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LABORATORY RESEARCH ON THE INCINERATION
OF MUSTARD

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LABORATORY RESEARCH ON THE INCINERATION OF MUSTARD

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INTRODUCTION

The decision to destroy surplus supplies of the vesicant agent, sulfur mustard (H, HD), happened to coincide with a growing awareness by the general world public of environmental pollution that might be due to all manner of wastes. The science of ecology itself as well as characteristics of materials relating to carcinogenicity, mutagenicity, teratogenicity and other toxicological properties (chronic and acute) have become household subjects. In addition, accurate data were required to meet the requirements of the National Environmental Policy Act (NEPA), PL 91-190.

Rapid laboratory studies were required to supply fundamental chemistry input for design, fabrication and operation of a mustard incinerator. Since sulfur mustard, bis(β -chloroethyl)-sulfide, is readily flammable, incineration was considered the most economical means for large-scale destruction of surplus stores. At the outset of this work, little information existed relative to material balances of mineralized product, or to the achievable level of conversion to inorganic products under a dynamic, continuous system. Furthermore, as the incineration research proceeded, requirements for almost total containment from the atmosphere of even mineralization products such as sulfur oxides (SO_x), hydrogen chloride, and nitrogen oxides (NO_x) became tighter. Thus, a problem which initially was limited to the determination of the quantity and nature of incompletely combusted agent, or organic pyrolysis products therefrom, now included the accountability for all potentially emittable material.

Prior to the work to be described, and the passing of the NEPA, mustard had been burned in the field when mixed with "petrol" in a preheated, modified smoke generator (1, 2); in open trays on a ton-a-day basis (3); in flashing studies related to artillery shells (4, 5, 6); and in the disposal of tail gases in H manufacture (7). All of these tests, including

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detection of agent, were mostly subjective and lacked sophistication in analytical methodology (including specificity, sensitivity and accuracy). In the continental United States, neat mustard had been incinerated and the effluent gases, grossly diluted with large volumes of air, were disposed of through a tall stack (200 ft) at Rocky Mountain Arsenal (RMA). Burning appeared to be highly effective, but no hard data had been obtained to indicate the degree of efficiency, the amount of mineralization, or the quantity of H that might be in the effluent (8). The closest to a fundamental thermal decomposition study of mustard was performed by the Garrett Corporation (9,10). In this effort, an Arrhenius plot of data from 250° to 400°C could be extrapolated through 468° and 482°C to give predicted incineration efficiencies of 99.9 and 99.99% at a burning residence time of 0.1 second. Knowledge gaps thus existed, especially when totally fail-safe agent destruction systems were being sought; and with need for at least two orders-of-magnitude more certainty than that previously calculated.

A fuel-fired incineration furnace should be capable of reaching and maintaining temperatures in excess of 800°C once a steady isotherm is obtained. Sulfur mustard has a flash point of 105°C, a heat of combustion of 7950 Btu/lb, and a heat of vaporization of 168 Btu/lb. Under these circumstances the philosophy becomes essentially one of "adding fuels to the fire", but without emitting intact agent or even nontoxic odoriferous, organic pyrolysis products.

EXPERIMENTAL

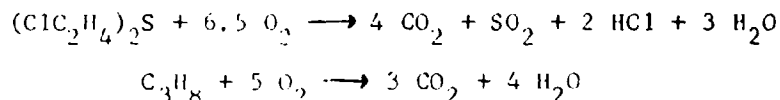
For purposes of laboratory study, an attainable isotherm of 800°C was selected with available laboratory propane gas as fuel. Design of equipment such as furnaces was based on the most likely arrangement of an eventual commercial burner, and of the general dimensions that would allow laboratory data to be extrapolatable to plant-size capacity. Gas collection systems (bubblers, scrubbers) were based on our best experience for collecting gases and aerosols. The designed all-glass burner is shown in Figure 1; that of the entire assembly, in Figure 2. The serial gas collection assembly was designed to include impaction filters and even an electrostatic precipitator for smoke (aerosol) collection. By insertion of a T-joint immediately after the water condenser (Figure 2), it was possible, when desired, to take a syringe air sample prior to the collector bubblers.

