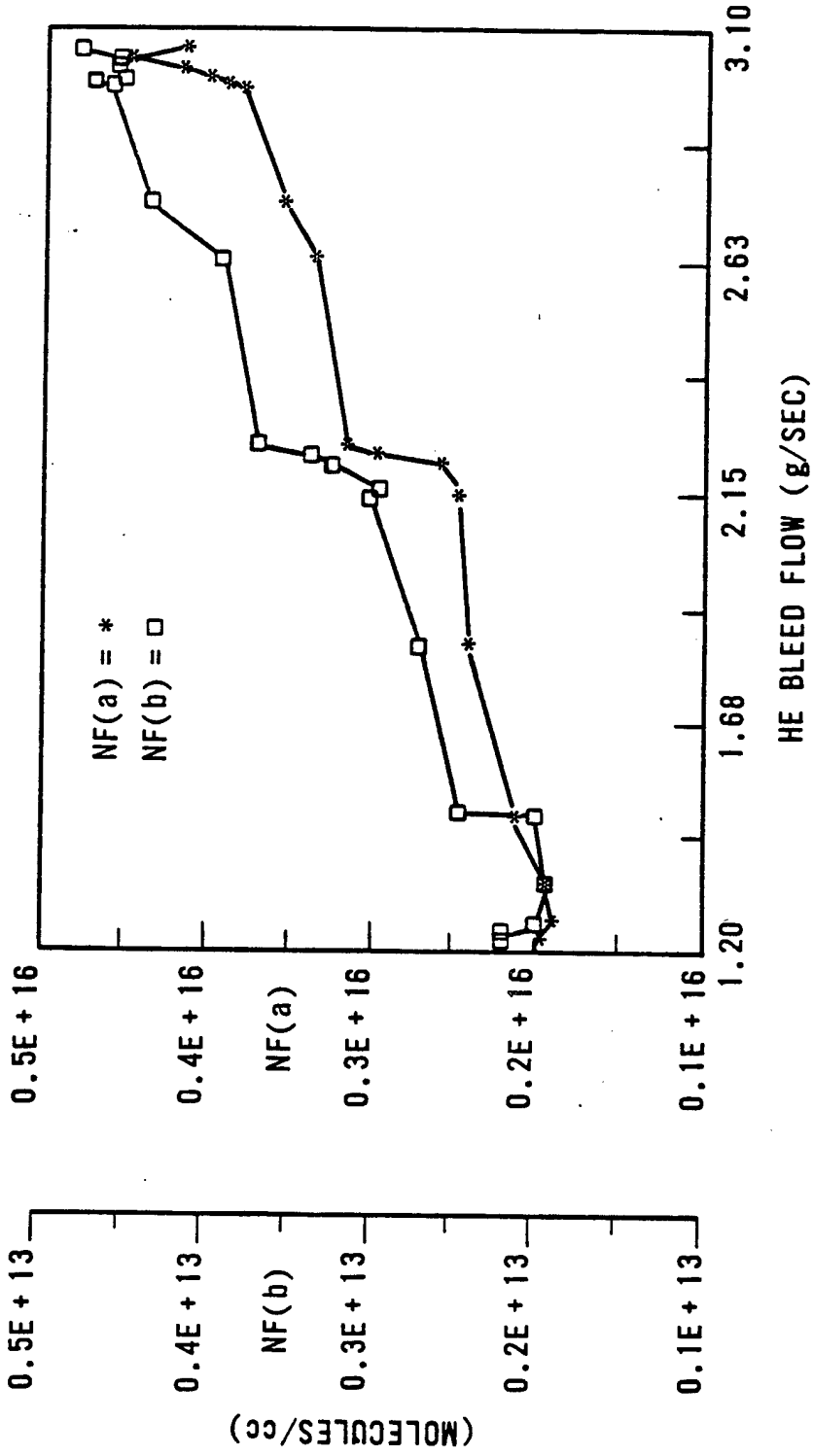


Figure 17. NF(a) and NF(b) variation with He bleed plate flow.

