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RESEARCH INSTITUTE

EXPLORATORY RESEARCH ON HIGH
ENERGY PROPELLANT SYSTEMS



A. V. GROSSE, T. R. FLINT, S. LIPSCHUTZ
AND C. S. STOKES

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**FINAL REPORT
ON
EXPLORATORY RESEARCH ON HIGH
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January 1961
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TABLE OF CONTENTS

	<u>Page</u>
Foreword	
Summary	
Introduction	1
Scope of Work	1
I. Modification of the Apparatus	3
A. Test Stand	3
B. Flowmeters	6
C. Instrumentation	10
D. Injectors	13
E. Nozzle Inserts	21
II. Theoretical Performance Calculations	22
A. Boron Hydrides-Fluorine System	22
1. Pentaborane-Fluorine System	24
2. Diborane-Fluorine System	27
B. Selected High Energy Systems	27
III. Performance Tests	31
A. JP-4-LOX	31
B. RP-1-LOX	37
C. Hydrogen-Oxygen	39
D. n-Hexane-Oxygen	40
E. Pentaborane-Fluorine	41
IV. Ozone Fluoride Investigations	46
A. Properties of O_3F_2	46

TABLE OF CONTENTS
(cont'd)

	<u>Page</u>
B. Preparation of O_3F_2	46
C. Handling Procedure of O_3F_2	48
D. Solubility of O_3F_2 in LOX	49
E. Initial Studies Using 0.05% O_3F_2 in LOX- Open Cup Tests	49
F. Micro-Rocket Tests Using 0.1 Wt.% O_3F_2 in LOX	51
G. Micro-Rocket Test Using 0.05 Wt.% O_3F_2 in LOX	52
H. O_3F_2 Solution Compatibility Tests	54
Conclusions and Recommendations	59

LIST OF FIGURES

1. Propellant Evaluator Schematic Diagram	2
2. Test Stand -Early Design	4
3. Redesigned Test Stand	5
4. Circuit Diagram - Electrical System	7
5. Combustion Chamber and Thrust Stand Close-up	8
6. Sectional View of Thrust Chamber	9
7. Master Control Panel	11
8. Brush Recorder and Minneapolis-Honeywell Circular Chart Recorder	12
9. Injectors - Type 1 and Type 4	14
10. Injector - Type 4A	15
11. Injector - Type 5	16
12. Injector - Type 6	17

LIST OF FIGURES
(cont'd)

	<u>Page</u>
13. Injector - Type 7	18
14. Injector - Type 8	19
15. Scheme for Calculating Equilibrium Composition of Combustion Products for Borane-Fluorine Systems.	26
16. Impulse Data for Pentaborane-Fluorine Reaction	28
17. Chamber Temperature Data for Pentaborane-Fluorine System	29
18. Average Specific Heats Ratio of Combustion Products of the Pentaborane-Fluorine System Vs. O/F	30
19. Pentaborane-Fluorine System - Run #03-61	43
20. Reaction Vessel for Preparation of O_3F_2	47
21. Solubility-Grams O_3F_2 /100 Grams Liquid O_2	50

LIST OF TABLES

I. Injector Data	20
II. Nozzle Inserts - 10 lb. Thrust	22
III. Physical Properties of the Boron Hydrides	23
IV. Diborane-Fluorine System	31
V. Theoretical Performance of Some High Energy Systems for a Chamber Pressure of 300 psig.	32
VI. JP-4 Fuel-Liquid Oxygen System	33
VII. RP-1 Fuel-Liquid, Oxygen System	38
VIII. Gaseous Hydrogen-Liquid Oxygen System	40
IX. n-Hexane-Liquid Oxygen System	41

LIST OF TABLES

	<u>Page</u>
X. Pentaborane-Liquid Fluorine System	45
XI. O_3F_2 Solubility in LOX	49
XII. O_3F_2 Open Cup Ignition Tests	51
XIII. Experiments Using 0.1 Wt.% O_3F_2 in LOX	52
XIV. 0.05% O_3F_2 in LOX	53
XV. Vaporization Compatibility Tests - 0.05% O_3F_2 Solution	55
XVI. Vaporization Compatibility Tests - 0.10% O_3F_2 Solution	56
XVII. Immersion Compatibility Tests - 0.10% O_3F_2 Solution	58

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FOREWORD

This is the final report on exploratory research on high energy propellant systems by the Research Institute of Temple University for the U. S. Army Ballistic Missile Agency under Contract No. DA-36-034-ORD-3152. It covers the work done from January 4, 1960 to March 4, 1961.

~~SUMMARY~~

↙
A versatile liquid propellant evaluation system was perfected which is suitable for use with laboratory quantities (50-200 ml.) of regular or cryogenic fuels and oxidizers.

Tests with the RP-1-LOX system provided values for specific impulse on the order of 90% of theoretical. ~~(Table VII)~~.
Data from a test of the pentaborane-fluorine system yielded a specific impulse which was 83% of theoretical. ~~(Table X)~~.

A dilute solution (0.05%) of O_3F_2 in liquid oxygen was used as an oxidizer in the apparatus and proved to be hypergolic with gaseous hydrogen, UDMH, UDMH-DETA, ethyl alcohol, propane, n-hexane, JP-4, and mixtures of JP-4 and UDMH. ~~(Tables XII-XIV)~~.

Compatibility tests (by simple contact) showed that dilute O_3F_2 -LOX solutions do not react hypergologically with most materials commonly used in LOX systems. ~~(Tables XV and XVI)~~.
↑

INTRODUCTION

The work reported herein is a continuation of the research performed under Contract Nos. DA-36-034-ORD-2713 and DA-36-034-ORD-2987.

The work as described in the final reports^{1,2} of the above contracts included system feasibility tests, and investigation of physical and chemical properties of the proposed materials. The information presented in this report describes the latest propellant evaluation studies making use of a perfected micro-rocket system, Fig. 1.

SCOPE OF WORK

The purpose of this research is the investigation of various systems as high energy propellants. Initial tests will include firing of the experimental system in a small micro-combustion unit of approximately 20 lb. thrust. The micro-burner shall be improved by development and by use of a thrust measuring device. Further development studies shall be made on the apparatus to modify and simplify it for possible test use with storable type propellants.

Prior to the use of the various propellants, proper handling procedures will be determined so that proper valves, piping and other items can be incorporated in the system.

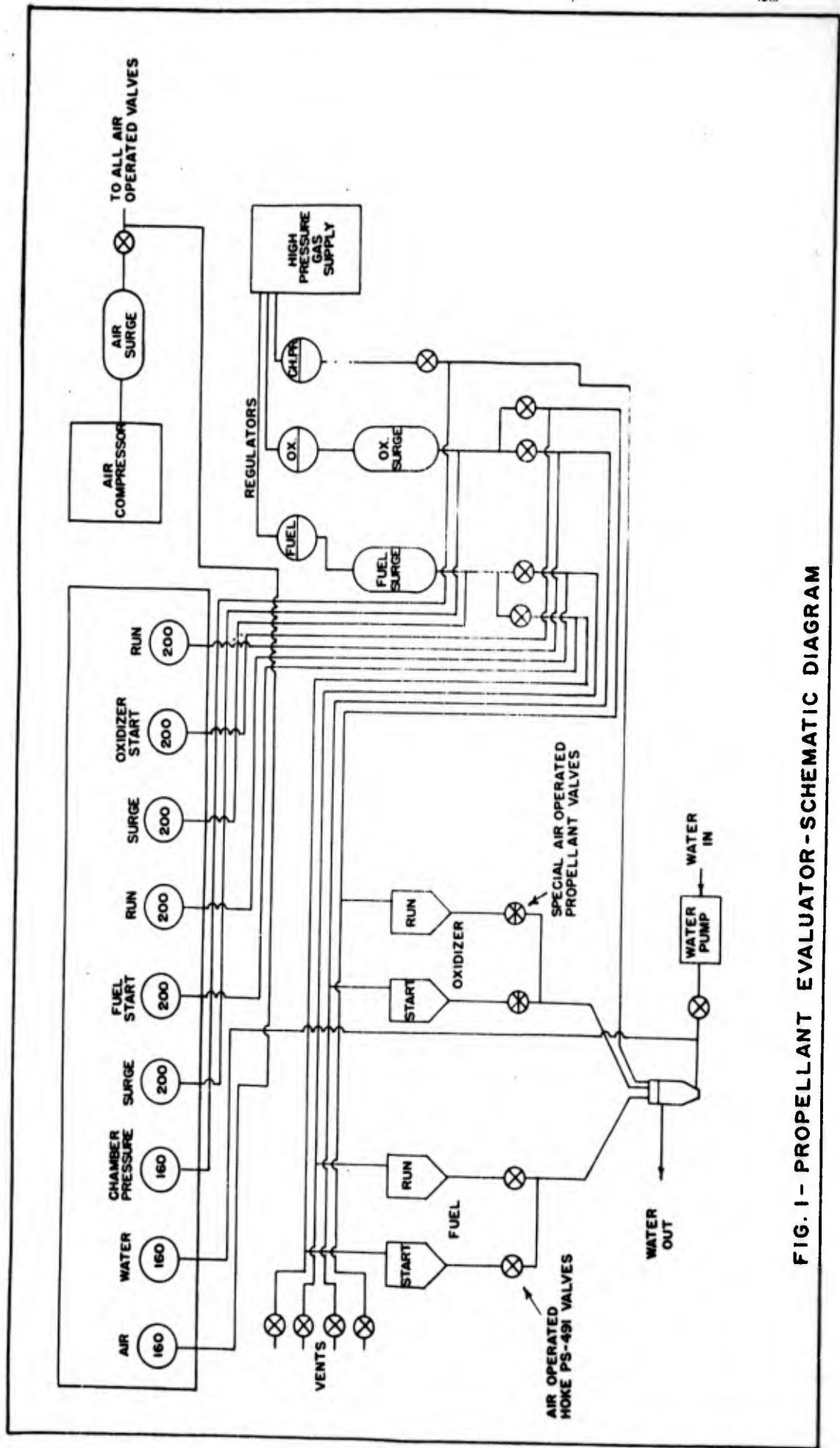


FIG. 1 - PROPELLANT EVALUATOR - SCHEMATIC DIAGRAM

I. MODIFICATION OF THE APPARATUS

A number of modifications and additions were made on the apparatus to increase its flexibility and to improve the caliber of the data obtained.