The proposed experiments required a furnace capable of operating at 800° to 900°C (1472° to 1652°F), a burner for both mustard (HD) and fuel (propane), effluent gas collectors (scrubbers), and methods for the analysis of the collected effluent. During the course of these experiments, two all-glass laboratory incinerators were fabricated and operated. The initially designed furnace system used 0.13 gm of HD per run with a flow rate of 4.3 ml/min for a total

of 100 ml. A second incinerator, having at least 100 times the capacity, burned 13.1 gm of HD per run (10 ml total) at 0.46 ml/min. Both systems were batch operations analytically, but with uniform burning for 22 to 23 min during each experiment.

The design of the furnace system was such that volume of equipment could be used to calculate the retention times of HD and other gases depending on air, fuel, and mustard flow rates. The quantities of HD (rate of flow) were selected to simulate, at very small scale, a 3 gpm flow of agent under macro conditions (Denver, Colorado); and air flows at the liters-per-minute level, calculable from 12,000 cfm to 113,000 cfm of air.

Each mole of HD (distilled mustard) requires 6.5 mole of oxygen for complete combustion. Each mole of propane requires 5 moles of oxygen for complete combustion.



In these experiments, as in the actual incinerator, oxygen was supplied in the form of air. The quantity of oxygen supplied was calculated from the volume of air per minute indicated by the equation below:

O_2 Supply (mmole/min) =

$$\frac{\text{Air (liters/min)} \times 20.9 (\% \text{ O}_2 \text{ in air}) \times 10^{-2}}{22.4 \text{ (liters/mole of gas at STP)}} \times \frac{273.1^\circ\text{K} \times 10^3}{T^\circ}$$

The oxygen demand is calculated from the sum of the amounts of oxygen required to completely burn the propane fuel and the HD. The ratio then representing the overall burning system is: mmole O_2 supplied/mmole O_2 required. As will be shown later, little difference in completeness of burning of HD could be observed between oxygen ratios of 1.13 to 9.94.

The ratio of air flow to mustard flow was varied to determine the minimum quantity of air (oxygen) that would be needed. The air flow also had a direct controlling effect on residence time of mustard at temperature, and was also related as a volume factor to emission levels of mustard from the furnace.

The residence time of HD at temperature (800°C) was calculated using the following equation:

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Residence time (sec) =

$$\frac{\text{Volume of heated furnace tube (liters)} \times 60 \text{ (sec/min)}}{\text{Volume of air at } 21^{\circ}\text{C (liters/min)} \times \frac{1073^{\circ}\text{K}}{273^{\circ}\text{K}}}$$

The residence time of HD at temperature was proportional to the combined air flow rates and volume of the heated area of tubing (glass furnace liner). The small-scale incinerator was operated so that the HD residence time range was 0.25 to 2.22 sec, and the large-scale laboratory incinerator was operated at residence times from 0.12 to 0.26 sec (Table I).

As can be seen by inspection of Table I, little difference is observed in recovery of "total" mineralization product (HCl and SO_x) within the tested ranges of residence time.

The HD available for these studies was 97.0% pure as determined by the freezing point (13.4°C). Analysis of this sample showed a total sulfur content of 20.71% (102.7% of theoretical) and a total chlorine content of 43.30% (97.1% of theoretical). Differences in the laboratory system (other than size) from planned RMA operation were: (a) glass versus metal and firebrick construction, (b) propane as fuel instead of natural gas (methane), (c) the burning fuel and mustard served as only part of the heat source, and (d) the geometrical design of furnace and scrubber systems.

A furnace tube isothermally heated by electrical means maintained a continuum of isothermal conditions that would be characteristic of a gas-fired, firebrick incinerator under equilibrium conditions. Both laboratory "incinerators" used automatic feeding syringes for control of mustard flow, and rotameters for maintaining fuel and air flows.