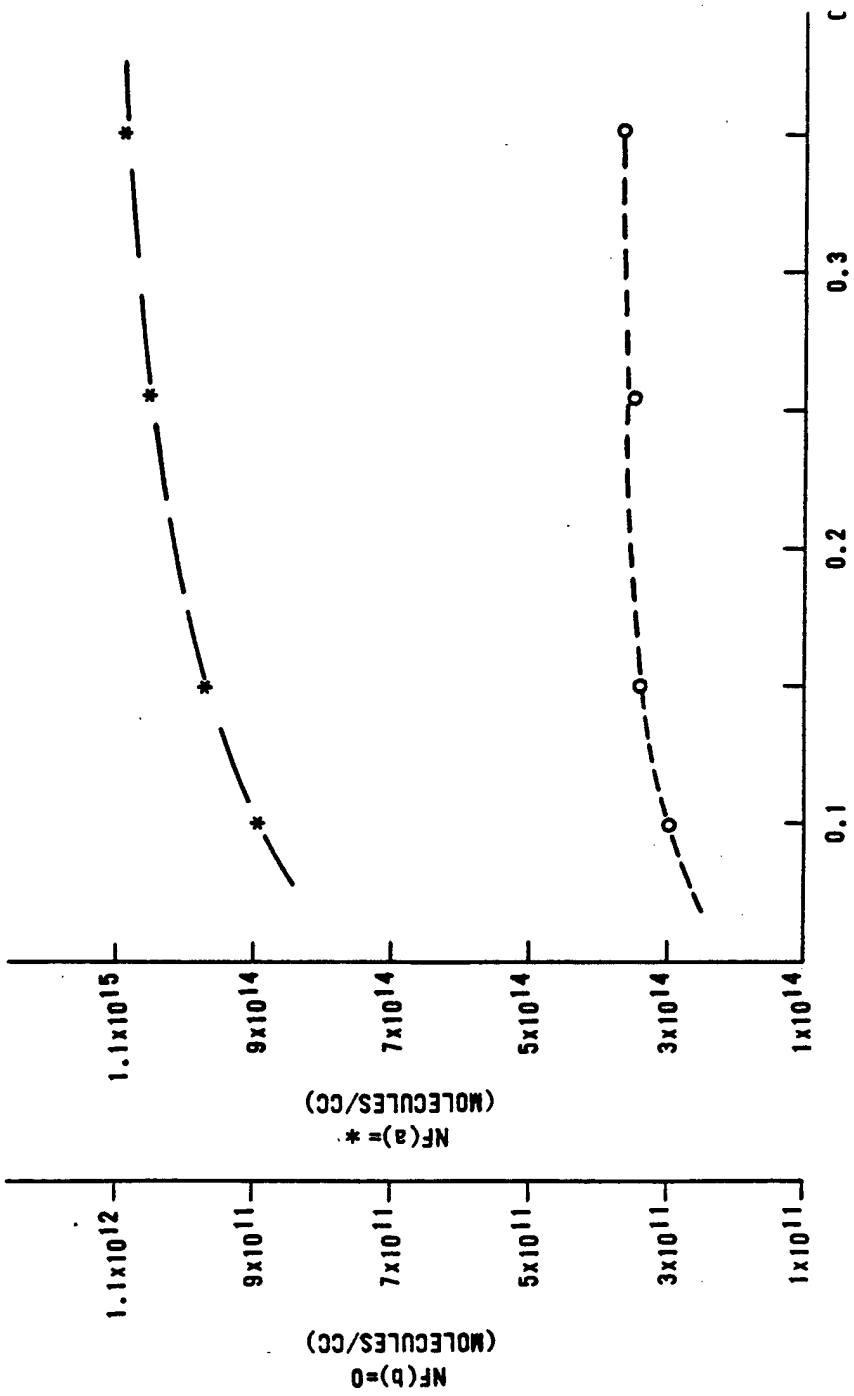


Figure 18. NF(a) and NF(b) variation with N_2F_4 flow.