A. Test Stand

The original test stand, Fig. 2, of the micro-combustion unit was redesigned and rebuilt with the following improvements:

1. A new basic test stand frame provided improved accessibility for propellant loading procedures, future modifications, etc., Fig. 3.
2. Cooling troughs were integrated for more efficient cooling. Two troughs are used, one cooling the oxidizer tanks and oxidizer feed valves, the other cooling the #2 fuel tank and feed valve.
3. A special low-temperature feed valve, (manufactured by Pressure Products Industries, Hatboro, Pa.), similar in design to those already in use as oxidizer feed valves, was installed as the #2 fuel feed valve. This valve is useable with any cryogenic or corrosive fuel.

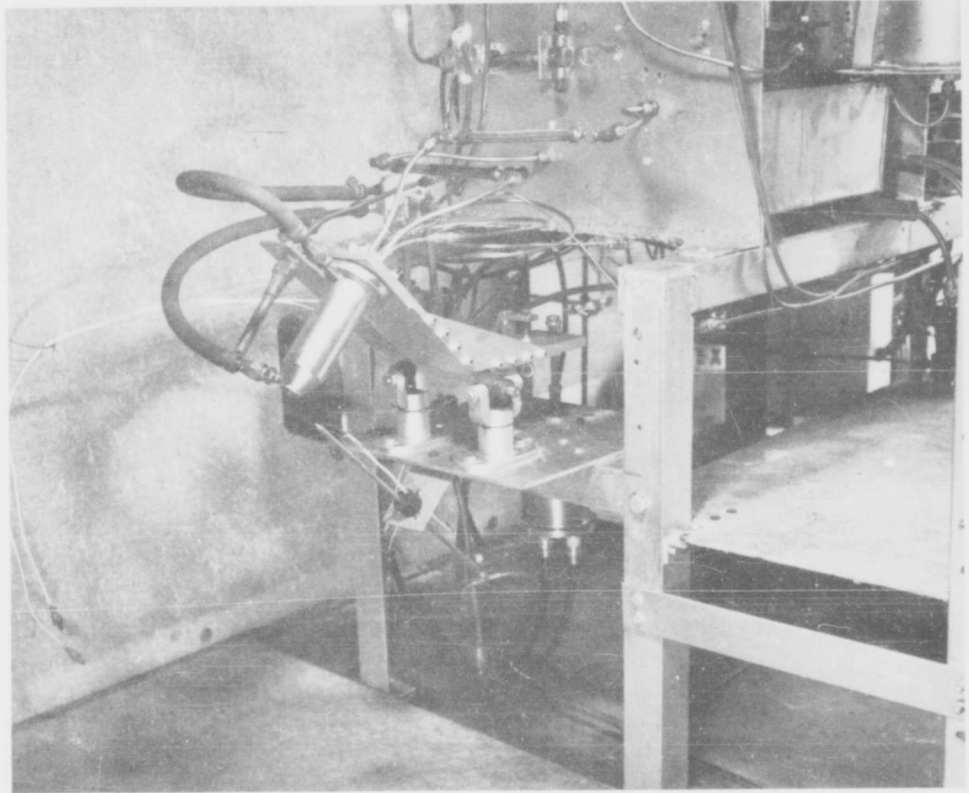


FIG. 2 - TEST STAND - EARLY DESIGN

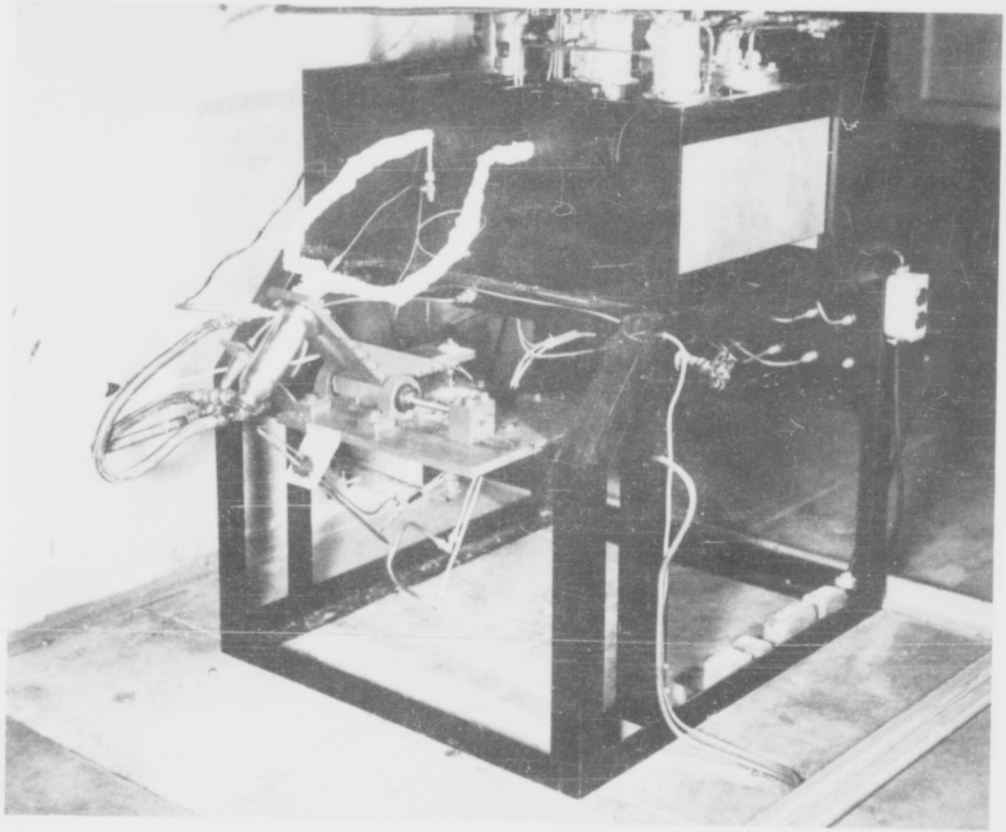
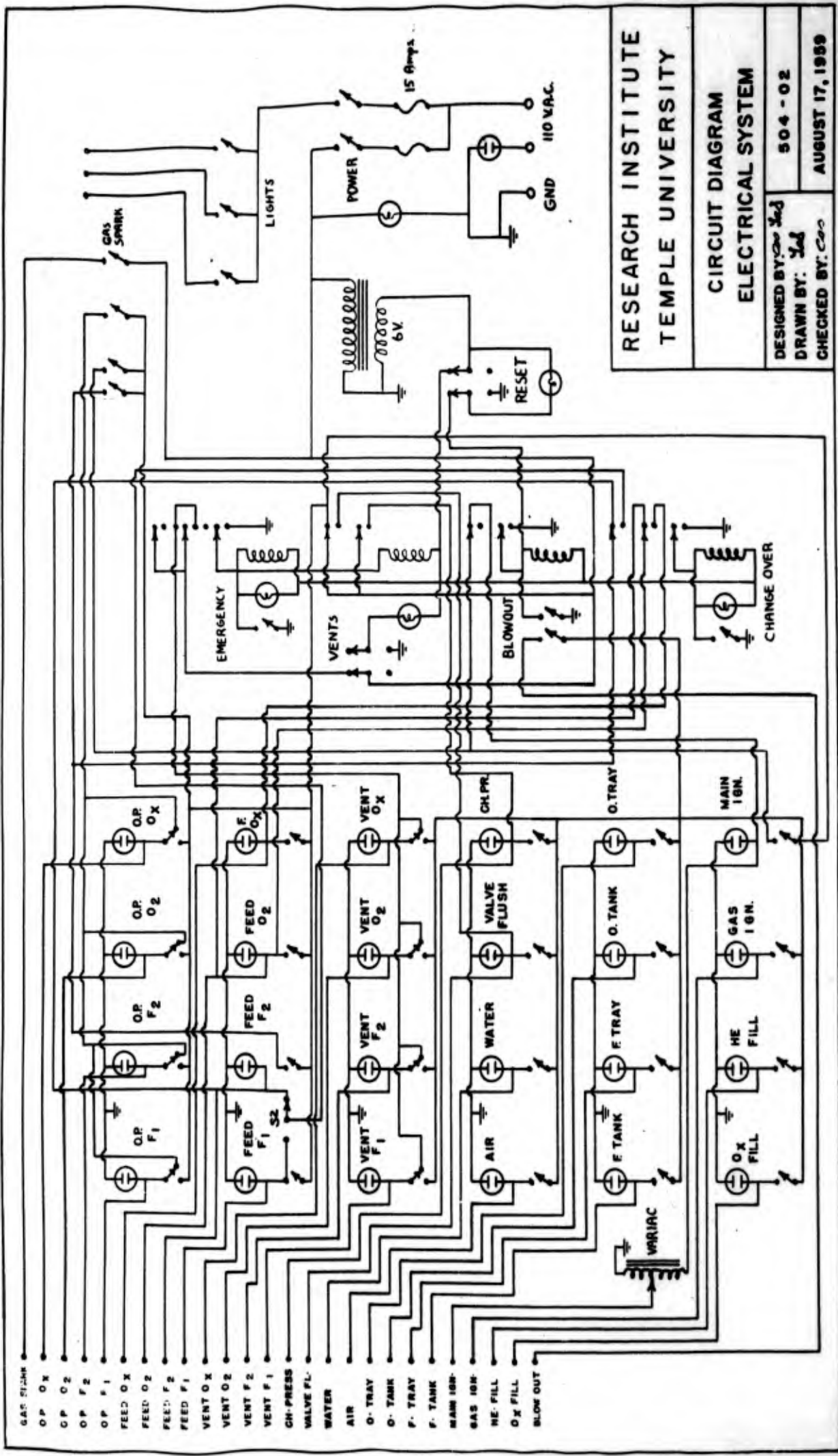


FIG. 3 - REDESIGNED TEST STAND

4. All solenoid valves controlling the air supply to air operated valves were removed from the test stand proper and installed on a wall-mounted subpanel, again providing greater accessibility to the test stand.
5. Electrical junction boxes and wiring, wherever possible, were mounted on the subpanel in order to minimize electrical failure in the event of fire or explosion. A complete circuit diagram is shown in Fig. 4.
6. A redesigned thrust stand, Fig. 5, was incorporated in the apparatus utilizing a torsion bar and Daytronic micro-deflection detector (see instrumentation section). The thrust stand was calibrated by applying a known force through the axis of the motor, Fig. 6. This force was obtained by suspending known weights on a fine wire connected to the motor and passing over a precision ball bearing pulley.