The bubbler types employed for the collection of effluent are illustrated in Figure 2. A U-tube chilled in an ice bath preceded the series bubblers. It served to cool the effluent gases and condensed, to a certain extent, water and hydrogen chloride that formed during combustion. The first bubbler usually contained 50 ml of methanol and was kept in an ice bath, whereas the second methanol bubbler (also 50 ml) was chilled in Dry Ice (-80°C). A third bubbler contained 100 to 150 ml of 1 to 2 M aqueous sodium hydroxide or sodium bicarbonate and was kept at ambient temperature. A single, ice-cooled bubbler containing 150 ml of 2.3 M sodium bicarbonate was found to be very efficient for collecting all of the mineralized species (SO_x, HCl, NO_x), but would be useless for HD. The combined U-tube and methanol bubblers collected all of the "intact" mustard, at least 90% of the hydrogen chloride, and 5 to 10% of the sulfur oxides. The caustic or carbonate bubblers trapped the remainder of the hydrogen chloride and practically all of the sulfur oxides (SO₂,

SO₂). The test for nitrogen dioxide was generally on the negative side; indicating that nitrogen fixation was negligible at 800°C. The highest results found for NO_x were of the order of 3 to 6 ppb.

The laboratory incinerator experiments showed that any HD which survived the burning would be collected intact in the methyl alcohol bubblers. Although water represents the largest single product of combustion, the high acidity (HCl and H₂SO_x) and common ion (Cl⁻) in the bubbler precluded any but slow hydrolysis of mustard (less than 25% in 24 hr, and 50% in 60 hr). This time study was made on both actual samples as collected from the incinerator and from simulated mixtures of HD, HCl, SO_x, H₂O and methanol.

Efforts were made to find and identify any organic fragments that might be present in the effluent gases. Neither the bubblers nor a particulate filter mounted aft of the bubblers showed any organic residue, even when checked by mass spectrometer. The apparent absence of organosulfur in the collection media and the reasonably high collection efficiency for hydrogen chloride (usually better than that for the sulfur oxides, SO_x) indicated that incineration was essentially complete. Higher collection efficiencies for SO_x, as obtained in instances when more bubblers were added to the train, indicated that the quantity of SO_x found was proportional to the efficiency of the collector system being employed.

RESULTS AND DISCUSSION

Two methods with modification were employed for the estimation of HD as intact effluent from the small and large laboratory incinerators. These methods included GLC (11) and a number of modifications of a colorimetric method employing the DB3 [4-(4-nitrobenzyl)pyridine] reagent (12,13). GC-mass spectrometry and thin-layer chromatography (TLC) (14) were employed for qualitative identification of HD and potentially existing organic breakdown residues. The GLC determinations were made employing the flame photometric sulfur, hydrogen flame ionization, and electron capture detectors, with the sulfur and/or electron capture detectors being applied to most of the analyses.

The total HD recovered intact from the incinerators ranged from the detection threshold of less than 7×10^{-1} mg/cu m of air to as high as 544 mg/cu m of air. Only five of at least 65 satisfactorily operated experiments showed any measurable intact HD as passing through the incinerator. The usual experiment showed no HD coming through intact at the detection threshold of a total of 0.075 mg in bubblers containing 250 ml of solvent. This value represented less than 0.3 mg/cu m of HD in the total effluent air and product gases. These results indicated an incineration efficiency of better than 99.9994%.

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An Arrhenius plot of the data described by Garrett (9,10) gives a straight line described by the equation:

$$\log k = 9.25 - \frac{5500}{T} \quad (I)$$

where k is the rate constant and T is the absolute temperature. Assuming first-order dependence upon HD concentration as indicated in the above equation, the disappearance of HD may be seen as:

$$\frac{-dC}{dt} = kC \quad (II)$$

where C is the concentration of HD and t is time. Integration and rearrangement of equation II yields:

$$\frac{C}{C_0} = e^{-kt} \quad (III)$$

where C_0 is the concentration of HD at $t = 0$. Rearrangement of equation III gives an expression describing the incineration efficiency:

$$\frac{C_0 - C}{C_0} = 1 - e^{-kt} \quad (IV)$$

The present limit of sensitivity for the estimation of HD thus limits certainty of experimental incineration efficiency to 99.9994%, as mentioned above. Using equations I and IV, it can be calculated that for a residence time of 0.14 sec, a temperature of 485°C would allow attainment of the above incineration efficiency. At 800°C the calculated incineration efficiency would be $1 - e^{-18000}$ if steady-state residence time at temperature is continuously achieved. The weight of the calculation would certainly leave no doubt that at incineration temperature (800°C+) it should be possible to insure burning at efficiencies which are orders-of-magnitude higher than our experimentally determined 99.9994%.