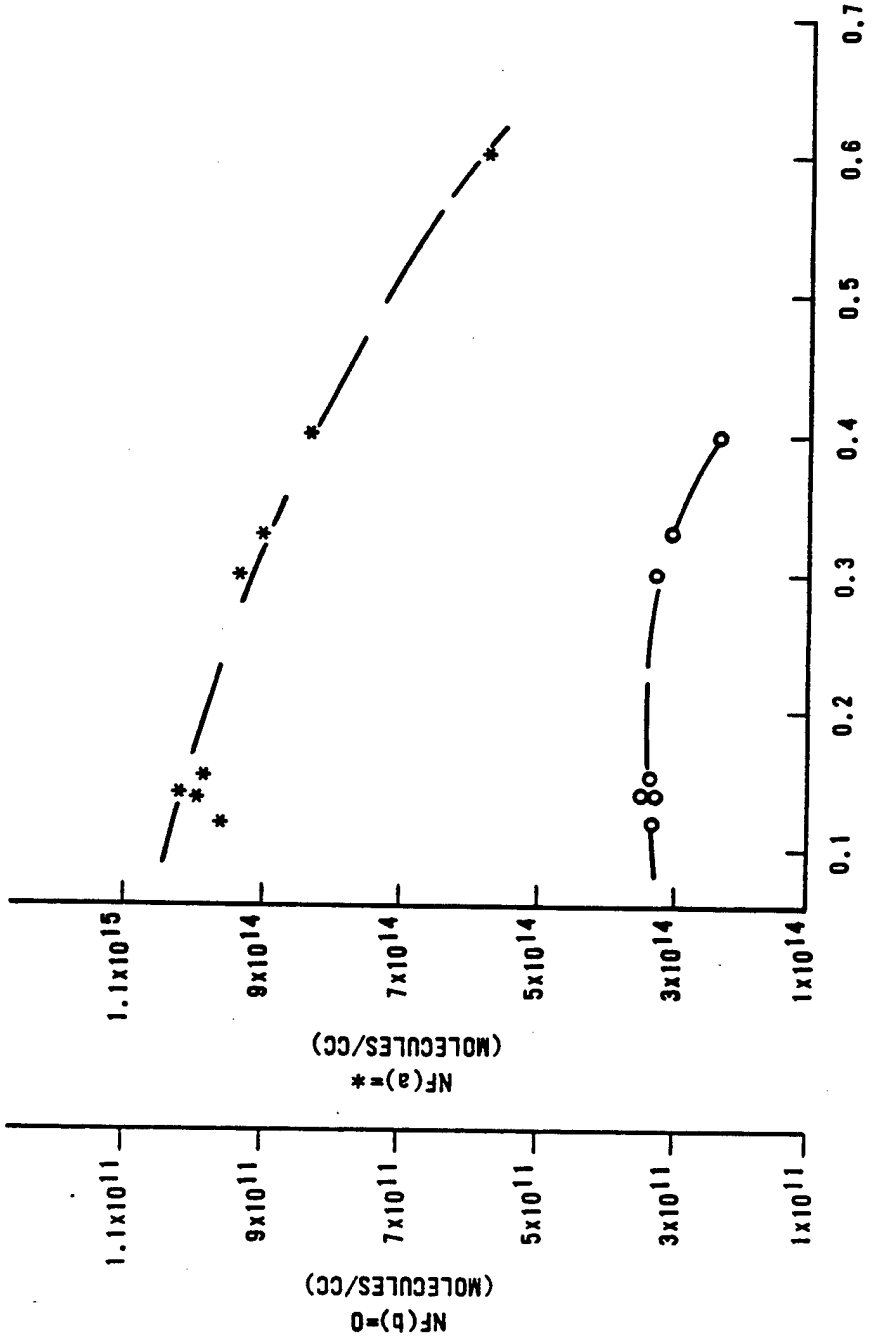


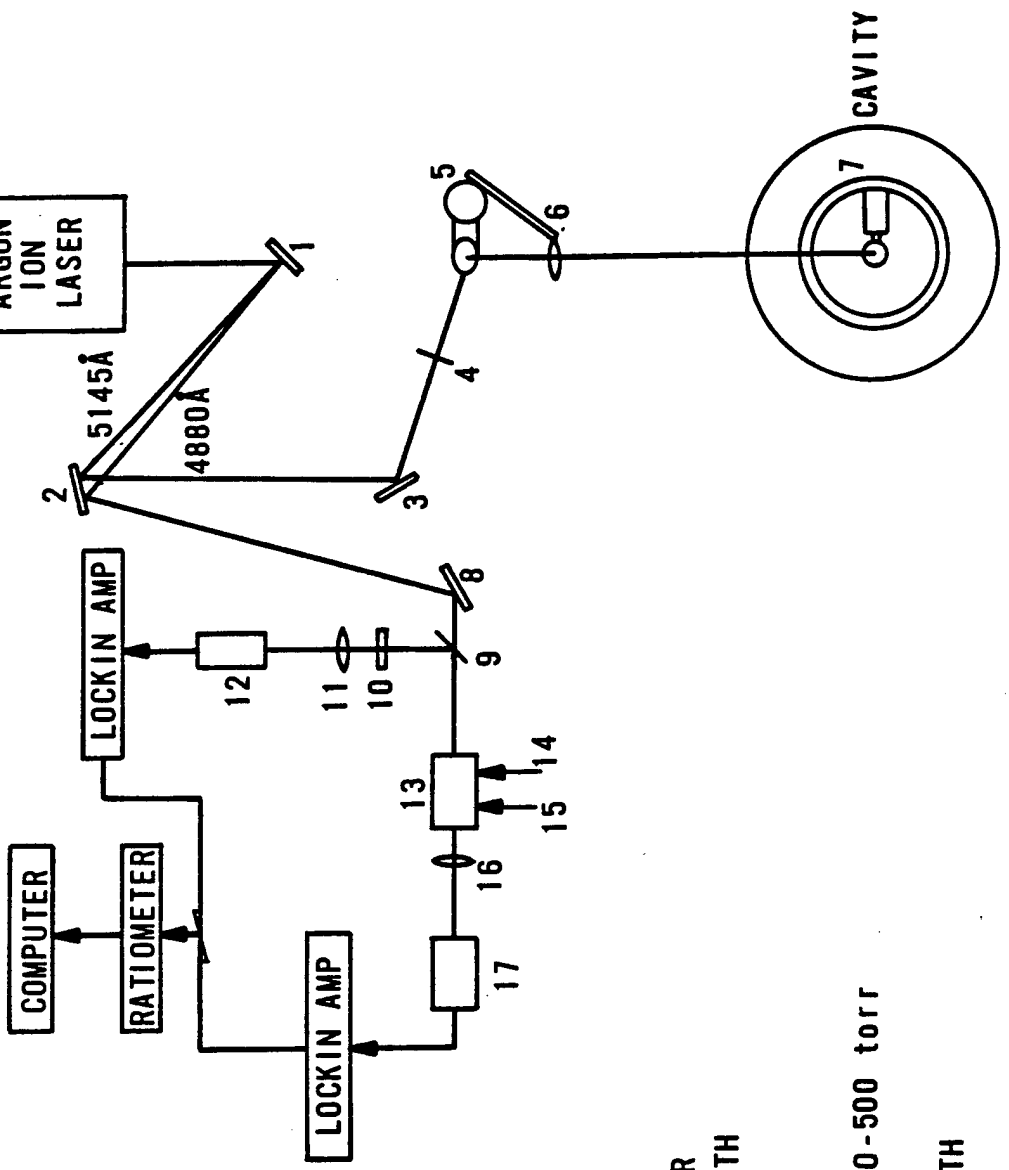
Figure 19. Variation of NF(a) and NF(b) with F_2 flow rate.

instead of the predicted 90 percent based upon early flow tube studies (Refs. 2,4), 35 percent yields were obtained. The yields were calculated based upon initial NF_2 concentrations assuming 100 percent disassociation of N_2F_4. The high concentration achievable in this system indicates that $NF(a^1\Delta)$ may be a viable candidate for pumping other molecules for use in chemical lasers.

The modelling results of Plummer (Ref. 15) using the JETMIX code predicts a maximum NF(a) population of 1×10^{15} molecules/cm^3 for Test 18-4. The shape of the profile does not compare; however, the consistent results support the test results. The modelling result levels out at a high population within 4 cm of the nozzle. However, experimental profiles rise and then drop off as shown in Fig. 9. The reason for this discrepancy is not clear and could be due to either a fluid dynamic or kinetic problem with the model.

NOZZLE MIXING STUDIES

To understand the fundamental mixing in the BCL-16 nozzle, refer to Reference 8. Since a totally different chemical reaction scheme is used here, a laser-induced fluorescence (LIF) study was employed using I_2 carried by He or N_2 and injected into the primary, secondary, and trip jet nozzles separately. While the I_2 flow was on, a sheet of 514.5nm Ar^+ laser light was scanned across the flow field. The fluorescence from the I_2 (B-X) emission was recorded via a fast camera system and digitized. The experimental arrangement is shown in Fig. 20. The digitized information was used to reconstruct the flow field showing the overlap of the nozzles. The overlap should be indicative of the mixed areas. Figure 21 shows a sample composite across a portion of the flow field indicating incomplete mixing using the BCL-16 nozzle. The technique has been described previously in reference 8 and 16.



