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NWC ltr 23 Jan 1979

NWC TP 5938

Hydroxyl Radical Chemical Laser

by
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for the
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NOVEMBER 1977

Naval Weapons Center
CHINA LAKE, CALIFORNIA 93555

Naval Weapons Center

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FOREWORD

This document is the final report on Contract N00123-75-C-0685. The work was performed at Calspan Corporation with Dr. J. W. Raymonda as the Principal Investigator for all of the Phase One work. Dr. J. W. Daiber was responsible for the design, fabrication, and testing of the transverse laser device. The testing portion of the Phase Two program was carried out with the added assistance of Mr. H. M. Thompson.

This final report presents results of an experimental investigation conducted from July 1975 to February 1977 to demonstrate continuous wave lasing in chemically excited OH. The work was sponsored by the Naval Weapons Center (NWC), China Lake, California, under Navy Contract N00123-75-C-0685 and supported by the Naval Air Systems Command under AirTask A350-3500/008B/6F32-382-502.

Dr. David Fenneman was the Navy Technical Coordinator and has reviewed this report for technical accuracy.

This report is released for information at the working level and does not necessarily reflect the views of NWC.

Approved by
BURRELL HAYS, *Head*
Ordnance Systems Department
6 October 1977

Under authority of
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RAdm., U.S. Navy
Commander

Released for publication by
R. M. HILLYER
Technical Director (Acting)

NWC Technical Publication 5938

Published by . . . . . Technical Information Department
Collation . . . . . Cover, 18 leaves
First printing . . . . . 230 unnumbered copies

UNCLASSIFIED

SECURITY CLASSIFICATION OF THIS PAGE *(When Data Entered)*

REPORT DOCUMENTATION PAGE

READ INSTRUCTIONS BEFORE COMPLETING FORM

1. REPORT NUMBER: NWC TP-5938	2. GOVT ACCESSION NO.
3. RECIPIENT'S CATALOG NUMBER	
4. TITLE *(and Subtitle)*: HYDROXYL RADICAL CHEMICAL LASER	5. TYPE OF REPORT & PERIOD COVERED: Final Report, 2 July 1975-2 Feb 1977
6. PERFORMING ORG. REPORT NUMBER	
7. AUTHOR(s): J. W. Daiber, J. W. Raymonda, H. M. Thompson	8. CONTRACT OR GRANT NUMBER(s): CALSPAN WG-6096-A-1; N00123-75-C-0685
9. PERFORMING ORGANIZATION NAME AND ADDRESS: Calspan Corporation, P.O. Box 235, Buffalo, New York 14221	10. PROGRAM ELEMENT, PROJECT, TASK AREA & WORK UNIT NUMBERS: AirTask A350-3500/008B/6F32-382502; WF32382502
11. CONTROLLING OFFICE NAME AND ADDRESS: Naval Weapons Center, China Lake, California 93555	12. REPORT DATE: November 1977
	13. NUMBER OF PAGES: 12 327
14. MONITORING AGENCY NAME & ADDRESS *(if different from Controlling Office)*	15. SECURITY CLASS. *(of this report)*: UNCLASSIFIED
	15a. DECLASSIFICATION/DOWNGRADING SCHEDULE

16. DISTRIBUTION STATEMENT *(of this Report)*

Distribution limited to U.S. Government agencies only; test and evaluation; 1 November 1977. Other requests for this document must be referred to the Naval Weapons Center.

17. DISTRIBUTION STATEMENT *(of the abstract entered in Block 20, if different from Report)*

18. SUPPLEMENTARY NOTES

19. KEY WORDS *(Continue on reverse side if necessary and identify by block number)*

Hydroxyl
Ozone
H-atom
Laser
Chemical Laser

20. ABSTRACT *(Continue on reverse side if necessary and identify by block number)*

See reverse side of this form.

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(U) *Hydroxyl Radical Chemical Laser*, by J. W. Daiber, J. W. Raymonda, and H. M. Thompson, Calspan Corporation. China Lake, Calif., Naval Weapons Center, November 1977, 34 pp. (NWC TP 5938, publication UNCLASSIFIED.)

(U) The reaction of H-atoms with O_3 is known to result in the product molecule OH being in the ninth vibrational level. Such a high level of chemiexcitation could result in chemical laser action. The motivation for developing such a laser is: (1) the wavelength at which lasing would occur has good atmospheric transmission, and (2) the reactants and products can be handled safely. In the present program, techniques were developed for generating large fluxes of H-atoms and O_3-molecules. An apparatus was constructed for the demonstration of lasing; however, full testing could not be completed in this program. Lasing was not achieved.

CONTENTS

INTRODUCTION

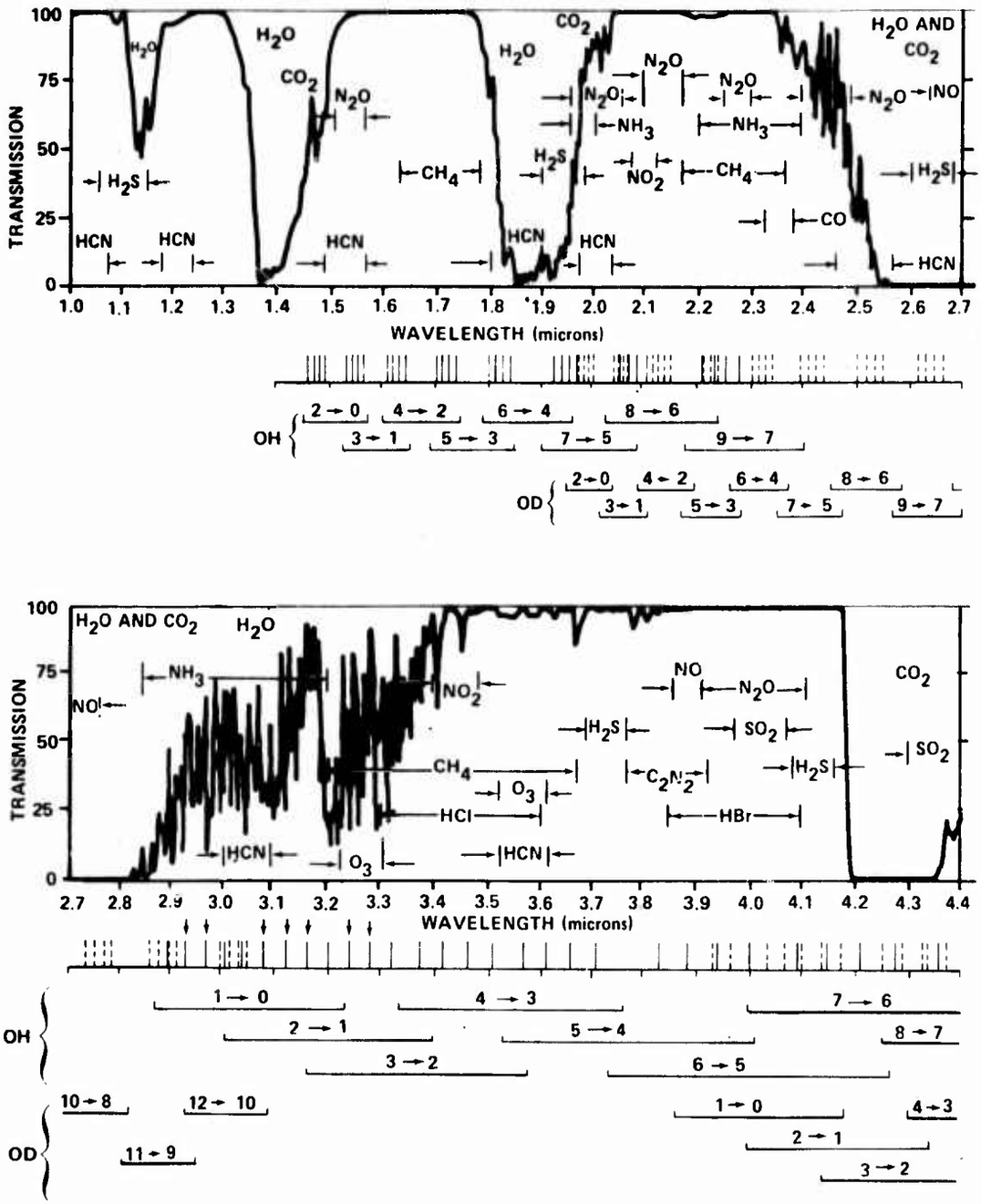
The objective of the program reported herein was to obtain continuous wave lasing on either fundamental or first-overtone vibration-rotation transitions of the hydroxyl radical. A secondary objective was to determine the effect of added oxygen on hydroxyl emission spectra and lasing in order to assess the potential for operating a hydroxyl laser on the ozone-oxygen mixture from an ozonator. If this mode of operation were possible, the need for carrying and storing ozone would be eliminated and a considerable systems advantage would be realized.