B. Flowmeters

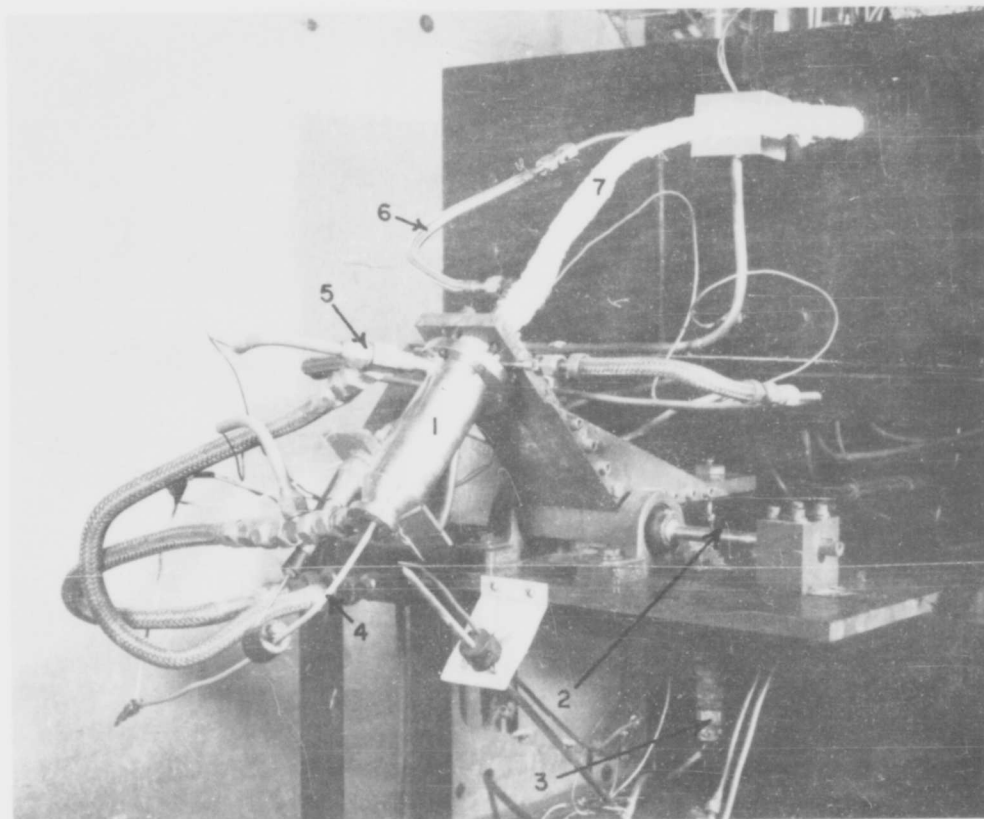
Indirect measurements of fuel and oxidizer flow rates were made by measuring the flow of helium overpressuring gas with



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CIRCUIT DIAGRAM
ELECTRICAL SYSTEM

DESIGNED BY: J.S.J.
DRAWN BY: J.S.J.
CHECKED BY: C.S.S.
504-02
AUGUST 17, 1959



1-COMBUSTION CHAMBER 2-TORSION BAR 3-MICRO-DEFLECTION
DETECTOR AND TRANSMITTER 4-SPARK IGNITOR 5-THERMOCOUPLE
6-FLEXIBLE FUEL LINE 7-INSULATED FLEXIBLE OXIDIZER LINE

FIG. 5 - COMBUSTION CHAMBER AND THRUST STAND CLOSE-UP

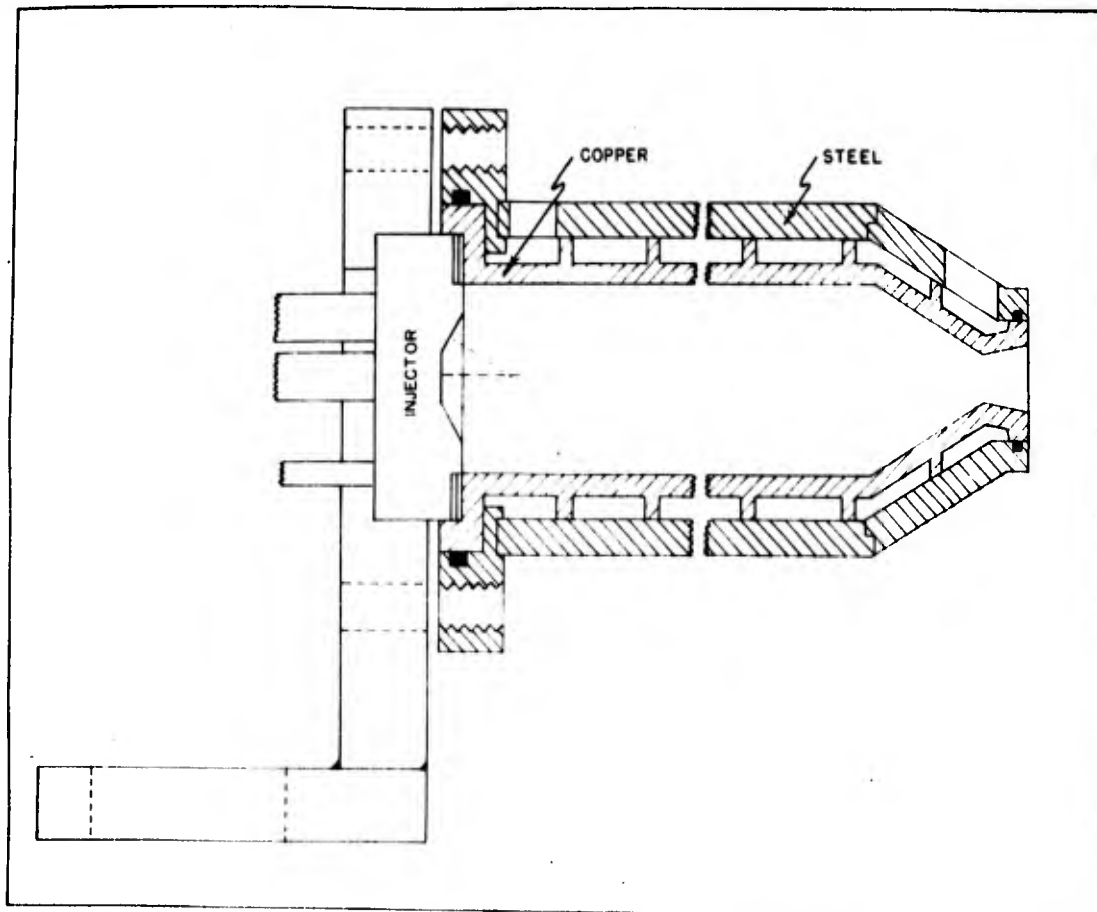


FIG. 6 SECTIONAL VIEW OF THRUST CHAMBER

two Brooks Co. Model FV-1110-XXD-S rotameters. Signals from the rotameter were recorded on a Brush Instrument.

Extreme oscillations of the rotameter floats called for the addition of a damping fluid to the float well of each meter. The fluid found most suitable in terms of viscosity, density, chemical properties, and damping ability was Kel-F Alkane 464, manufactured by M. W. Kellogg Co.

It was found that helium bubbles became entrapped in the hollow magnetic core of the rotameter floats resulting in error due to increased float buoyancy. Capping of the float bottom has eliminated this error.

C. Instrumentation

The operation of the propellant evaluator was activated by the master control panel, Fig. 7.

All critical experimental measurements were recorded on a Brush 4-channel ink recorder, a Minneapolis-Honeywell 3-pen 1-rpm circular chart recorder, Fig. 8, and an 8-channel Minneapolis-Honeywell Visicorder.

Propellant flow rates, measured with the Brooks rotameters, were recorded on the Brush recorder. The output signal of the rotameter was amplified with a Brush medium gain amplifier.