In order to obtain the required information, analytical methods were adapted, improved, or developed to allow determination at different levels of sensitivity of intact HD or organosulfur products (11,13,14); sulfur and nitrogen oxides (nitrogen fixation) (13); and hydrogen chloride that might be present in effluent gases. Some of the developed or adapted methods (gas chromatography and colorimetry) proved to be applicable also to detection (for alarm) or measurement of intact HD from stack or at perimeter areas.

A summary of the data for intact HD and the calculated burning efficiencies as obtained in our two laboratory incinerators are shown in Table I. The data illustrating collection and determination of mineralization products are shown in Table II.

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TABLE I. DETERMINATION OF HD AS EFFLUENT FROM LABORATORY INCINERATION
(Analysis of bubbler samples by GLC)

No. of Runs	HD		Quantity/ Air Flow	Burning Efficiency	Residence Time (at 800°C)
	Burned	Found in Effluent ^a			
n	gm	mg x 10 ⁻³	mg/cu m	%	sec
10	0.13	< 1.3	< 15.	99.999+	1.70
3	0.13	< 25.	< 15.	99.77+ ^b	1.70
2	3.84	< 75.	< 0.7	99.98+	0.26
5	6.4	< 75.	< 0.7	99.999+	0.26
25	13.1	< 75.	< 0.3	99.9994+	0.14
3	13.1	< 300.	< 5.	99.998+ ^c	0.14
1	13.1	150,000.	544.	98.85 ^d	0.14
1	13.1	62,000.	225.	99.53 ^d	0.14

^a Threshold sensitivity as limited by available aliquots from bubblers.

^b Analysis of air samples taken immediately aft of furnace and condenser.

^c Results from less-sensitive colorimetric method = 99.998% efficiency.

^d Simulated flame-out, "cold" furnace tube; temperature from 500° to 600°C during run.

TABLE II. SUMMARY OF LABORATORY INCINERATOR EXPERIMENTS
(Simulated 3 gpm of mustard; 269 cfm of propane)

No. of Runs ^a	HD Burned gm	Analytical Recovery				Air Flow		Residence Time at 800°C sec	Oxygen, Supply/Demand ^b ratio
		SO ₂ %	SO _x ^b as SO ₂ %	HCl %	ft ³ /min	(cfm)			
5	0.13	90.3	91.6	98.1	1.00	(113,300)	0.25	9.94	
2	0.13	92.1	93.4	97.1	0.41	(46,450)	0.61	5.58	
2	0.13	91.5	93.9	98.1	0.34	(38,100)	0.77	3.34	
3	0.13	93.5	95.8	98.3	0.15	(16,800)	1.70	1.46	
5	0.13	96.5	96.6	100.1	0.11	(12,900)	2.22	1.13	
2	6.4	89.6	97.2	97.9	6.8	(13,600)	0.26	1.32	
2	13.1	76.3	96.9	98.9	12.25	(13,000)	0.14	1.26	
6	13.1	96.1	96.6	99.2	12.25	(13,000)	0.14	1.26	
11	13.1	87.9	97.4	99.1	12.25	(13,000)	0.14	1.26	
2	13.1	87.9	97.4	98.8	15.10	(16,100)	0.12	1.56	

^a Results in each category represent the mean of determinations.

^b SO_x = sulfur oxides (SO₂ + SO₃) calculated as SO₂.

The studies described below were designed to approach the real plant operation while assisting in the selection of scrubber material, and defining constants and criteria for optimum scrubber operation under design conditions.

Two series of experiments were run in which calcium carbonate (CaCO_3) slurries served as the alkaline scrubbers. In the first series, an aqueous 20% calcium carbonate slurry was substituted for the sodium hydroxide systems that had been used in previous experiments. The hydrogen chloride (now hydrochloric acid) and water formed on incineration were collected for the most part in a pre-scrubber that also served to cool the gas stream. Under these circumstances relatively little HCl reached the calcium carbonate slurry. In these runs calcium carbonate proved to be very poor for absorbing sulfur oxides, retaining only about 30% of the SO_x expected from the incinerator effluent. This poor efficiency could be readily explained on the basis of the relatively low solubility of calcium carbonate and also calcium sulfite in water.