The first overtone vibration-rotation transitions of the hydroxyl radical span several wavelength regions in which the atmosphere possesses high transmission, namely the regions around 1.5 μm to 1.75 μm where OH overtone lines occur and 2.05 μm to 2.35 μm, where the OD overtone lines occur. Figure 1 is a plot of the spectral transmissivity of the atmosphere. The location of the various hydroxyl bands are also indicated on this figure. As can be seen, a laser operating on hydroxyl or deuteroxyl has the potential for penetrating the atmosphere with relatively low losses.

Further, the exhaust products from such a laser would be non-toxic and non-corrosive, containing only (besides an inert gas diluent) oxygen and water and possibly some hydrogen. This is in sharp contrast with the exhausts from HF and DF lasers. In addition, there is the potential for operating the laser directly on the stream exiting an ozonator, with the result that the stores for the laser would consist only of hydrogen and oxygen (plus any diluent).

The above properties make the hydroxyl laser a good candidate for use in airborne and seaborne missions where safety and ease of handling are important considerations.

The following section contains a review of prior programs which have been concerned with achieving lasing in OH. The results of the current program are then presented in the next two sections. The first of these sections is concerned with Phase One of the contract; namely, the design philosophy of a new discharge tube, the measurement of H-atom yields, and the spectral studies of the O_3 induced chemiluminescence. Phase Two of the program was concerned with the design, fabrication, and testing of a transverse flow laser apparatus. The results of this portion of the program are presented in the next to last section. The last section contains the conclusions and recommendations of the program. Various aspects of ozone safety and handling are treated in Appendix A.



NOTE: Absorption bands of possible impurities are indicated. Locations of OH and OD fundamental and over-tone bands are shown. The four lines in each band with highest predicted gain are indicated by vertical lines, solid for OH, dotted for OD. Arrows mark OH lines on which pulsed lasing has been observed.

FIGURE 1. Transmission of 1000 ft Sea-Level Air Path at 59°F and 5.7 torr Water.

PREVIOUS STUDIES

The hydroxyl radical, OH, has been made to lase in pulsed systems by several authors[1,2,3] and pulsed lasing of CO_2 pumped by energy transfer from chemiexcited deuteroxyl (OD) analogously to the DF-CO_2 transfer laser has also been observed.[4] In all of these works, OH or OD has been made by reaction of $O(^1D)$ with molecular hydrogen, where the $O(^1D)$ has been derived from ozone (O_3) by either photo-decomposition[1] or electric discharge.[2,3]

The fact that the reaction of hydrogen atoms (H) with O_3 generates exclusively vibrationally excited OH (v = 7, 8 and 9 appear to be formed preferentially) was reported by Polanyi and coworkers in 1971,[5] but no laser, either pulsed or cw, using this reaction as the primary pumping reaction has been reported yet.

The present authors made a first attempt at developing such a laser under ONR Contract N00014-72-C-0460.[6] In that work a longitudinal flow reactor was employed to carry out the reaction between pure ozone eluted from a chilled silica-gel-filled cylinder and hydrogen atoms formed in a d.c. glow discharge. The vibrationally excited OH formed thereby was assayed spectroscopically by observing the $\Delta v = 5$ and 6 transitions out of vibrational levels v = 5, 6, 7, 8, 9 (the so-called Meinel Bands of the night sky). It was found that about 30 mtorr of OH could be obtained having an apparent vibrational temperature of 6000 to 8000 K and rotational temperature of 500 to 800 K.[6] The hydrogen atom concentrations obtained from the discharge tube were estimated by isothermal wire calorimetry and by gas titration with nitrogen dioxide (NO_2) and were found to be about 30 mtorr also.

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[1] A.B. Callear and H.E. Van den Bergh. "An Hydroxyl Radical Infrared Laser", Chem. Phys. Lett. Vol. 8, pp. 17-18 (1971).

[2] E. Vietzke, H.I. Schiff and K.H. Welge. "Stimulated Infrared Emission on OH Produced by a Pulsed Electric Discharge", Chem. Phys. Lett., Vol. 12, pp. 429-430 (1971).

[3] T.S. Wauchop, H.I. Schiff and K.H. Welge. "Pulsed-Discharge Infrared OH Laser", Rev. Sci. Instrum., Vol. 45, pp. 653-655 (1974).

[4] S.K. Searles and J.R. Airey. "New Chemically Pumped CO_2 and N_2O Lasers", Appl. Phys. Lett., Vol. 22, pp. 513-514 (1973).

[5] P.E. Charters, R.G. Macdonald and J.C. Polanyi. "Formation of Vibrationally Excited OH by the Reaction H + O_3", Applied Optics, Vol. 10, pp. 1747-1754 (1971).

[6] J.W. Raymonda and J.W. Daiber. "OH/OD cw Chemical Laser Studies and New Supersonic Flow Laser Geometries. Part I. OH Chemical Laser Studies", Calspan Report No. WG-5179-A-2, Part I, October 1973.

Thus, hydrogen atoms were the limiting reagent and the low atom yields resulted in OH number densities that were insufficient for lasing in any practical-sized device.

It was clear from the results described above that the key to obtaining lasing in OH using the H + O_3 reaction is increased H atom production. Several definite ways to improve the discharge device were available. The first and probably most important of these is based upon the work of R.W. Wood.[7,8] He observed strong atomic hydrogen spectra from the central region of hydrogen discharge tubes, well away from the metal electrodes, but only H_2 molecular spectra in the regions near the metal electrodes. The tube used in our previous work had the gas flow passing by the cathode just before exiting to the flow reactor. It seemed likely that a large fraction of the atoms produced in the center of the discharge were being lost by recombination on the metal electrodes. Accordingly, a discharge design in which the flow exits to the reactor from the center of the discharge was conceived. In addition, passivation of the interior surface of the glass discharge tube using syrupy phosphoric acid is found to be helpful in reducing recombination in the discharge. Finally, the current available in our previous work was only 35 mA; larger values should result in larger atom yields.

The potential of the OH chemical laser was studied further under Calspan IR and D funding.[9] Here a peculiarity of the spectroscopy of OH was uncovered. The dipole-moment function of OH has a maximum near the equilibrium internuclear separation; this results in anomalously large transition probabilities for first overtone ($\Delta v = 2$) vibration-rotation transitions, relative to the fundamentals ($\Delta v = 1$). Calculations of small-signal gains for overtone transitions suggested that lasing on the overtones should be achievable at vibrational temperatures obtainable in the H + O_3 reaction, especially if a cavity employing optics coated for high reflectivity at the short wavelengths is employed. The accomplishment of lasing at the shorter wavelengths was an added objective of the present program.

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[7] R.W. Wood. "Hydrogen Spectra from Long Vacuum Tubes", Phil. Mag., Vol. 42, pp. 729-745 (1921).

[8] R.W. Wood. "An Extension of the Balmer Series of Hydrogen and Spectroscopic Phenomena of Very Long Vacuum Tubes", Proc. Roy. Soc., Vol. A97, pp. 455-470 (1920).

[9] J.W. Raymonda. "Feasibility and Desirability of an OH Overtone Laser", Memo to C.E. Treanor, W/A 85-141, 1 November 1973.

HYDROGEN-ATOM PRODUCTION

In this section the construction and testing of the first center-extraction discharge tube is described. The methods used for determining the H-atom concentrations from this discharge tube are also described.

AUTOMATED WIRE CALORIMETER

A wire calorimeter is a convenient means for monitoring H-atom flux. Atoms impinging on a wire which is made of platinum have high probability for recombining on the surface. This molecule formation releases heat (the molecule dissociation energy) which is taken up by the wire. The usual method for measuring this heat is to maintain the wire at constant temperature (and thus resistance) by passing electric current through it; the difference in the currents required to maintain a chosen resistance with and without atoms present yields directly the rate of heat release to the wire by recombining atoms.

An electronic controller was constructed which senses the resistance of the calorimeter and automatically adjusts the current fed to the wire to maintain the resistance at a preset value. A schematic of the circuit is shown in Figure 2.

The flux of H-atoms at the wire under a chosen set of discharge conditions was measured as follows. Desired flow-rates of hydrogen and diluent, if any, were established and the resistance of the wire allowed to settle to the desired value. The required current through the wire and the resulting voltage across it were read out on a chart recorder. Increasing the gas flow rate naturally forced the controller to feed more power to the wire to maintain the chosen resistance (temperature) owing to conduction of heat away from the wire by the flowing gases. The discharge was then turned on and brought to the desired running condition as rapidly as possible. This typically took 10 sec or less. The current through the discharge was the principle independent variable since the discharge was run in the normal glow regime where the voltage-current characteristic is flat. Formation of hydrogen atoms was manifested at the wire by a decrease in the electrical power needed to maintain the desired resistance. The difference in electrical power to the wire with the discharge on and off measured the rate of energy transport to the wire by atoms. The powers were calculated from voltage and current values passed to a chart recorder from output terminals in the controller.