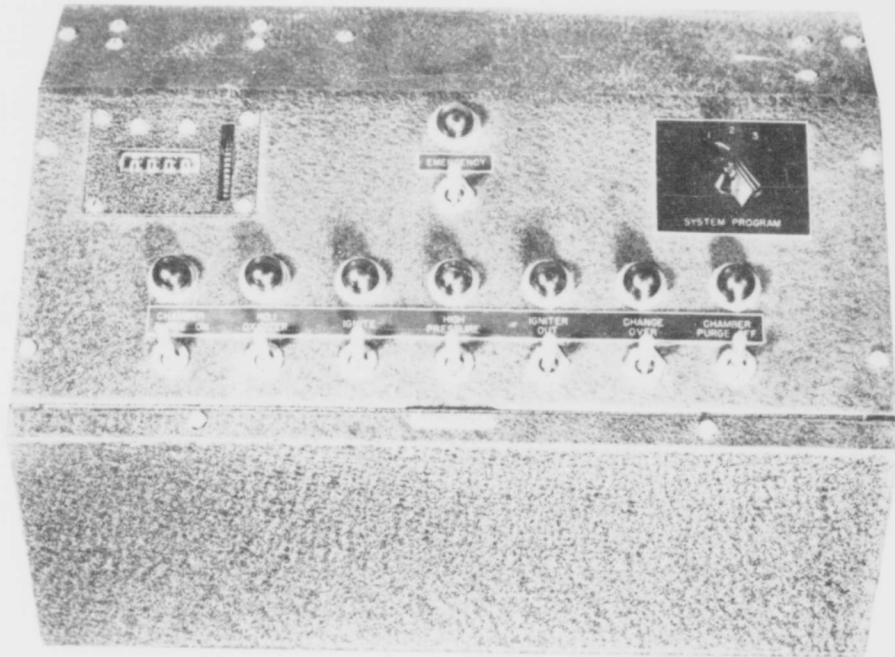


FIG. 7 - MASTER CONTROL PANEL

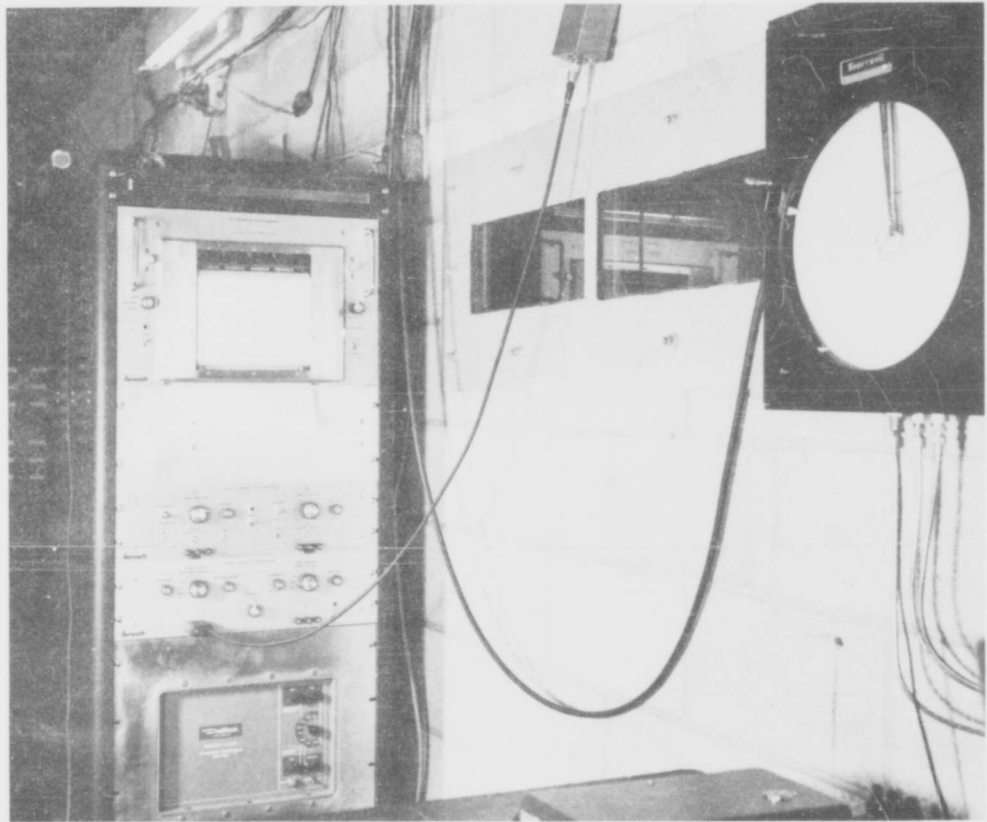


FIG. 8 - BRUSH RECORDER AND MINNEAPOLIS-HONEYWELL
CIRCULAR CHART RECORDER

Thrust measurements were picked up on a Daytronic micro-deflection detector and transmitter coupled to the Brush recorder through a Daytronic amplifier.

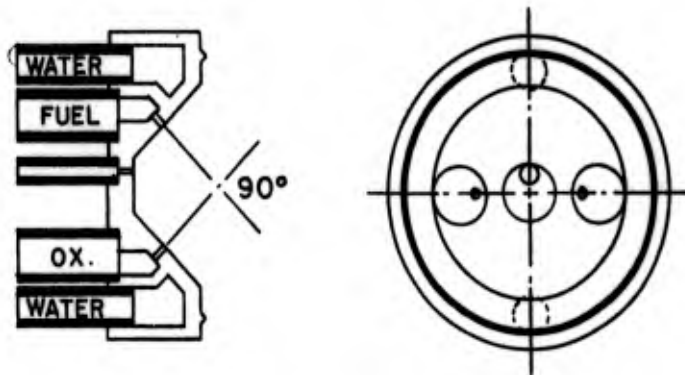
The chamber cooling water temperature rise was detected with a copper-constantan thermocouple. The resultant voltage was fed to the Minneapolis-Honeywell Visicorder.

Propellant tank over-pressures and chamber pressure were recorded on the Minneapolis-Honeywell circular chart recorder.

A Hallemore transistorized television camera and monitor permitted close-up visual observation of the thrust chamber nozzle during operation.

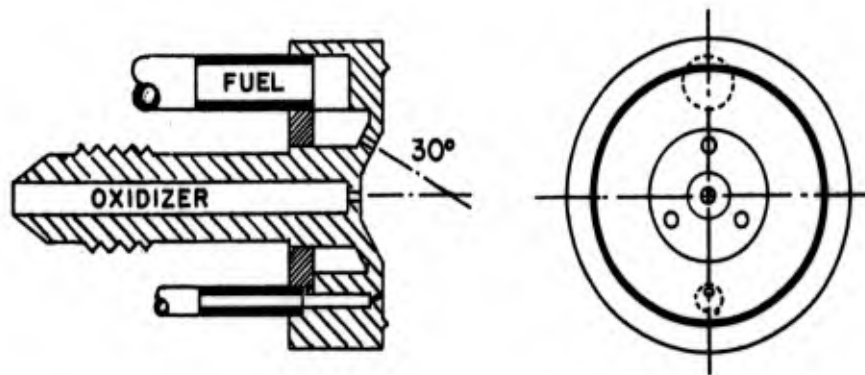
D. Injectors

Figures 9-14 show the injectors used in this work. Table I lists the essential facts about each. For the initial five runs (03-1 to 03-5) a type 4 injector was used. This was a water cooled injector with single peripheral oxidizer and fuel ports. The type 4 injector gave good performance but the flow in the ports appeared to be in the transition range between laminar and turbulent flow. This resulted in unpredictable flow rates over the operating pressure range. More predictable flows were obtained with a type 4A design using two pairs of oxidizer and fuel ports with separately impinging streams.