In the second series of runs, a single, ice-cooled bubbler containing 30% calcium carbonate slurry was used to collect both the sulfur oxides and the hydrogen chloride. Under these conditions, the single bubbler was found to absorb at least 86% of the hydrogen chloride and increased the SO_x retention from 30% to at least 85%. The effectiveness of this system could be explained by the reaction of HCl with the poorly soluble CaCO_3 to produce the significantly more soluble, and thus more available, calcium chloride (CaCl_2). The SO_x or their acids upon reaction with CaCl_2 formed the relatively insoluble calcium sulfite and sulfate and released hydrochloric acid to dissolve additional calcium carbonate.

Sodium hydroxide was used in most of the HD incineration experiments as a final trap and also as the main trap for collection of the sulfur oxides. For purposes of incinerator-scrubber design, information was required on the quantities of carbon dioxide (in addition to the sulfur oxides and hydrogen chloride) that might be collected in the scrubber. Also needed was information on the pH requirements for retaining the incinerator effluent and collecting the scrubber salts. At the same time, the minimum pH required to maintain the wet or dry scrubber salts in an odorless state was to be ascertained. This latter test was to insure that the sulfur oxides would remain in odorless salt form under storage conditions.

In other experiments, the hot effluent gases were cooled and the hydrogen chloride and sulfur oxides were collected in single, ice-cooled bubblers. One was charged with an excess of aqueous sodium hydroxide (A); and another, with a slight deficiency of base (B). After typical incinerator runs the hydrogen chloride, sulfur oxides, carbon dioxide, and pH were determined on the bubbler

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contents. Analysis of the bubbler contents of two experiments (A and B) are summarized in Table III.

TABLE III. ANALYSIS OF TWO SODIUM HYDROXIDE SCRUBBER BRINES

<u>Scrubber charge, collection products and final pH</u>	<u>A</u>	<u>B</u>
mmoles NaOH added	325.0	233.8
mmoles SO ₂ collected as NaHSO ₃ and Na ₂ SO ₃	65.2	71.8
mmoles SO ₃ collected as Na ₂ SO ₄	5.8	9.9
mmoles HCl collected as NaCl	136.0	149.4
mmoles CO ₂ collected as NaHCO ₃ and Na ₂ CO ₃	53.3	1.0
Final pH (at termination of experiment)	7.79	6.41

Of importance in the containment of effluent from the atmosphere or in dried salt cake is the elimination of odor. No odor was detected in the pH 7.79 liquor, or in the dried solids after treatment under an infrared lamp. The pH 6.41 liquor had no noticeable odor when cool. However, when this liquor was heated, a definite sulfur dioxide odor was noted. It was ascertained that neither the liquor nor the dried cake had a detectable odor when the pH was maintained at 7.5 or above.

Carbon dioxide and water represent the largest quantity of combustion products from the incineration of HD. It was planned that sodium hydroxide would be fed to the scrubber to absorb all acidic mineralization products. Because of the relatively large amount of carbon dioxide present in the effluent gases, the sodium hydroxide will be converted to sodium bicarbonate in the scrubber (Table III). Therefore, an aqueous 2M sodium bicarbonate slurry was tested and used in most of the large-scale laboratory incinerator experiments as the absorbing solution. The sodium bicarbonate dissolved and released carbon dioxide as it was consumed by the more strongly acidic hydrogen chloride and sulfur oxides. The absorption efficiency for hydrogen chloride and sulfur oxides in sodium bicarbonate solution was equal to that of a sodium hydroxide solution. The analysis of a typical aqueous sodium bicarbonate scrubber is shown in Table IV. A single, ice-cooled bubbler was used in this experiment.

TABLE IV. RECOVERIES OF HD COMBUSTION PRODUCTS:
RESULTING SALT SOLUTIONS IN BICARBONATE SCRUBBER^a
 (Calculated versus found)

<u>Products, Scrubber Charge and pH Range</u>	<u>Calculated</u>	<u>Experimental^b</u>
Hydrogen chloride, %	100.0	99.1
Sulfur dioxide, %	85.0	83.0
Sulfur trioxide, %	15.0	16.0
Sodium bicarbonate charged in scrubber, gm	29.64	29.0
pH (initial → final)	8.5 — 8.0	8.5 — 7.85
As salts, %:		
NaCl	42.4	42.3
Na ₂ SO ₄	7.7	8.5
NaHSO ₃	4.4	6.1
Na ₂ SO ₃	33.5	32.7
NaHCO ₃	11.8	10.3
Na ₂ CO ₃	0.1	0.04

^a Sodium bicarbonate selected as buffer for pH 8 operations.