Experience showed that the time required for the controller to adjust the current to attain the proper resistance after the discharge was turned on and H-atoms began to flow to the wire was less than one

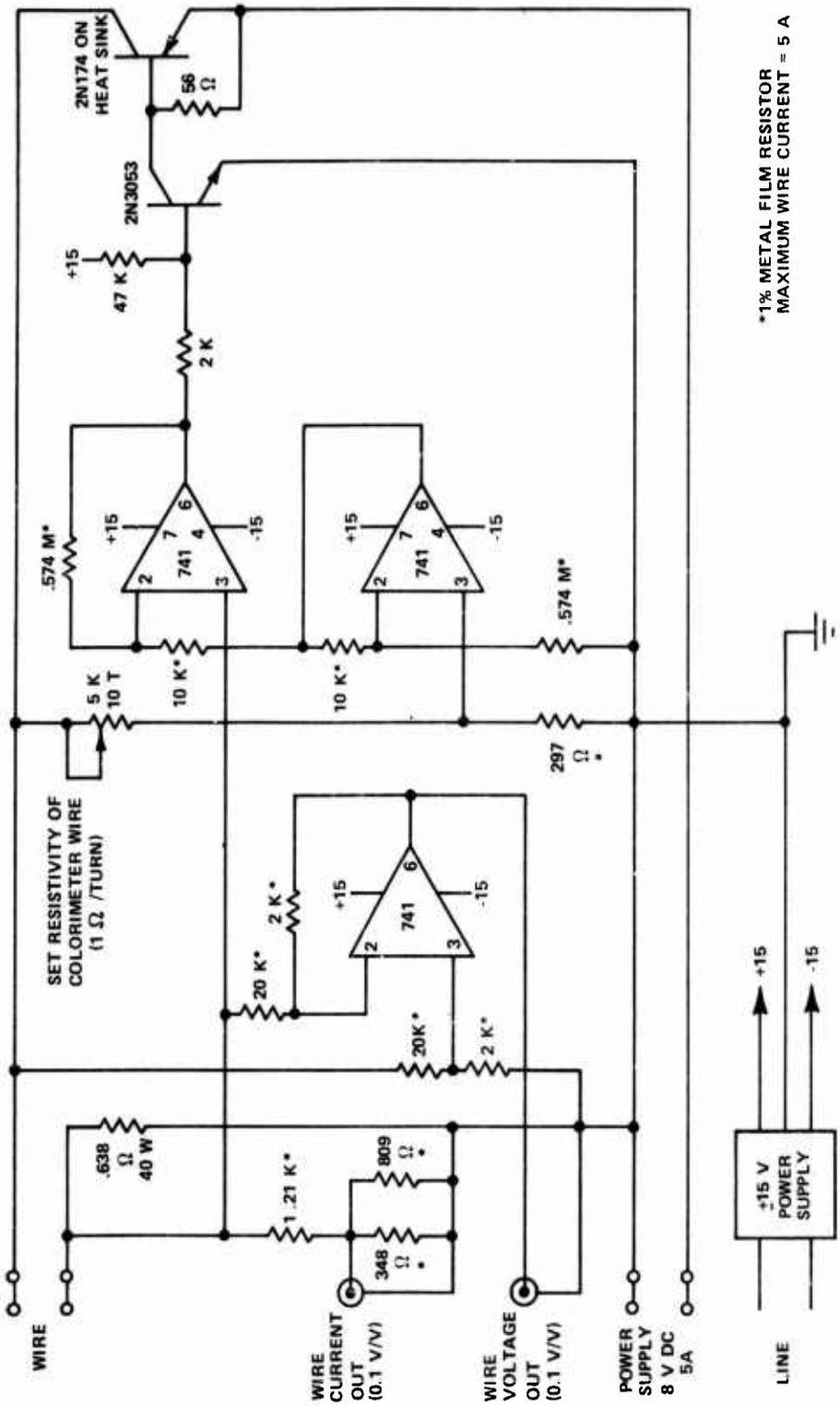

FIGURE 2. Schematic of Electronic Circuitry Employed to Sense and Control the Resistance of the Wire Calorimeter.

second. Consequently, parametric studies of H-atom production efficiency were done by changing variables slowly compared to 1 sec. The wire resistance was usually kept at 3 Ω during the tests described herein. Tests with pure argon and helium showed no detectable energy flux from metastable excited atoms and only a slow drift due to gas and apparatus heating. This drift was allowed for in calculating powers due to recombining atoms.

DISCHARGE TUBE DESIGNS

The discharge tube evolved through several designs during the Phase One portion of the program. Only brief mention of key features of the final design will be made here.

Excessive heating of both the discharge tube and the electrodes was a problem early in the program. A tube constructed of crosses, tees and straight lengths of Corning conical pyrex pipe was finally settled upon. This offered both ruggedness and ease of modification and assembly. The pipe withstood the severe discharge environment well except near the cathode, where heat-induced cracks necessitated replacement of several tees.

The electrodes were made as large as possible while still fitting inside the pipe and they were castellated at the end faces to increase the surface area and create sharp places for attachment of the discharge. OFHC copper was used for the anode and 99% pure aluminum was used for the cathode. The cathode was water cooled, since earlier uncooled cathodes were quickly damaged by overheating.

SURFACE PASSIVATION

Early discharge trials were done with discharge tubes having bare interior surfaces, and the atom yields from these were generally poor. Passivation of the discharge surfaces with phosphoric acid proved very successful in improving the atom yields.

The coating of the tubes was at first a tedious and messy process. The glass to be coated was cleaned by soaking for several hours in hot sulfuric acid-sodium dichromate cleansing solution formulated in the standard way. This was followed by several rinses in distilled water. The resulting glass was then free of impurities which could prevent the phosphoric acid from wetting the surface. The phosphoric acid was Reagent Grade material heated to drive off water until it became so syrupy as to pour only very slowly (solid phosphor pentaoxide was sometimes added to the mixture) to decrease the required heating time. The syrupy acid could be applied to the interiors of the tube with a stirring rod or by careful pouring and rolling of the tubes to distribute it. Successful coatings could also be made with less

viscous acid, but this required long periods of heating the glass to render the acid syrupy in situ and was much more tedius. Successful coatings were also made by dusting dampened insides of tubes with anhydrous P_2O_5. The P_2O_5 which stuck to the tubes was allowed to absorb water from the surroundings and become syrupy. Coatings made by the first mentioned method seemed to be the most uniform and were preferred for discharge experiments.

ATOM PRODUCTION RESULTS

Early tests of the discharge were carried out using the wire calorimeter to gauge the atom yields. Clearly, while the atom fluxes implied by the calorimeter might not be quantitatively correct, the data surely give a lower limit to the actual atom fluxes. This is true because the major sources of systematic errors in the calorimeter readings are (1) failure of all the atoms to collide with the wire and (2) failure of some atoms that do reach the wire to recombine and yield heat to the wire. Both of these errors produce a lower response than is representative of the true atom flux.

A major concern surrounding the use of the calorimeter was that the discharge might prefer to attach to the wire, rather than the intended cathode, since the wire was in a lower pressure region than was the cathode. Accordingly, the calorimeter was located about 50 cm downstream from the orifice separating the discharge from the flow tube. Discharge flow conditions that would make this orifice choke were preferred so that, in later tests, ozone or nitrogen dioxide (for gas titrations) could be added at the injector without changing the discharge flow conditions. It was found that pressure ratios of up to 3-to-1 could be maintained across the discharge orifice while holding the discharge on the cathode. This was determined in early tests using a well-grounded dummy calorimeter made of wire screen in order to avoid damage from possible arcs to the actual calorimeter.

However, the atom yields measured with the calorimeter at this location were rather low, implying only about 5% dissociation or less (the discharge tube was not passivated with phosphoric acid during these early tests).

In order to test the hypothesis that major loss of atoms owing to surface and volume recombination was occurring over the 50 cm of travel required to reach the wire, the wire was moved some 20 cm closer to the orifice. The resulting calorimeter data indicated about a 10-fold increase in atom fluxes over those at the previous location. The data shown in Table 1 are exemplary of the results obtained in these tests. The discharge was run at about 400 mA through 1 to 2 torr of pure H_2. It should also be noted that an early discharge design employing small diameter tubing and uncooled electrodes was used to obtain the data of Table 1.

TABLE 1. Hydrogen Dissociation Efficiency.

H_2 Flow Rate (Moles/Sec)	Power Delivered By Hydrogen Atoms (Watts)	H Atom Flow Rate (Moles/Sec)	Fraction of H_2 Dissociated at Calorimeter Location
2×10^{-5}	1.67	0.77×10^{-5}	0.19
4×10^{-5}	2.24	1.03×10^{-5}	0.13
10×10^{-5}	1.21	0.56×10^{-5}	0.03

Tests with the passivated conical pipe discharge tube produced powers of over six watts delivered to the wire by atoms, and it was decided to proceed immediately to gas titrations to measure quantitatively the H-atom fluxes at the ozone injector.