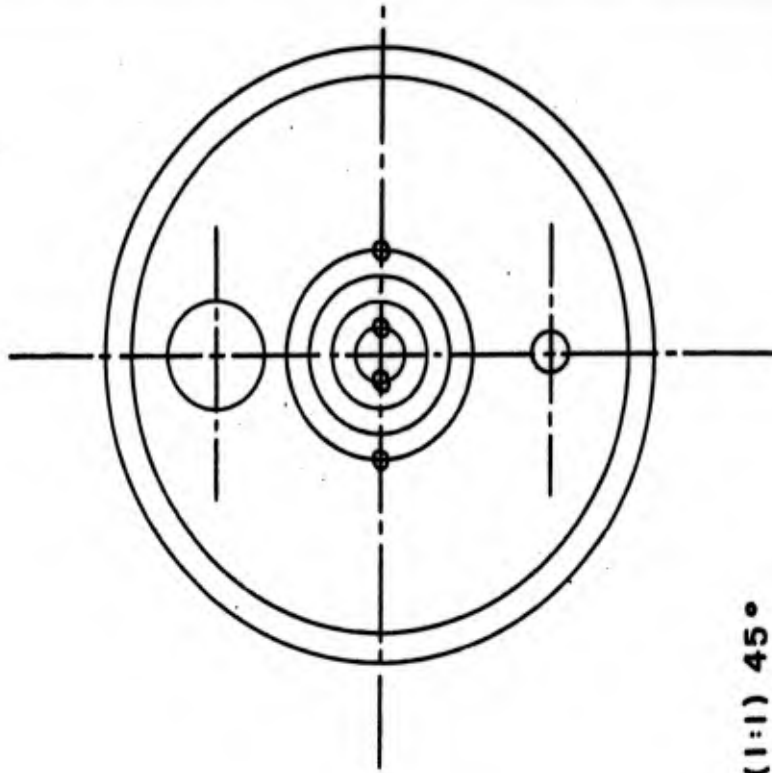
INJECTOR - TYPE 4

SCALE - 1:1



INJECTOR - TYPE 1

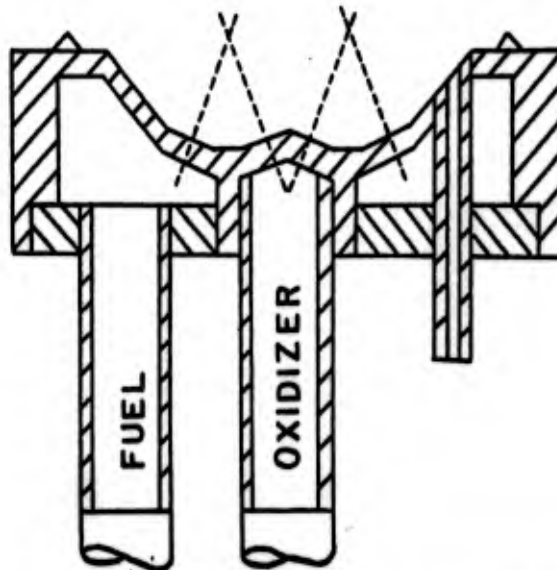
FIG. 9

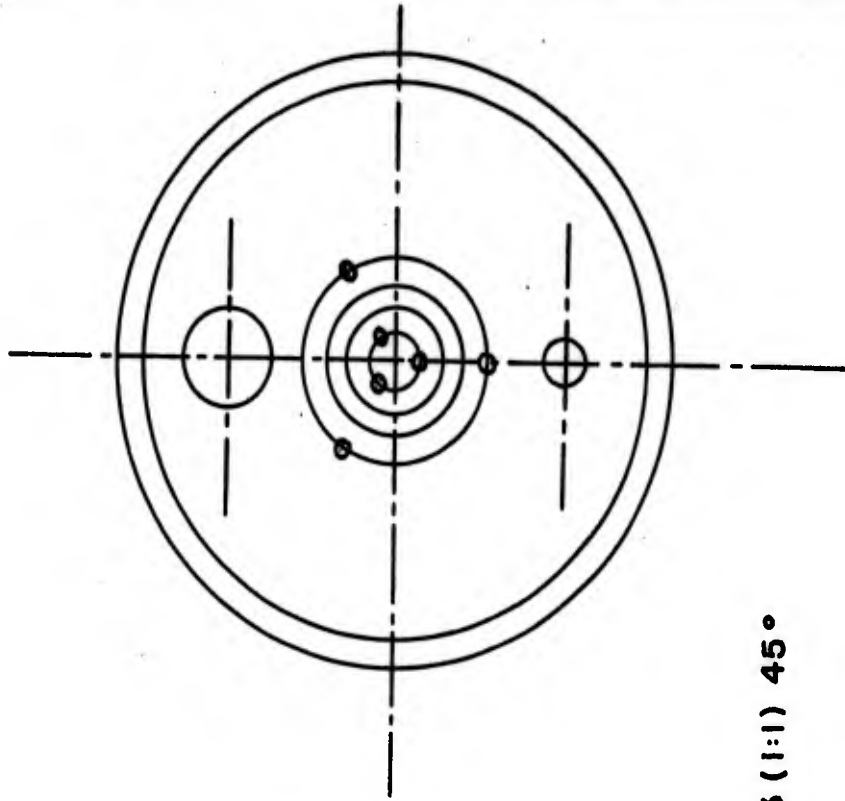


SCALE 2:1

INJECTOR TYPE 2(1:1) 45°
TYPE 4 A

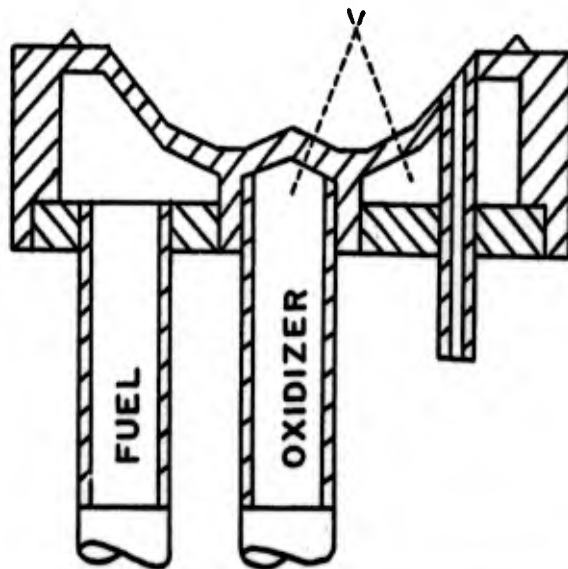
FIGURE 10

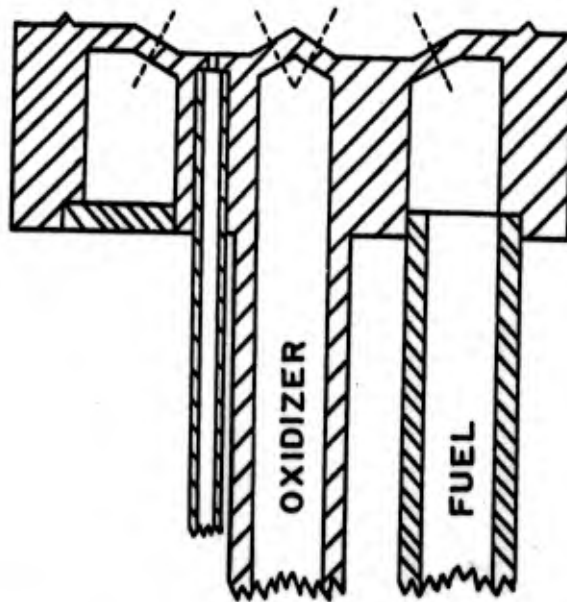
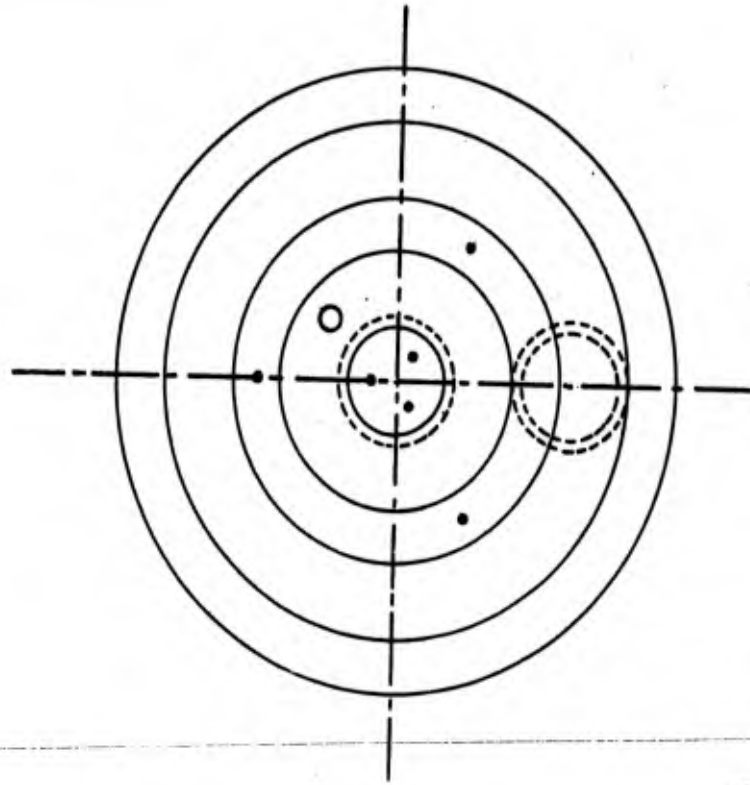




SCALE 2:1
INJECTOR TYPE 3 (1:1) 45°
TYPE 5

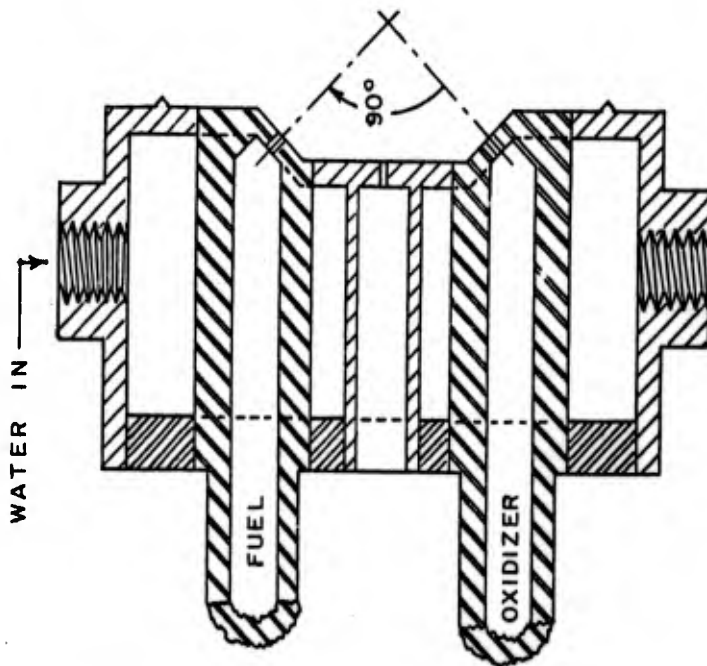
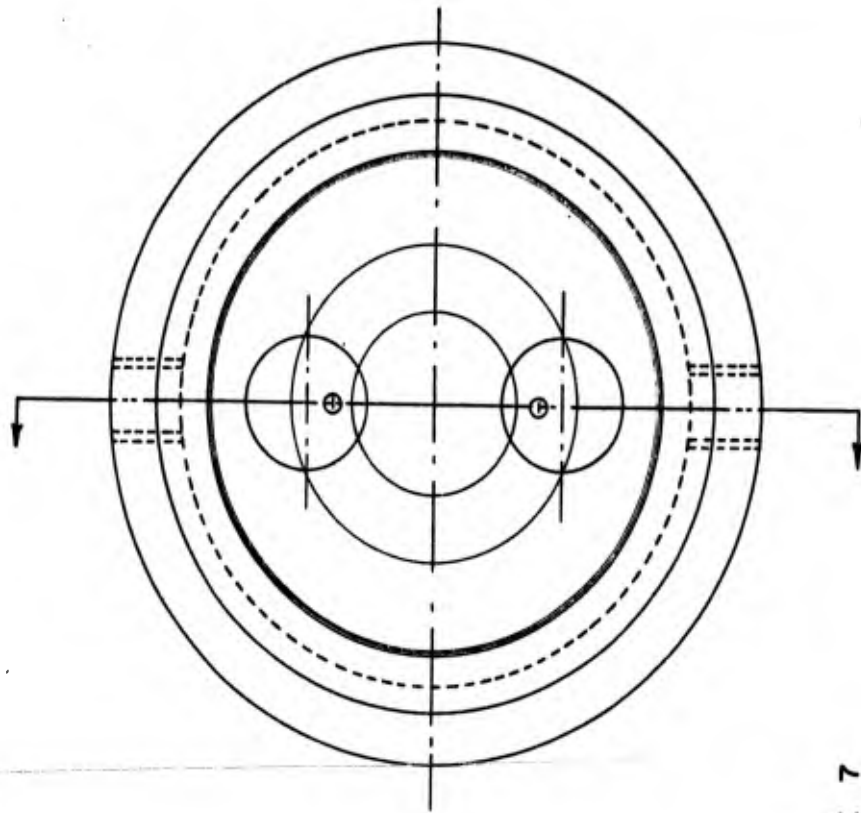
FIGURE 11