1. GRATING
2. MIRROR
3. MIRROR
4. IRIS DIAPHRAGM
5. BEAM STEERER
6. LENS; 1m FOCAL LENGTH
7. RASTER MIRRORS
8. MIRROR
9. BEAM SPLITTER
10. NEUTRAL DENSITY FILTER
11. LENS, ~7cm FOCAL LENGTH
12. Ge PHOTODIODE
13. I_2 CELL
14. PRESSURE TRANSDUCER, 0-500 torr
15. THERMOCOUPLE
16. LENS, ~7cm FOCAL LENGTH
17. Ge PHOTODIODE

Figure 20. Schematic of the LIF experimental apparatus.

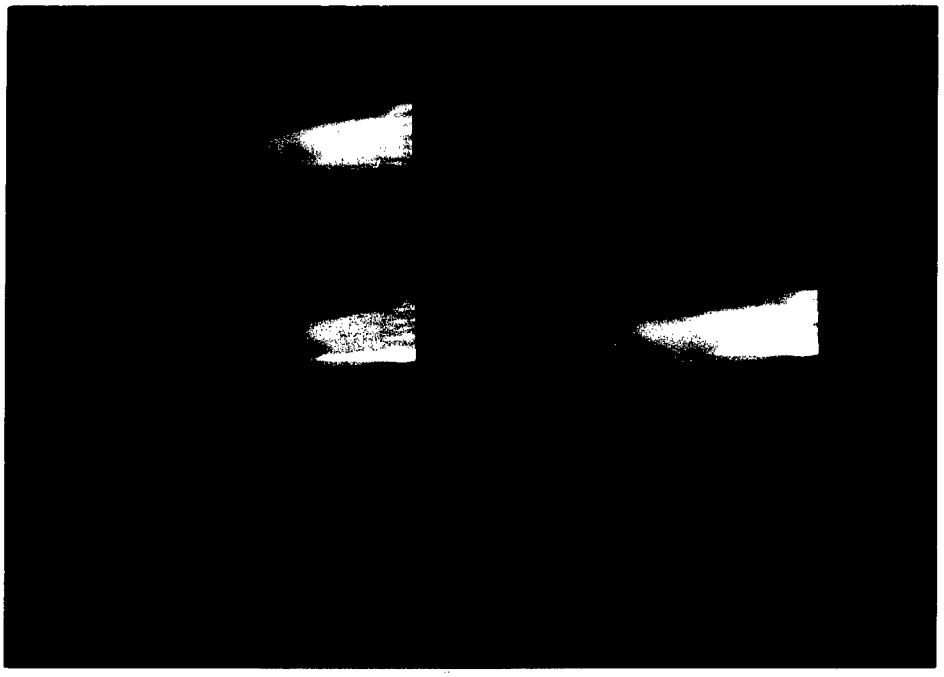


Figure 21. Composite of trip, secondary and primary jets using LIF.

CONCLUSIONS

The $NF(a^1\Delta)$ concentrations produced using the BCL-16 nozzle and the $NF_2 + D_2(H_2)$ system are at sufficient levels to be used in energy transfer studies. The actual production level may be improved by designing a nozzle to mix the heavy NF_2 into the relatively light D_2 stream. It is not clear at this point whether the $NF(a^1\Delta)$ branching ratio originally used is incorrect, or if the lower than expected yields are strictly due to mixing. A combination of the two effects may be the actual condition. The branching ratio must be remeasured in an accurate method to adequately decouple the two effects and solve the problem.

The other concern is that N_2F_4 is of limited supply. Preliminary efforts to use NF_3 have shown that this may be a viable alternative either by combustion with D_2 or by addition through the trip jets. Barring the lack of NF_2 from N_2F_4, the $NF(a^1\Delta)$ production via $NF_3 + H_2/D_2$ is a successful method and should be implemented to pump other molecules such as BiF to create energy inversions.

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