The gas titrations were carried by injecting metered flows of NO_2 into the flow through the same orifices used for ozone injection in later tests. The injector block carried sapphire windows placed to allow a direct view of the orifices themselves. The progress of the titration was monitored through these windows using an EMI 9558 PMT with an S-20 cathode. A monel bourdon gauge was used to measure the NO_2 plenum pressure. All of the feed lines up to the metering valve, the gauge and the rotameter were heated to over 100 C with heating tape to minimize the amount of N_2O_4 in the NO_2 flow. Any N_2O_4 in the NO_2 plenum surely dissociated in the low pressure region beyond the metering valve, thus delivering two molecules of NO_2 for every N_2O_4 molecule reaching the valve. Since the N_2O_4 molecules are expected to register on the flowmeter similarly to NO_2 molecules, it is concluded that the presence of N_2O_4 in the NO_2 will cause the H-atom fluxes to be underestimated.

The reaction of nitrogen dioxide (NO_2) with hydrogen atoms is very fast, and its usefulness as a titration reaction stems from a continuing sequence of reactions entered into by the products thereof. A simplified scheme includes the following reactions:

$$H + NO_2 \longrightarrow OH + NO \qquad (1)$$

$$H + NO + M \longrightarrow HNO^* + M \qquad (2)$$

$$HNO^* + M \longrightarrow HNO + M \qquad (3)$$

$$HNO^* \longrightarrow HNO + h\nu \qquad (4)$$

Here, reaction (2) between the NO formed in (1) and remaining H-atoms gives rise to electronically excited HNO, radiation from which is monitored as mentioned above to find the titration end-point. The intensity of this radiation reaches a maximum at some intermediate NO_2 flow rate. When the mole rate of flow of NO_2 just equals the original H-atom flow, there are no atoms left to form HNO from the NO and no light is emitted. Thus, the end point is signaled by the reduction of the HNO^* radiation intensity to zero.

In the present system, this radiation was detected against a rather bright background of radiation from the discharge itself and the resulting system noise prevented detection of a sharp end point. Instead, the detection system response was plotted versus the NO_2 mole rate of flow, and the data were extrapolated to zero response.

In the sample data plot, shown in Figure 3, the influence of wetting the hydrogen was tested. Two ways of extrapolating the data to zero seem apparent. One way results in the lowest possible estimate for the titration end point. This involves extrapolation of only the points on the steeply decreasing part of the response plot. In the second way, the more slowly decreasing responses at larger flow rates are extrapolated.

An argument favoring the former attributes the slowly decreasing tail to secondary hydrogen atoms formed by the reaction of OH from reaction (1) with undissociated H_2 according to

$$OH + H_2 \longrightarrow H_2O + H \qquad (5)$$

However, it is well known that the OH produced in (1) is largely in the ground vibrational state and the reaction between H_2 and ground state OH is slow with respect to (1). Furthermore, reaction (2) is also quite slow. Finally, the orifices through which the NO_2 are injected are right at the upstream edge of the observation port, and the NO_2 and its reaction products pass by the window and out of sight quite rapidly. These facts make it unlikely that any appreciable HNO^* could be formed from such secondary hydrogen and have sufficient time to radiate before passing out of the field of view of the detection system.

It is, therefore, believed that the second extrapolation gives a better estimate of the original H-atom flow rate. The initial rapid fall-off of the radiation intensity could be due to the flow speed increasing and carrying the radiators more rapidly out of the viewing field as the NO_2 addition increases. It was observed that the pressure in the flow tube did not double when the mole rate of gas flow doubled; therefore, the flow velocity did increase as the gas flow rate increased.

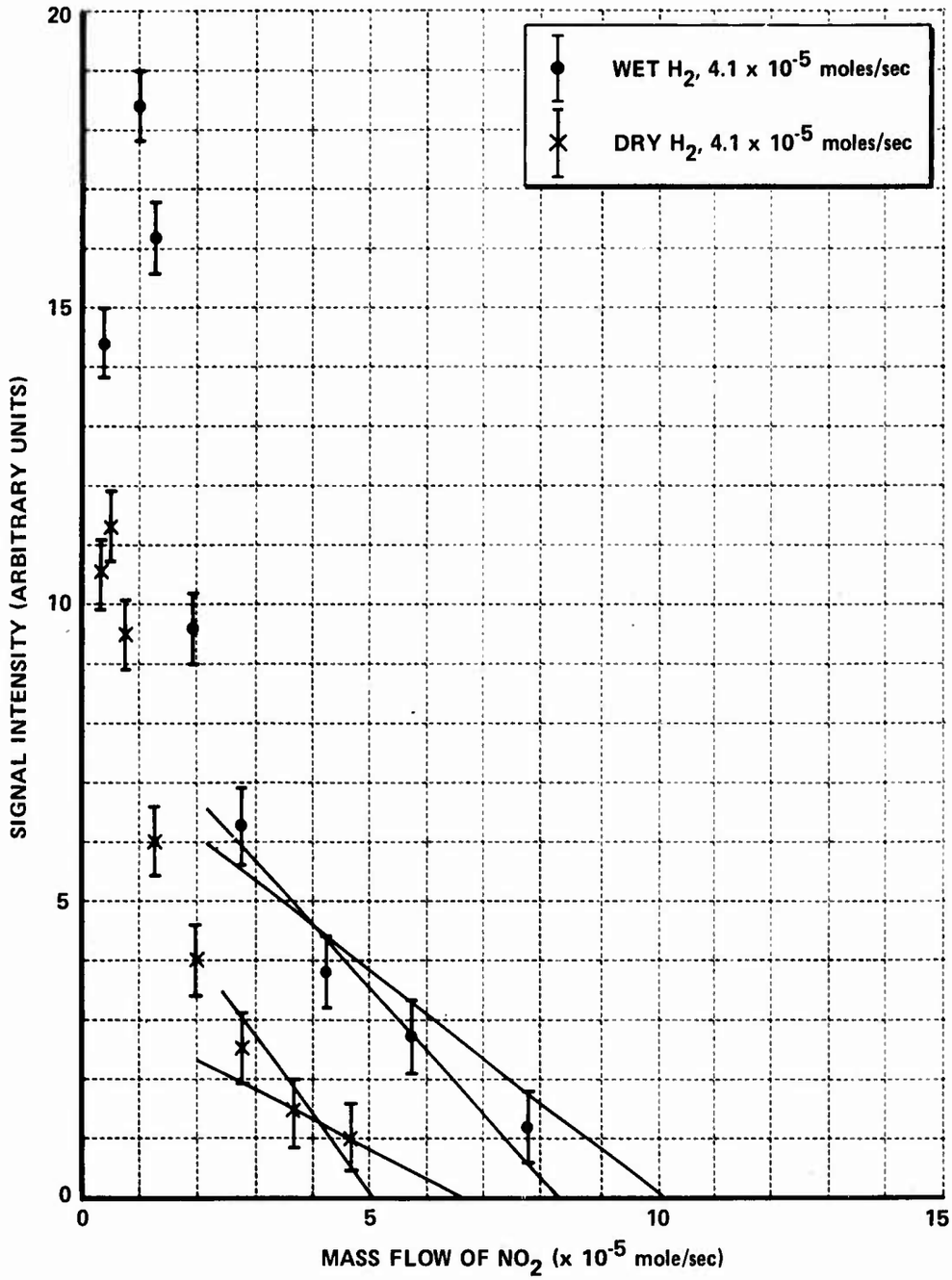

FIGURE 3. Titration Signal vs. Molar Flow Rate of NO_2. Two possible ways of extrapolating each data set are shown.

In Figure 3, the intensity of the light emitted by HNO^* is plotted in arbitrary units versus the mole rate of NO_2 flow. The error bars are a reflection of the noise in the system. Phase sensitive detection with a time constant of 0.3 sec was used to improve the signal-to-noise ratio. The most troublesome source of noise was the discharge itself; it emitted a strong background light, especially in the Balmer α line of the H-atom, which entered the spectrometer after reflecting off the flow tube walls; the discharge also seemed to generate a lot of strong fields that were easily picked up by the amplifier. Two runs are shown, one with pure dry hydrogen and the other with wetted hydrogen; the same H_2 flow rate, 4.1×10^{-5} moles/sec, was used in both cases. Complete dissociation of the H_2 would produce about 8.2×10^{-5} moles/sec of atoms, which would require 8.2×10^{-5} moles/sec of NO_2 for complete titration. It is seen that the data for the wet hydrogen may be extrapolated to about 8×10^{-5} moles/sec of NO_2 and is, therefore, strongly suggestive of complete dissociation. The pressure in the flow tube was about 0.3 torr before any NO_2 addition and the H atom pressure at the injector was, therefore, about 0.3 torr. In the case where dry hydrogen was used, a fractional dissociation of about 65% is implied by the data. (It should be noted that even the most pessimistic extrapolation of the wet hydrogen data from the steeply falling part of the curve implies 50% dissociation. This is surely a lower limit on the degree of dissociation achieved.)

On the basis of these results, and similar results from other titrations, it was decided that the addition of water is beneficial for hydrogen atom production, and this was continued in all subsequent tests.

Addition of argon in any proportion seemed deleterious to hydrogen atom production, according to tests with the calorimeter. Titrations in flows containing argon were not done, however. The hydrogen gas flows yielding the greatest atom fluxes were from 40 to 120 μ moles/sec, corresponding to discharge pressures between 0.5 and 1.5 torr.