^b Represents five separate runs.

CONCLUSIONS

On the basis of laboratory investigations made on two all-glass incinerators operating at 800°C (1472°F), it was found experimentally that a mustard destruction efficiency of better than 99.9994% can be assured. The level cited for efficiency is limited only by the relatively small amount of mustard burned (up to 13.1 gm/experiment) as reflected in the sensitivity threshold for mustard detection.

Air flows tested represented oxygen supply to oxygen demand ratios of 1.13 to 9.94. The 1.13 ratio was sufficient for complete combustion. Experimental residence time of mustard at furnace temperature ranged from 0.14 to 2.22 sec. Practically all of the mustard was mineralized, even at a residence time as low as 0.14 sec. Calculations based on steady-state conditions at this residence time indicate attainable incineration efficiencies that are orders of magnitude higher than that proved experimentally.

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Scrubber tests under direct absorption conditions (no recycle) showed that sodium hydroxide and sodium bicarbonate allowed collection efficiencies of 99%+ of the hydrogen chloride and 95%+ of the sulfur oxides (sulfur dioxide and sulfur trioxide). Chemical analysis of scrubber salt composition and subjective odor tests versus pH, indicated that absence of odor can be assured from hot salt solution or dry salt, if the scrubber system is maintained above pH 7.5.

Analytical methods were adapted or developed as an adjunct to this study. All of these methods (trace determination of HD, analysis for SO_x, NO_x, HCl and CO₂) are applicable to laboratory control of incinerator effluent. In addition, studies with GLC suggested this type of system for direct indication of intact mustard in stack effluent (alarm). The colorimetric DB3 method was suggestive of a system for bubbler sampling for perimeter or enclosure monitoring.

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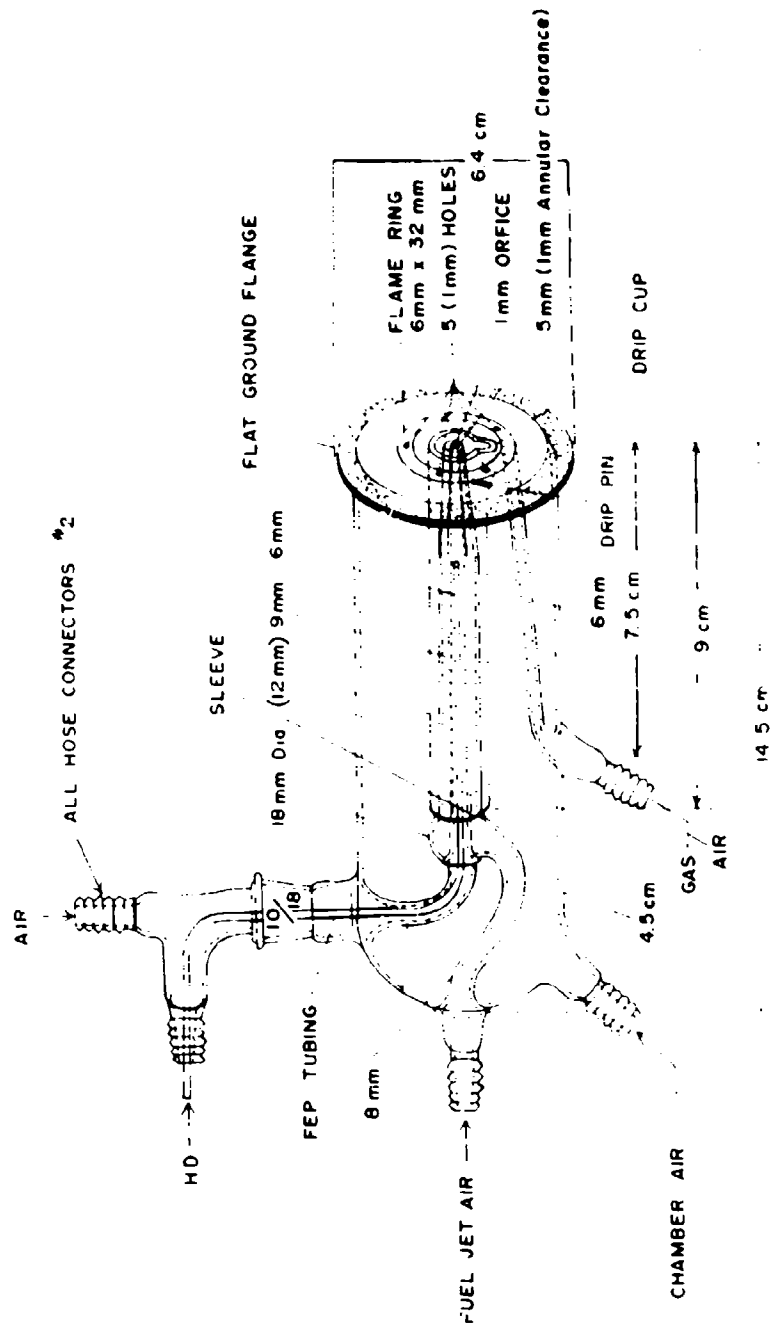