SCALE 2:1
INJECTOR TYPE 3 (1:1)
TYPE 6

FIGURE 12

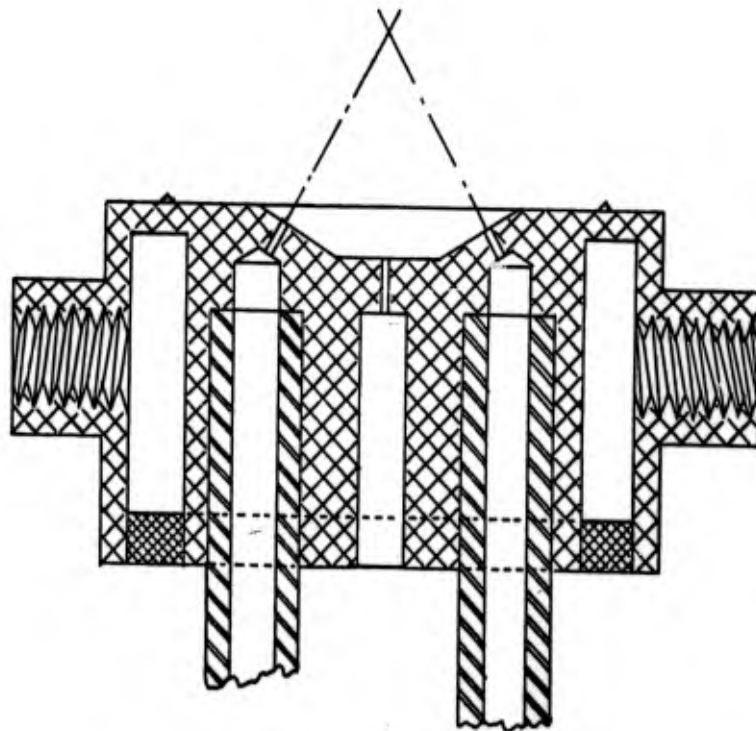
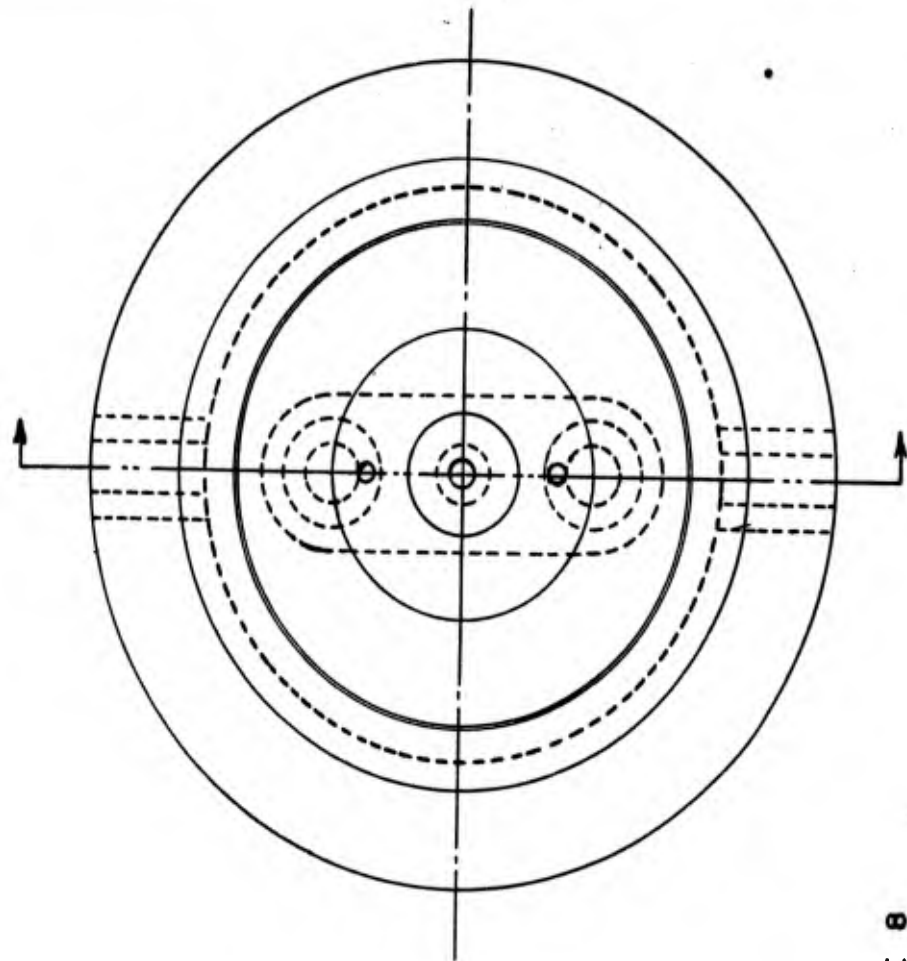


INJECTOR - TYPE 7

1:1 - 90°

SCALE - 2:1

FIGURE 13



INJECTOR TYPE 8

1:1 - 60°

FIG. 14

TABLE I

<u>Type</u>	<u>Model</u>	<u>Angle of Impingement</u>	<u>Fuel No. and Size of Holes</u>	<u>Oxidizer No. and Size of Holes</u>	<u>System</u>
1	1	60°	3-0.018"	1-0.033"	JP-4-LOX
1	2	60°	3-0.018"	1-0.042"	"
2	1	90°	2-0.018"	1-0.033"	"
3	1	130°	2-0.018"	1-0.033"	"
4	1(a)	45°	1-0.024"	1-0.038"	"
4A	1A, 1B(b)	22.5°	2-0.016"	2-0.0225"	"
4A	2	22.5°	2-0.016"	2-0.025"	"
4	3(a)	45°	1-0.055"	1-0.038"	H ₂ -O ₂
5	1	45°	3-0.0145"	3-0.025"	JP-4-LOX
6	1(c)	45°	3-0.0145"	3-0.025"	"
6	2	45°	3-0.0135"	3-0.028"	B ₅ H ₉ -F ₂
6	3	45°	2-0.0135"	2-0.033"	"
6	4	45°	2-0.0156"	2-0.0225"	"
6	5	45°	2-0.0180"	2-0.0260"	"
7	1(a)	90°	1-0.0240"	1-0.0360"	"
8	1(a)(d)	60°	1-0.0210"	1-0.0360"	"

(a) water cooled

(b) 1A - 1/4" SS tubing feed lines soldered to injector

1B - 1/4" SS Swagelok fittings used as feed lines

(c) Integral chamber pressure tap

(d) Brass injector

For the hypergolic ignition tests using O_3F_2 - LOX mixtures with JP-4 fuel, a type 1 injector was unsuccessful. With the two separately impinging oxidizer streams of injector type 4A it was also difficult to establish a hypergolic ignition. Type 5 injector was designed to improve the mixing of the O_3F_2 -LOX-JP-4 system. The three pairs of oxidizer and fuel ports in this injector seemed to achieve this purpose, since hypergolic ignitions were easily accomplished with O_3F_2 in LOX and JP-4 fuel.

Leaks at the soldered joints in the face of the type 5 injector led to the design of the type 6 injector for the JP-4-LOX system. The face of the type 6 injector had no soldered joints and had an integral chamber pressure tap.

Model Nos. 2,3,4 and 5 of type 6 injector were all unsuccessful designs of an injector for the pentaborane-fluorine system. They were all similar except for the size and number of fuel and oxidizer ports. It was found that these uncooled injectors caused thermal decomposition of the incoming pentaborane before entering the combustion chamber.

Types 7 and 8 water-cooled injectors were designed for better heat transfer to eliminate this decomposition.

E. Nozzle Inserts

A nozzle insert for each propellant system under investigation was designed and fabricated. These nozzles were

designed for the optimum thermodynamic expansion and for high heat transfer from the walls to eliminate dimensional instability from melting and erosion.

Table II lists the nozzle inserts designed for use with the propellant systems under investigation.

TABLE II

Nozzle Inserts - 10 lb. Thrust

<u>System</u>	<u>Atm. (in²)</u>	<u>ε (Area ratio)</u>
JP-4-LOX	.0857	1.800
UDMH-fluorine-LOX	.0641	1.825
Pentaborane-fluorine	.0788	1.810
Diborane-fluorine	.0786	1.860
Methane-fluorine-LOX	.0656	2.230

II. THEORETICAL PERFORMANCE CALCULATIONS

A. Boron Hydrides-Fluorine System

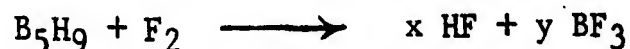
A literature survey on the boron hydrides was made to provide the latest physical and thermodynamic data needed for theoretical performance calculations.³⁻¹² This data is tabulated in Table III.

TABLE III
Physical Properties of the Boron Hydrides

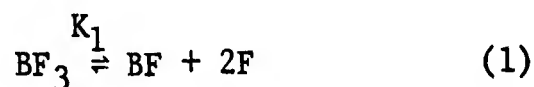
<u>Name</u>	<u>Diborane</u>	<u>Pentaborane</u>	<u>Decaborane</u>
Mol. Wt.	27.69	63.17	122.31
Melt. Pt.	-164.86°C.	-46.6°C.	99.7°C.
Boil. Pt.	-92.43°C.	58.4°C.	ca. 218°C. (interpol.)
Sp. Gr.	0.333 at 243.6°K. to 0.66 at 0°F.	0.61 at 77°F.	0.94 at 20°C.
Heat of Combustion - H _e	481.9 Kcal/mole at 25°C.	1003 Kcal/mole	1950 Kcal/mole
Heat of Formation - H _f	7.53 Kcal/mole-25°C.	15.02 Kcal/mole (g)-25°C. 7.72 " " (1)	27 Kcal/mole (g) 8 Kcal/mole (1)
Free Energy of Formation - F _f	19.78 Kcal/mole-25°C.	39.32 Kcal/mole (g)-25°C. 38.56 " " (1)	71 Kcal/mole (g) 65 Kcal/mole (1)
Entropy - S _o	55.34 cal/deg. mol.-25°C.	65.95 cal/deg. mol. (g)-25°C. 44.03 " " (1)	85.09 cal/deg. mol. (g) 42.20 cal/deg. mol. (1)
Heat Capacity - C _{oP}	13.30 cal/deg. mol.-25°C.	23.52 cal/deg. mol. (g)-25°C. 36.12 " " (1)	40.0 cal/deg. mol. (g) 52.09 cal/deg. mol. (1)

1. Pentaborane-Fluorine System

Reliable theoretical performance data were unavailable from the literature for this system. For the calculation of this data on a frozen basis, the following reaction was used:



The following dissociations take place at chamber temperature



All dissociations above except 5 are considered. The formation of F_2 at chamber temperatures of 4000°K . or higher is negligible as F_2 is completely dissociated at temperatures exceeding 2000°K .

Since so many dissociations take place at the chamber temperature, a partial pressure scheme is used:

1. Calculate the heat of reaction under the initial conditions and set a chamber pressure (P_c).
2. Assume a chamber temperature and tabulate K_1 , K_2 , K_3 and K_4 for that temperature.

3. Assume the partial pressure of BF_3 (P_{BF_3}).
4. Assume the ratio of the partial pressure of HF to H ($\frac{P_{\text{HF}}}{P_{\text{H}}}$).
5. Use the scheme given in Fig. 15 to calculate the partial pressures of BF, B, H, F, HF, and H_2 .
6. If the partial pressures add up to the chamber pressure, P_c , the calculation is correct for the assumed T.
7. From the partial pressure data, calculate the molar amounts of each species present and sum heat content at the assumed temperature including dissociation heat for each equilibrium.
8. When heat content of an assumed T equals the heat of reaction, the assumed T is the chamber temperature (T_c).
9. From T_c and the molar amounts of each species present, the average molecular weight (\bar{m}) and the ratio of the specific heats (γ) may be evaluated. With these values the theoretical performance of the system may be calculated.