SPECTROSCOPIC STUDIES

Following the favorable results obtained with the passivated conical-pipe discharge tube, it was decided to begin tests with ozone injection. The ozone was withdrawn from its storage cylinder by warming the cylinder and also by use of a diluent, argon, to elute it. The ozone was admitted via a metered orifice; the plenum partial ozone pressure was monitored using a mercury lamp emitting the 254 nm line and the flow rate was deduced with the aid of a Fisher and Porter ball-in-glass rotameter. The total pressure in the ozone storage cylinder was measured with a Viatran pressure transducer. The discharge was run mainly on pure hydrogen, although both helium and argon

were tried as diluents. Helium seemed not to degrade discharge performance as argon did.

Radiation, which required the presence of both ozone and H-atoms for its production, was observed. However, it was pale yellow to yellow-green, rather than the dull orange color observed in the previous program. It could be made to be quite bright and to extend the length of the flow tube. It could not be completely extinguished by excess ozone, at least not with the flows which could be established with the present system.

Spectra of this visible radiation were recorded using the 9558 PMT and phase-sensitive detection. Discharge noise made the recorded spectra noisy. To the limit of the resolving ability under the existing noisy conditions, the spectra appeared to be continuous with a broad maximum at about 560 nm. No indication of the strong R and Q heads characteristic of the visible Meinel bands was seen. Scans in the 2 to 5 micron region of the infrared, using an InSb detector, revealed no discreet emission.

In an attempt to increase the luminosity, a heavy flow of pure ozone was established. The result was a very bright and hot flame right at the injector location. Unfortunately, the heat released caused the lexan and teflon components of the apparatus to fail before any spectra could be recorded.

It is felt that this intense flame could well have been producing usable amounts of OH and that a larger, more durable apparatus should be constructed to study it further.

The design, fabrication and testing of such an apparatus was done under the Phase Two portion of this program and these matters form the subject of the next section of this report.

TRANSVERSE LASER APPARATUS

INITIAL DESIGN

The configurations tested through Phase One utilized the peripheral injection of O_3 into a stream containing H, H_2, and a diluent. Such a geometry does not conveniently lend itself to a laser configuration. The product molecules of the main reaction are themselves highly reactive and will quickly destroy the inversion. Consequently, the mixing of initial reactants must occur immediately before reaching the cavity region and the products quickly removed from the cavity. The geometry

which appears simplest, yet meets the requirements, involves positioning the resonator mirror axis normal to the flow direction.

A schematic diagram of such an apparatus is shown in Figure 4 and the flow injection sequence in more detail in Figure 5. The gain length must be sufficient to overcome outcoupling and other losses. Estimates based on the anticipated vibrational temperature and OH concentrations indicated that 10 cm was adequate.

In sizing the device, it was considered desirable to maintain reactant fluxes similar to those studied in Phase One, thereby assuring that adequate delivery rates of H-atoms and O_3 would be obtained and that the total flow could be exhausted by the available pump.

An H_2 flux of 1 mmole/sec corresponds to a mass flux of 0.002 gm/sec. If a dissociation level of 10% of the initial molecules is assumed, then the molecular weight, MW, is 1.83 and the specific-heat ratio is 1.44. Nominal operating conditions within the discharge tube correspond to a pressure of 5 torr with a temperature of 400 K. Considering the velocity of the flow in the discharge tube to be negligible, then the stagnation sound speed is

$$a_s = (\gamma RT / MW)^{1/2} = 1.62 \times 10^5 \ cm/sec$$

and the stagnation density is

$$\rho_s = 1332\, PMW/RT = 3.67 \times 10^{-7} \ gm/cm^3$$

By taking the minimum area of the H-atom exit slot as 10h, where h is the width of the 10-cm long slot, the isentropic mass flow equation

$$m_f = \rho_s a_s A^* \left(\frac{2}{\gamma+1}\right)^{\frac{\gamma+1}{2(\gamma-1)}}$$

can be used to infer a required slit width of 0.006 cm or 0.002 inches. An ideal slit nozzle with this width having a discharge coefficient of unity should then pass the desired mass flow.

This slit width is very small and will be operated at low stagnation pressures. This leads to the possibility that the mass flux through the nozzle may be limited by viscosity. That this is likely can be seen from a comparison of the mean-free-path in the discharge which is approximately 0.0015 inches to the planned slit width of 0.002 inches. To compensate for this effect, the slit was actually made three times larger. The larger-width nozzle was found to give very nearly the desired mass flow. The 0.006-inch wide slit was cut lengthwise into a glass tube by use of a diamond-toothed circular saw. The slit width was constant over the 10-cm length to better than 0.0001 inches.

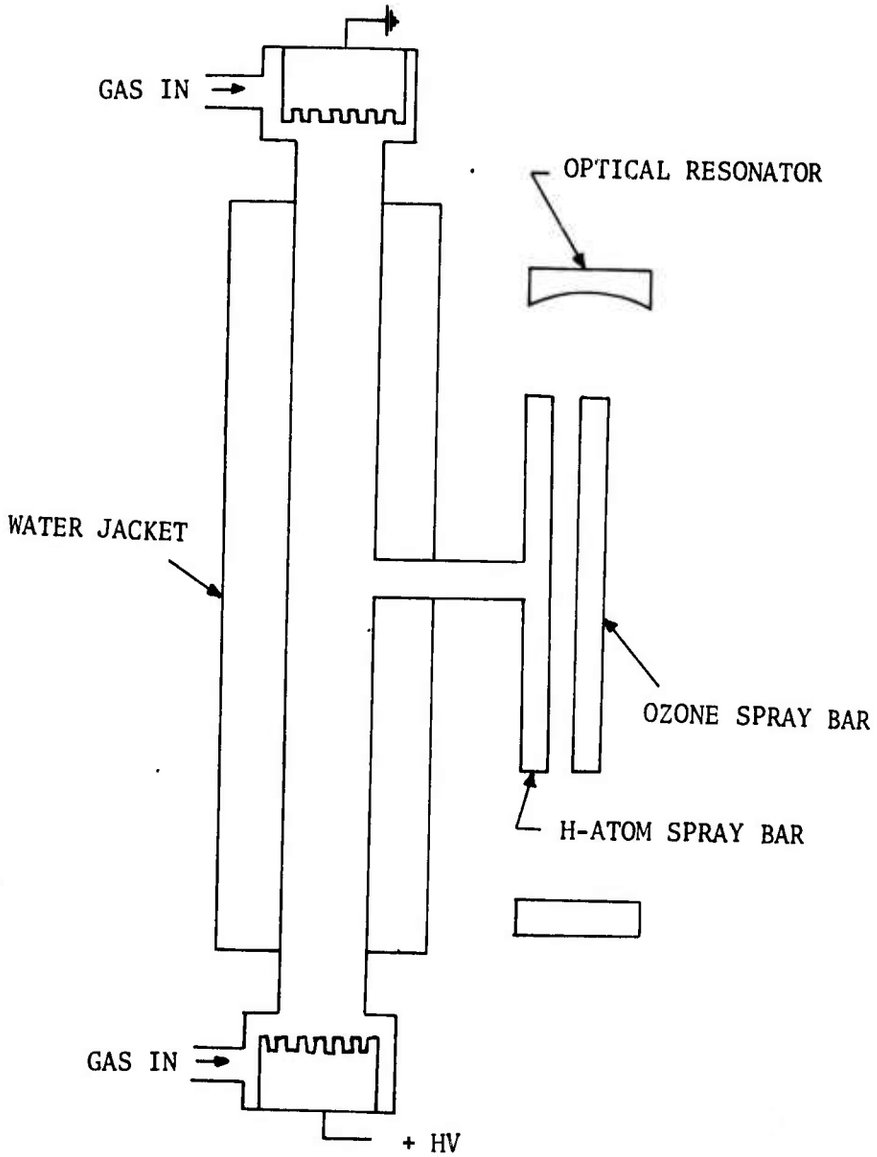

FIGURE 4. Schematic Drawing of a Transverse OH Chemical Laser.

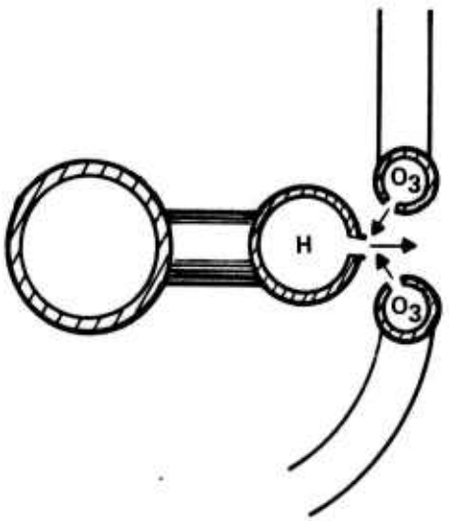

FIGURE 5. Schematic Drawing of the Spray Bar Flows.