Figure 1. Laboratory Burner

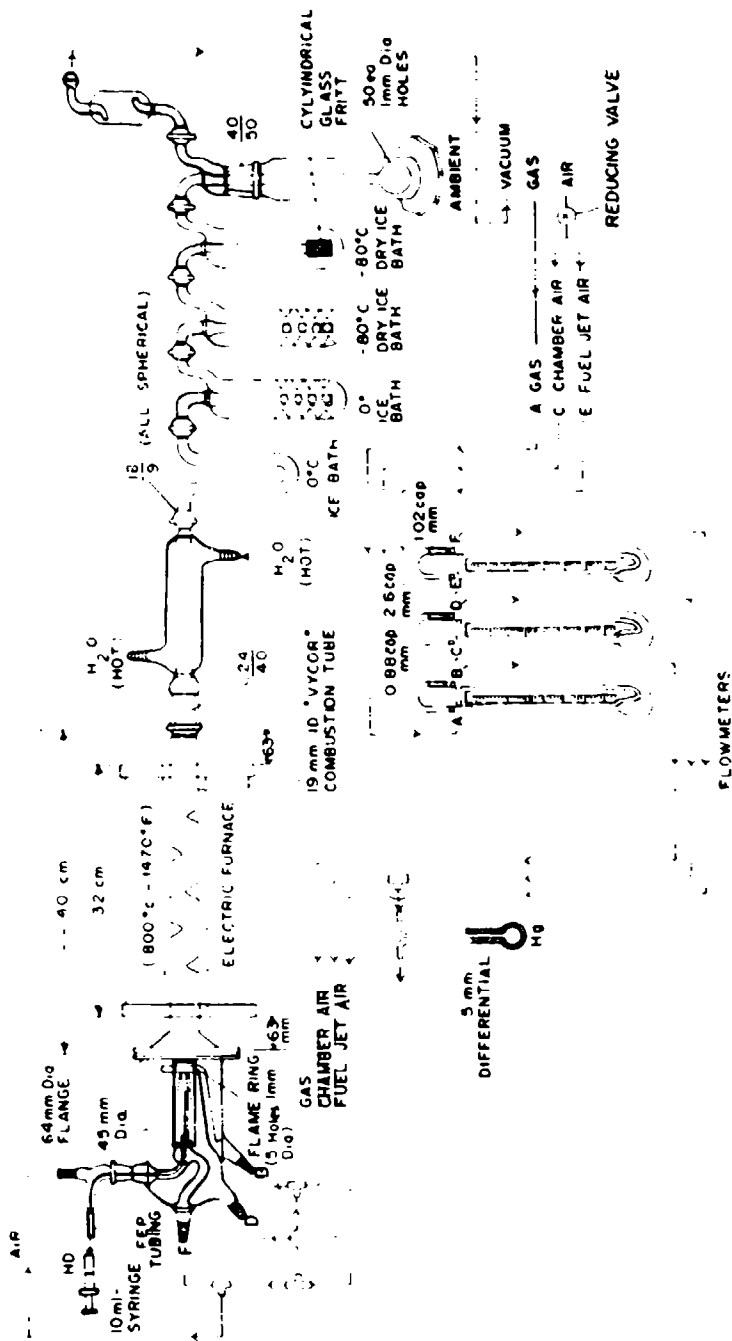


Figure 2. Incinerator and Collector Assembly