Calculations were made on a frozen basis for chamber pressures of 7.50 atm. and 40.83 atm. and for a range of oxidizer to fuel ratios, see Figs. 16, 17 and 18.

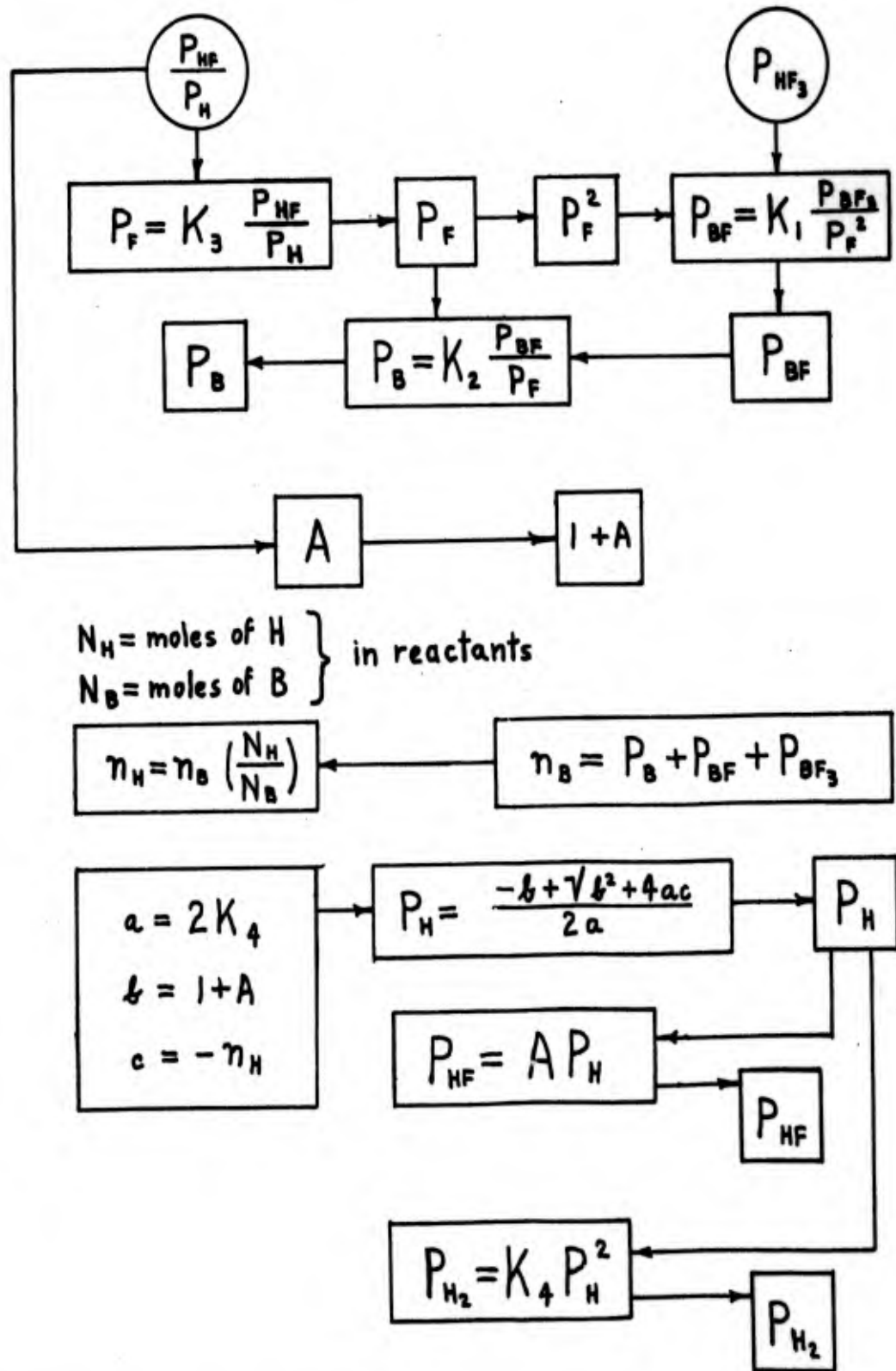


FIGURE 15 — SCHEME FOR CALCULATING EQUILIBRIUM COMPOSITION OF COMBUSTION PRODUCTS FOR BORANE-FLUORINE SYSTEMS

From the calculations for 7.50 atm. a new nozzle insert was designed and fabricated for the micro-rocket combustion unit.

2. Diborane-Fluorine System

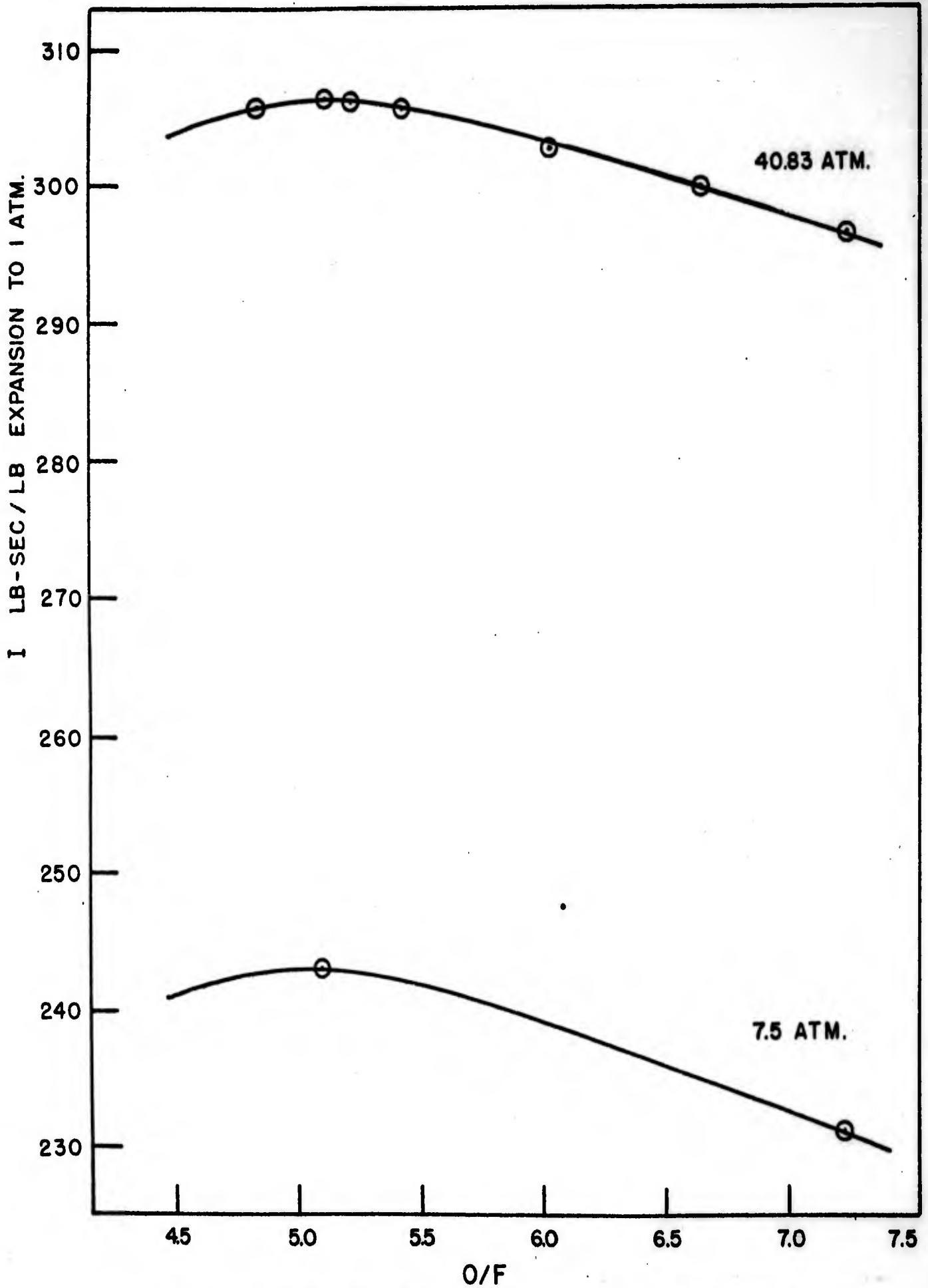
Diborane is supplied in one pound lots in small gas cylinders. For use in the micro-rocket combustion unit it would be liquified by cooling with Freon 12.

Some preliminary theoretical performance data for the diborane-fluorine system were calculated as described for the pentaborane-fluorine system using an oxidizer to fuel ratio of 8.27. These data are tabulated in Table IV.

Calculations showed that the insert nozzle for the pentaborane-fluorine system would also be suitable for the diborane-fluorine system.