The nominal mole flux of H-atoms is 0.2 mmoles/sec. The ozone mole flux should be of the same order. This corresponds to an O_3 mass flux of 0.0096 gm/sec. The pressure of ozone in a storage vessel must be kept below 100 torr to prevent catastrophic spontaneous decomposition. The actual drive pressure is even lower than this for the present apparatus. The O_3 is obtained by driving it off the silica gel by warming the storage vessel; for the present system the heating rate available limits the drive pressure to near 10 torr while O_3 is being extracted. The total exit area required for the O_3 injectors is thus found to be 0.024 cm^2. In this calculation, the delivery temperature was taken as 300 K, the molecular weight as 48, and the specific-heat ratio as 1.4.

The configuration selected for the O_3 injector was a series of holes of 0.007 inch diameter spaced on 0.040 inch centers over the full 10 cm length of the reaction zone. The injectors were fabricated from stainless steel. They had approximately 98 holes cut into them by the EDM process. Two spray bars were used so that the O_3 could be mixed with the hydrogen stream from both the top and bottom. The total orifice area available from both spray bars is then 0.048 cm^2. This is twice the calculated area and, therefore, compensates for viscous effects which will reduce the discharge coefficient. Tests carried out with the spray bars indicated that the desired mass flow was achieved.

ENGINEERING DESIGN

After the size of the injection ports for the hydrogen and ozone were calculated, a sizing of the overall system was made. The system shown in Figure 4 is most easily reduced to hardware by enclosing the discharge tube and both spray bars within a large vacuum chamber. The best relative location of the spray bars as well as the downstream distance where maximum OH concentration would occur were not initially known. Therefore, the H-atom spray bar was placed on a stage which could translate in the stream direction. The O_3 spray bars were placed on a separate translation stage and had the further freedom of each bar being independently movable in the vertical direction. A photograph of the final system assembled inside the vacuum box is shown in Figure 6.

The discharge tube was fabricated of glass. In planning the fabrication of the discharge tube, it appeared possible to obtain a compact and flexible design by using a sealing tape manufactured by the Vitta Corporation to form glass-to-glass seals. Samples were ordered and tested. The assembly procedure recommended by the manufacturer proved to be totally inadequate. Variations were tried and a successful procedure finally developed. This involved a very slow initial

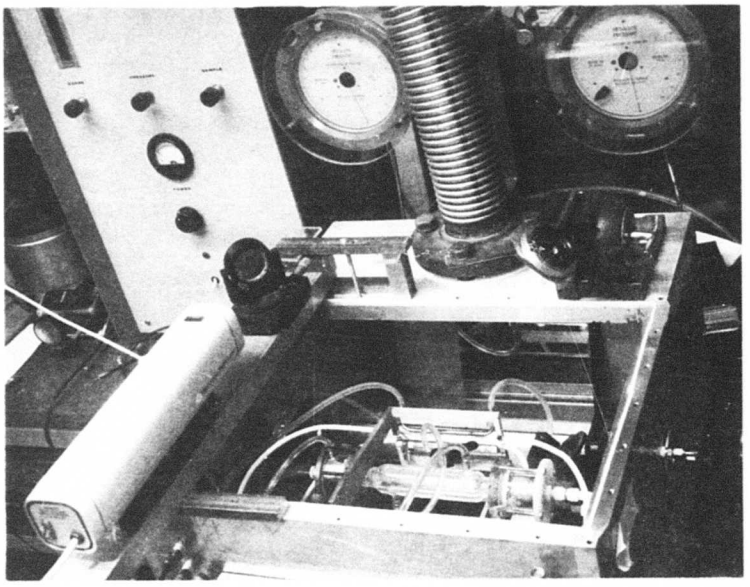

FIGURE 6. Photograph of the Transverse Laser Device Showing the Resonator Alignment Procedure.

warm-up and a long soak at a temperature just below that where the pyrex would deform. The active region for the discharge was approximately 8 inches long and 1 inch in diameter. A phosphoric acid coating was placed on the walls of the discharge tube and H atom spray bar. The cathode was constructed of aluminum and the anode of brass. Both electrodes were water cooled and had numerous slots cut on the active frontal area. A photograph showing more details of the discharge tube is given in Figure 7.

The system was assembled with the O_3 spray bars positioned 0.61 cm downstream of the H spray bar and $\pm$0.63 cm in the vertical direction. The spray holes for the ozone were aimed 20° from vertical into the upstream direction. The centerline of the resonator was positioned 0.30 cm downstream of the H spray bars. In assembly, great care was taken to insure that all three spray bars were parallel to the resonator axis. This was done by using an intracavity He-Ne laser beam to define the resonator axis and to serve as a reference for the spray bar alignment.

EXPERIMENTAL RESULTS

The first run with the assembled device used the H_2 flow, but no O_3. The objective of these tests was to verify the proper operation of the discharge. Corona was observed to leak from the anode assembly to various metal components in the vacuum box. Silicon compound was placed over various joints and a small rf bypass capacitor (500 pfd, 20 kV) was placed between the anode and ground. These two fixes substantially improved the discharge operating characteristics and operating range. The discharge now required approximately 1000 V across the electrodes to drive a 0.5-A current. A ballast resistance of 5 kΩ was used.

After proper operation of the discharge was obtained, the ozone flow was established. In the first runs, the elution technique was used to deliver O_3 to the device. This involves the passage of Ar through the ozone storage cylinder. The Ar elutes the O_3 and the mixture is delivered to the spray bars. Flow rates of H_2 and Ar were varied until a bright green glow could be observed when the flow was viewed through the windows on top of the vacuum box. A power meter with a 1-sec time constant was placed immediately beyond the outcoupling mirror. No laser radiation was detected. By varying the flow rates, operating pressure, and discharge current, the green glow could be made to persist for a substantial downstream distance. During these tests, runs were made in which the O_2-O_3 mixture coming out of the ozonator was fed directly into the test device. The same visual appearance to the green glow could be obtained with the O_2-O_3 mixture as was obtained with the Ar-O_3 mixture. However, lasing did not occur.

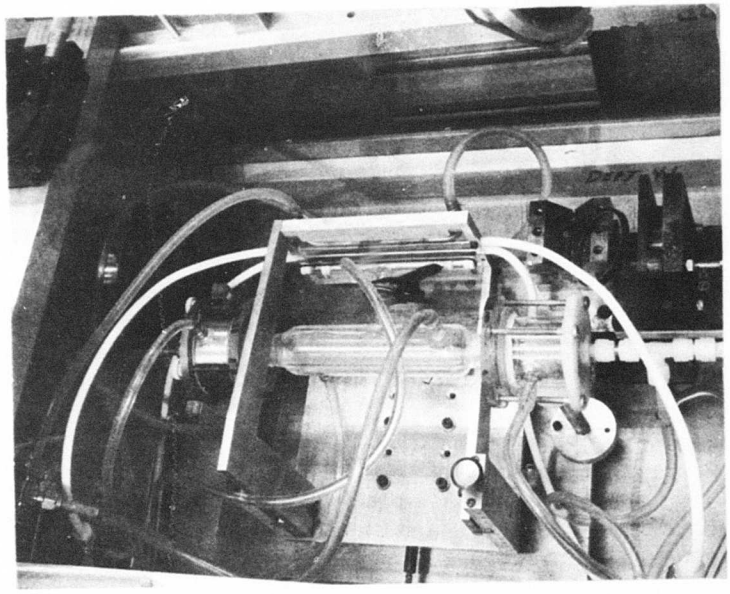

FIGURE 7. Photograph Showing the Discharge Tube and Spray Bars Inside the Vacuum Box.

After these runs, the resonator mirrors were removed and a new optical system installed which incorporated a 1/4-meter monochrometer. The objective of the next test series was to identify the green glow. An RCA 8575 photomultiplier tube was used to detect the radiation. The output of the PMT was amplified with a PAR Type 124 lock-in amplifier, the signal was then displayed on a chart recorder. A second pen on the chart recorder served as an event marker by noting every tenth turn of the screw rotating the grating. A copy of a run log obtained with flow conditions which maximized the green emission is shown in Figure 8. The green glow is seen to be a broad continuum peaked near 580 nm. The atomic hydrogen lines are also a prominant feature.

Several runs similar to this were made with similar results. Visual observation of the interaction region through the side window which served as the monochrometer viewing point, revealed that, in the first moments after O_3 was turned on, the luminosity was light yellow-orange, not green. A speculation was made that the green glow could be associated with the chemical destruction of OH by residual gases in the vacuum box. The background pressure in the box could not be reduced in the present system because the exhaust pumps were separated from the vacuum box by 60 feet of 3-inch diameter pipe. The losses in the pipe limited the throughput so much that even a tripling of pump capacity made no difference in background pressure. Therefore, a different method of O_3 delivery was used which did not require the Ar flow. The ozone storage vessel was heated to drive the O_3 off the silica gel. The pressure in the vessel was continuously monitored to be sure it stayed below the 100-torr limit.

With this modified system, the spectral runs were repeated. Flow conditions were found wherein there was a yellow-white glow near the H-atom slit with the green glow commencing downstream. The run log for this case is shown in Figure 9. Peaks occurred in the spectrum which correlated with the known position of $\Delta v = 5$ and $\Delta v = 6$ transitions of OH. The conditions for maximizing the visible OH emission were different from that used to test for lasing and this, therefore, is thought to be the reason that lasing was not observed.

A complete optimization requires, however, that emission from the $\Delta v = 1$ transition be maximized. The PMT was, therefore, replaced by a cooled PbS detector. The first run in which an infrared sensitive monitor was employed gave an indication of the $\Delta v = 1$ bands. The spectrometer was then set to one of these peaks and conditions varied. Movement of the image of the monochrometer slit 5 mm downstream increased the signal. Increasing the discharge current increased the signal. Decreasing O_3 flow reduced the signal. Addition of He to the H_2 flow reduced the signal. A run log showing a spectrum taken after these optimizations is shown in Figure 10. The wavelengths of the various peaks are consistently 0.01 microns below the known band-head positions in the fundamental sequence. This difference is

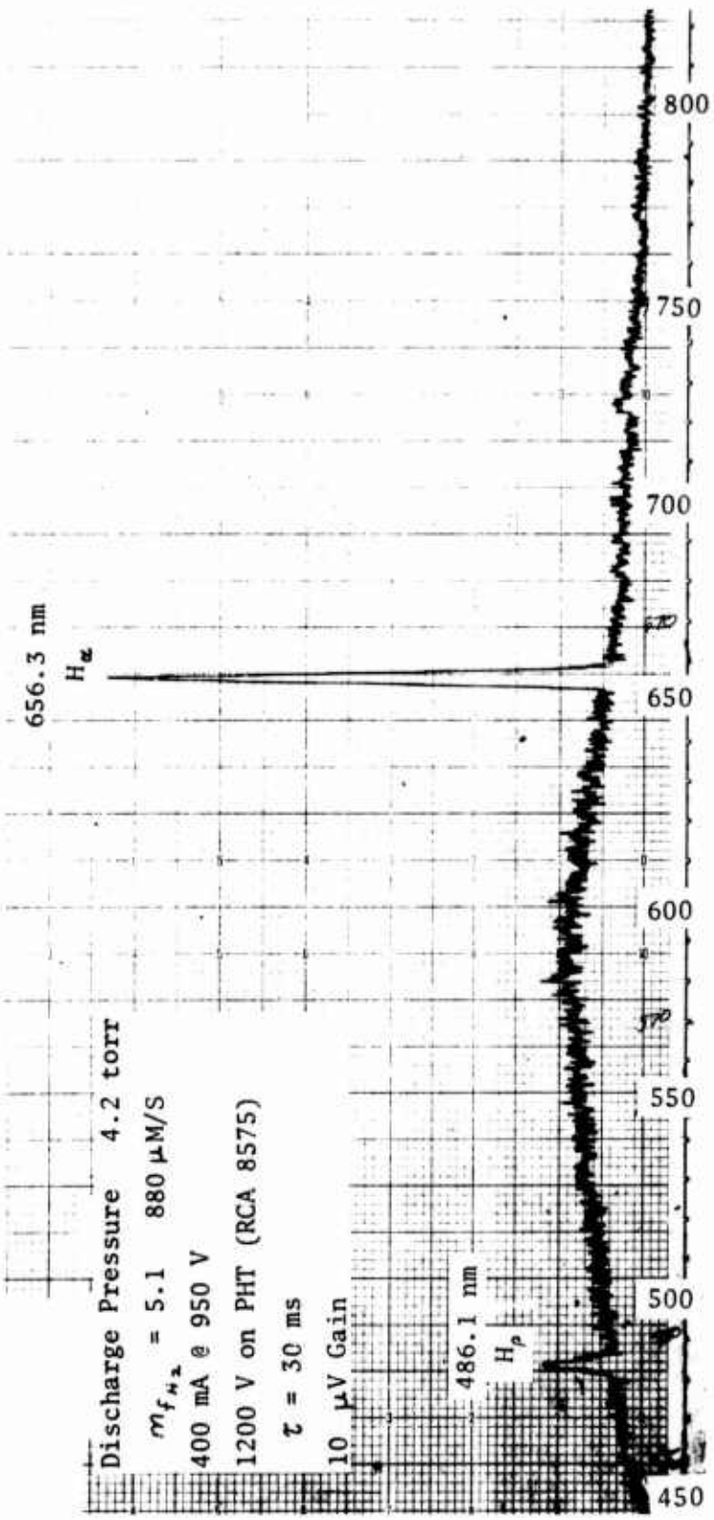

FIGURE 8. Copy of the Run Log Showing the Spectral Content of the Green Chemiluminescence.

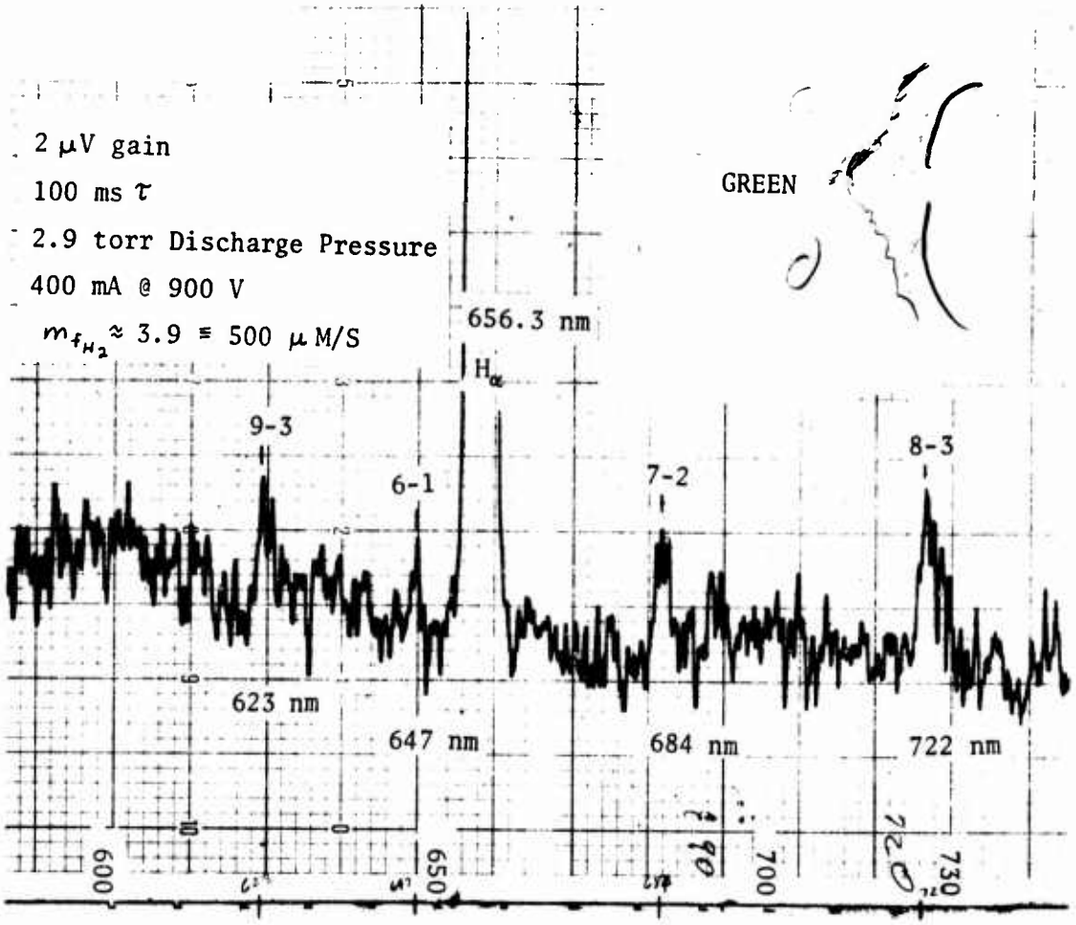

FIGURE 9. Copy of the Run Log Showing the OH Emission in the Visible Spectral Region.

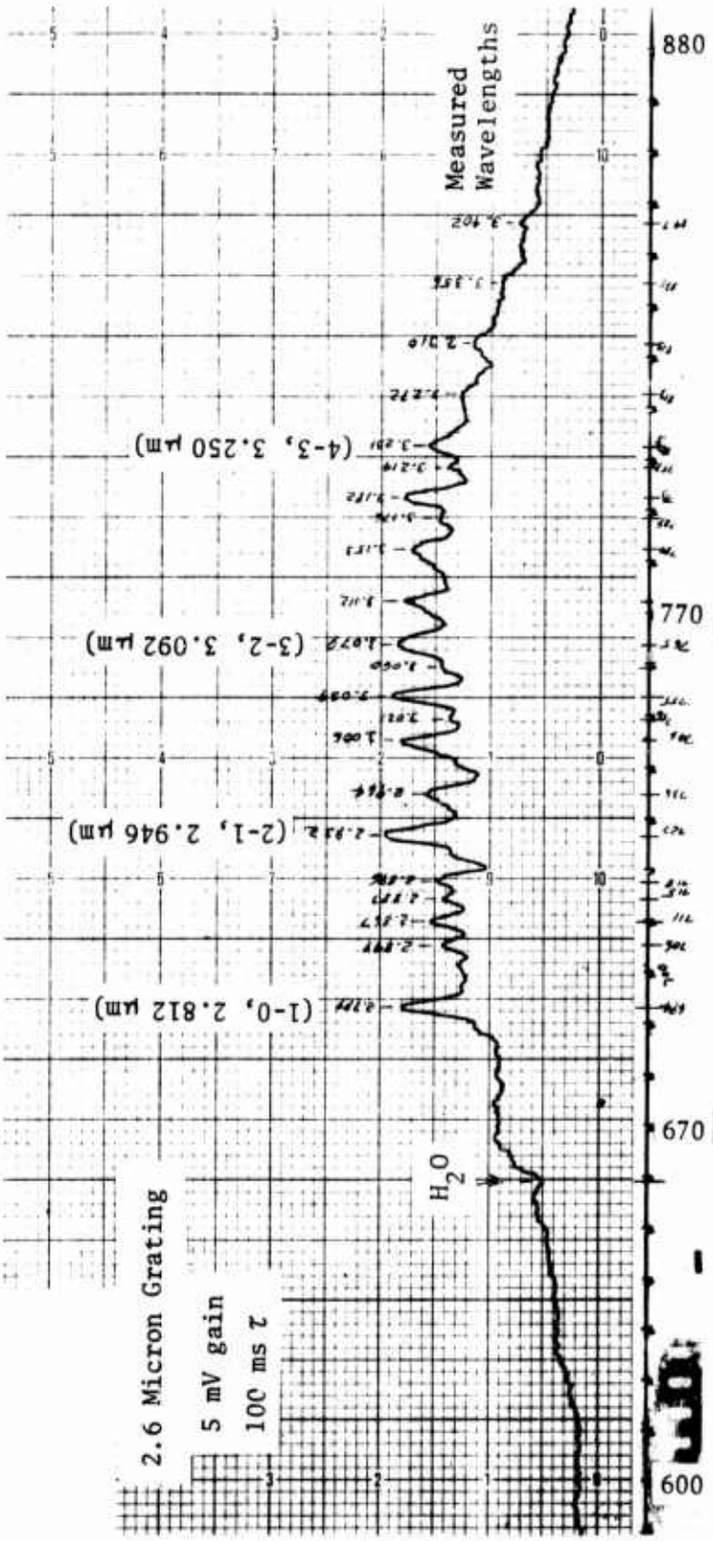

FIGURE 10. Copy of the Run Log Showing the Fundamental Bands of OH.

well within the drive chart calibration accuracy. The fall off in signal at long wavelengths is caused by decreasing sensitivity of the detector. This was verified by scanning a blackbody source.

Further optimization runs were planned. These involved varying the vertical position of the O_3 spray bars and pushing all flows to their limit. Unfortunately, these runs were never made. Shortly after the spectrum shown in Figure 10 was obtained, a TYGON line which carried cooling water away from the discharge tube ruptured. The cold water sprayed throughout the vacuum box. Some of this water reached a short uncooled segment of the discharge tube. The thermal shock caused a structural failure of the discharge tube. The time remaining in the program did not permit a new discharge tube to be fabricated. Repairs to the tube were attempted but proved fruitless.

CONCLUSIONS AND RECOMMENDATIONS

The attractiveness of an OH cw chemical laser is its (1) operation at wavelengths within atmospheric transmission windows, (2) utilization of safe, storable fuels, and (3) generation of non-toxic final products. The achievement of such a laser is, therefore, desirable. The present program has brought much nearer the realization of such a goal.

Methods for the safe generation of large fluxes of H atoms and O_3 have been developed. Studies in a flow device potentially capable of achieving lasing were made. The spontaneous emission from the reaction chemiluminescence indicated that excited OH was being produced. Optimization of the OH emission indicated that the flow conditions and spatial location for maximum OH concentration were not those initially thought to be suitable for lasing. The accidental breakage of the discharge tube prior to those runs which were planned to test for lasing under optimum flow conditions was unfortunate. This prevented the ultimate test for the potential of the OH cw chemical laser from being carried out.

The fact that lasing was not achieved on this program is not indicative that such lasing is not possible. It is recommended that a short test series utilizing a new discharge tube be carried out. The monitoring of the spontaneously emitted OH fundamental band spectra should also be done so that an effective vibrational temperature of the OH could be inferred. From this, the anticipated gain could be calculated and compared to the experimental observations.

Appendix A

OZONE SAFETY AND HANDLING

Ozone is a hazardous substance and, accordingly, a considerable body of literature is devoted to its properties and behavior. An excellent summary of chemical and physical information pertaining to ozone is the American Chemical Society publication,[10] Advances in Chemistry, No. 21. The following is a brief summary of the precautions taken at Calspan to minimize the hazard from ozone.

Ozone is rather poisonous; the 8 hour tolerance level usually quoted is 1 ppm. The odor of ozone is pungent and readily detected by a "fresh nose", but olfactory fatigue sets in rapidly. This has the result that low but potentially dangerous levels of ozone can be present over an extended period without the fatigued workers being aware. The first and most important step taken to reduce this hazard was careful construction of the gas handling system using only compatible metals (aluminum and stainless steel - copper containing apparatus must not be present) and a minimum of glass or other breakables. In practice, the only non-metals used were a heavy glass flowmeter, sapphire observation windows and the heavy-walled pyrex-pipe flow tube (used only in Phase One). The second precaution was venting all lines to the outside via either a fume hood or through the pumping system. However, the ozone had to pass through a steel trap heated to 300 C before it could reach the pumps. Ozone is known to be rapidly and quantitatively decomposed to oxygen at 300 C.[11] This trap served both to protect the pumps from the explosive hazard (to be discussed below) and to protect personnel outside the building from accidental exposure.

The third precaution was to keep handy a supply of potassium iodide solution. This solution, applied to a towel or tissue, is an excellent ozone detector, yielding the deep yellow-brown color of free iodine in water solution when exposed to ozone. Such detectors were used to locate small ozone leaks, when they were first detected by smell, in order to eliminate such leaks immediately, before olfactory fatigue could set in. The laboratory could be ventilated quickly via large overhead doors if ozone leaks occurred.

Ozone can also be hazardous because of its tendency to detonate when in the pure state either in liquid form or at excessive pressures (to be defined below). It should be noted that, in spite of its strong oxidizing properties, ozone gas does not cause combustibles to enflame spontaneously on contact as flourine does. (Nor is it as corrosive to the skin as F_2.)

[10] American Chemical Society, Washington, D.C., March 1959.

[11] E.W.J. Daiper. "Ozone-Practical Aspects of its Generation and Use", Chemtech., pp. 368-375 (June 1972).

Ozone can be stored easily and safely in the pure state if it is kept absorbed at dry ice temperature on the surface of silica gel in an aluminum or glass container. (Stainless steel is not acceptable because the slightest corrosion will catalyze the decomposition of ozone, even at -78 C.) To obtain ozone in this form, the ozone/oxygen mixture (about 5 moles percent ozone usually) from a corona discharge ozonator is passed through a container (an aluminum cylinder was used which was obtained from Hoke Corporation), cooled to -78 C and containing coarsely divided 6 to 12 mesh silica gel. The ozone is thereby adsorbed and and held on the gel while the oxygen passes through. Ozone could be stored in this way for weeks at a time with only minor losses due to slow decomposition. So stored, it is stable to physical agitation, sudden pressurization with argon, helium or oxygen and even to exposure to copper and brass.

In order to remove the ozone from such storage, one heats the cylinder slowly. Ozone begins to desorb at a significant rate at about -40 C. Removal can be accomplished at lower temperatures by elution with an inert gas.

The ozone partial pressure in any container, including the low temperature silica gel bed, should not be allowed to exceed 100 torr (2 psia) because the risk of detonation increases dramatically at such pressures. Also, liquid ozone should never be allowed to accumulate unless it is in the presence of an adequate amount of silica gel so that deactivation by absorption can take place for all the ozone present. This is usually not a problem when temperatures below the dry temperature are avoided since pure ozone has a normal boiling point of -112 C.

Instruments containing any copper should not be exposed to ozone, since copper catalyzes the decomposition of ozone. In particular, Wallace and Tiernan gauges employ diaphragms made from a beryllium-copper alloy and they should, therefore, not be used to measure ozone pressures. An acceptable device for measuring ozone pressures are strain gauge type transducers in which the ozone contacts only stainless steel.

It is important that ozone not be allowed to reach any vacuum pumps since it can be compressed therein to pressures at which detonation can occur. As mentioned above, a steel trap heated to 300 C was used in the vacuum line to protect the pumps from ozone.

It has been found feasible to use a floating ball glass rotameter to measure ozone flow rates. It was enclosed in a plexiglass shield for protection of personnel in case of an explosion (which did not occur during this program).

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