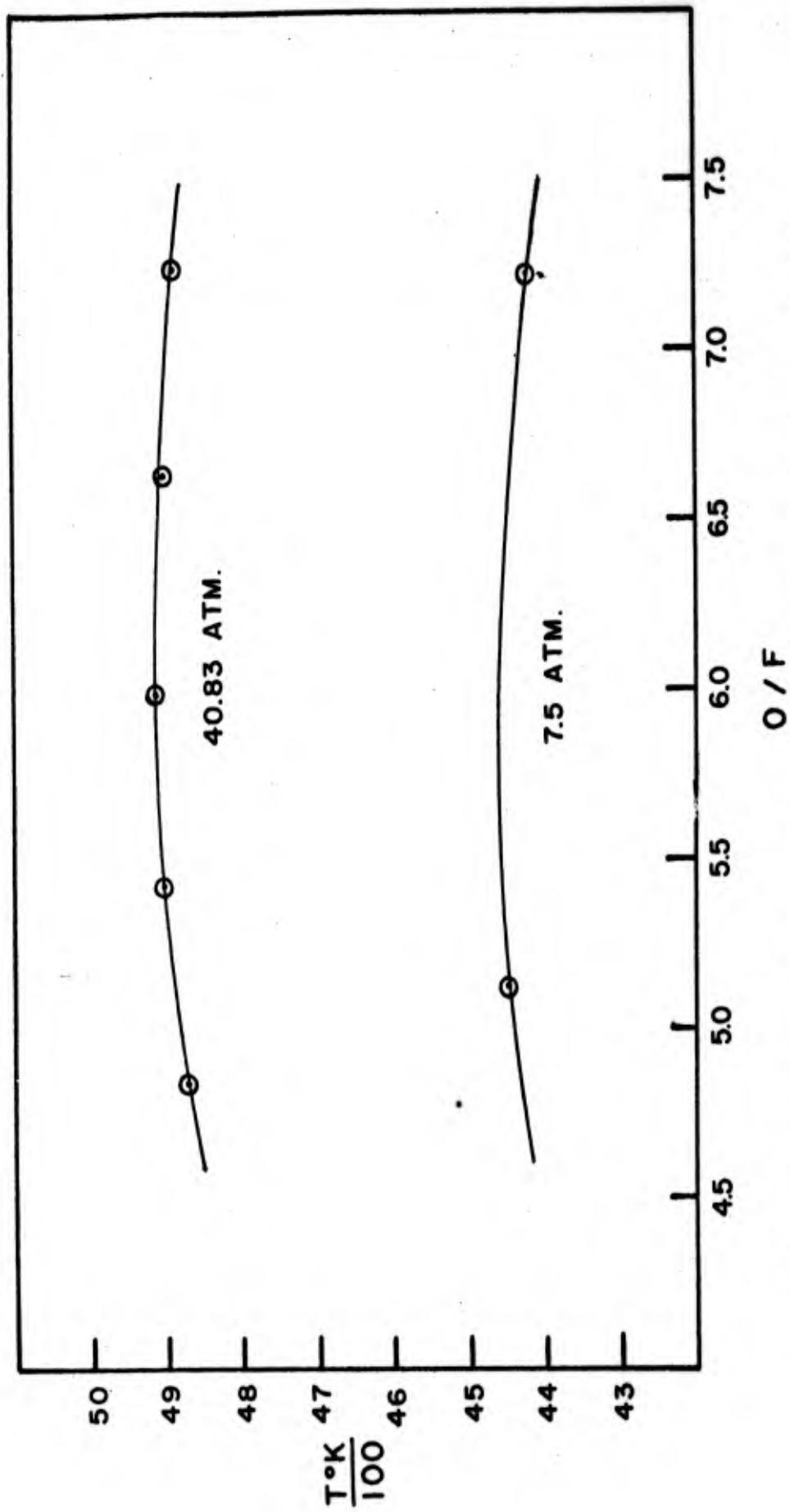
B. Selected High Energy Systems

Those high energy systems which were considered for investigation are listed in Table V along with some of the calculated theoretical performance data.



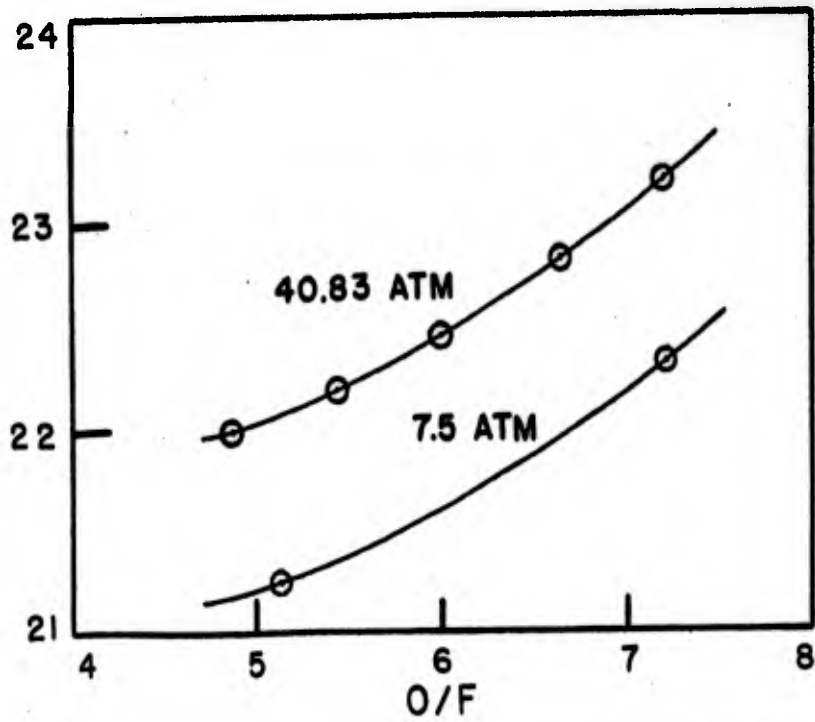
IMPULSE DATA FOR PENTABORANE-FLUORINE REACTION

FIGURE 16

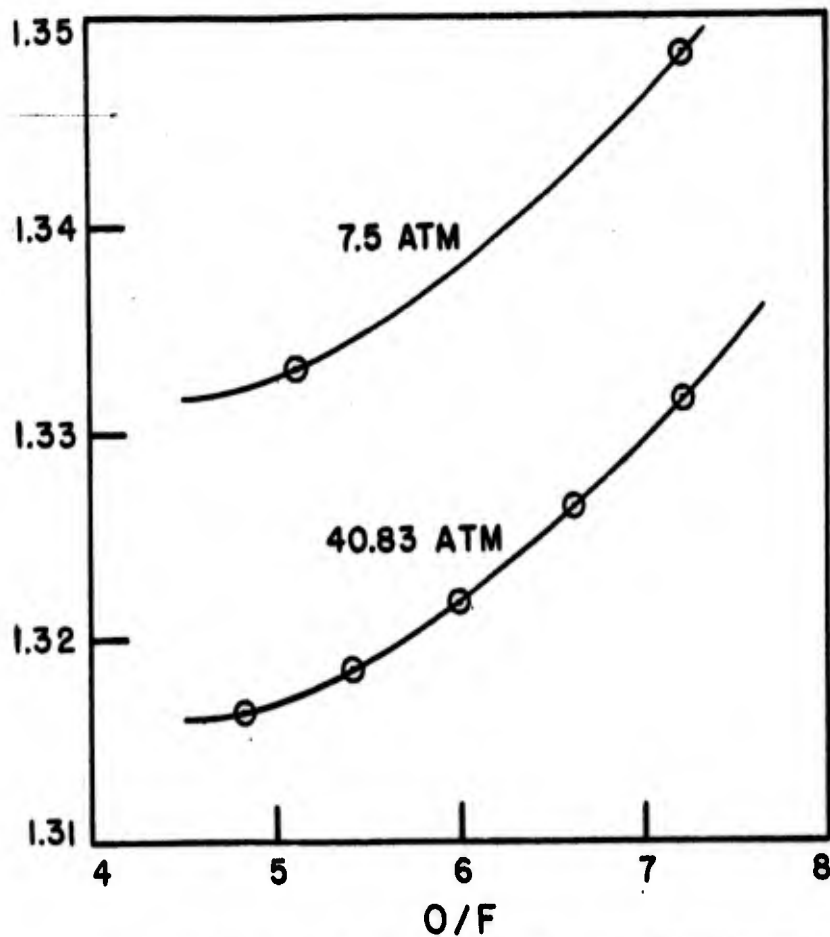


CHAMBER TEMPERATURE DATA FOR PENTABORANE-FLUORINE SYSTEM

FIGURE 17



AVERAGE MOLECULAR WEIGHT OF COMBUSTION PRODUCTS OF THE PENTABORANE-FLUORINE SYSTEM VS O/F.



AVERAGE SPECIFIC HEATS RATIO OF COMBUSTION PRODUCTS OF THE PENTABORANE-FLUORINE SYSTEM VS O/F.

FIGURE 18

The systems which were investigated will be described.

TABLE IV

Diborane-Fluorine System

Chamber Pressure	-	7.5 atmospheres
Oxidizer to Fuel Ratio	-	8.27
Chamber Temperature	-	4457°K.
Specific Heat Ratio	-	1.350
Avg. Molecular Weight of Products	-	20.840
Specific Impulse	-	240.8 sec.

III. PERFORMANCE TESTS

A. JP-4-LOX

As each instrumentation and test stand modification was completed, a number of trial runs using JP-4, LOX were performed to check operation and calibration. In most cases the run was started with ethanol and liquid oxygen and then changed over to JP-4 and liquid oxygen. This method provided a sure starting procedure. All data from these runs are included in Table VI.

TABLE V

Theoretical Performance of Some High Energy Systems for a Chamber Pressure of 300 psia

System	P_c/P_e	$T_c^{\circ}K.$	\bar{m}	ρ g/cm ³	γ	V_e ft./sec.	I sec.	ρI $\frac{lb. sec.}{ft.} \times 10^{-2}$
CH ₄ + 1/2 O ₂ + 2F ₂ (Stoich.)	20	4410	19.34	1.08	1.32	9320	290	196
CH ₄ + 1/2 O ₂ + 1-1/2 F ₂	20	4200	18.21	0.93	1.31	9450	293	170
CH ₄ + 1/3 O ₃ + 2F ₂ (Stoich.)	20	4530	19.20	1.11	1.32	9480	294	203
CH ₄ + 1/3 O ₃ + 1-1/2 F ₂	20	4285	18.00	1.04	1.31	9550	297	193
C ₂ H ₈ N ₂ ^a + 2H ₂ O + 6F ₂ (Stoich.)	20	4256	19.72	1.314	1.32	9060	282	231
C ₂ H ₈ N ₂ + O ₂ + 4F ₂ (Stoich.)	20	4495	19.70	1.284	1.32	9320	289	232
C ₂ H ₈ N ₂ + 2/3 O ₃ + 4F ₂ (Stoich.)	20	4545	19.50	1.314	1.32	9450	293	240
H ₂ + F ₂ (Stoich.)	20	4891	17.55	0.753	1.34	11150	347	163
H ₂ + F ₂ (O/F = 6)	20	3392	10.36	0.383	1.31	11700	363	87
H ₂ + 1/2 O ₂ (Stoich.)	20	3437	15.73	0.430	1.20	9600	298	80
H ₂ + O ₂ (O/F = 3.18)	20	2565	8.39	0.246	1.24	11150	346	53
B ₂ H ₆ + F ₂ (Stoich.) ^b	20	5374	23.53	1.22	--	9450	293	222
B ₂ H ₆ + F ₂ (O/F = 4.98) ^b	20	4900	20.80	1.07	--	9630	299	200
B ₅ H ₉ + F ₂ (O/F = 7.218) ^c	40.8	4947	23.38	1.28	1.16	10600	329.3	262

a. unsym. - dimethyl hydrazine

b. Ref. 4 - data in question.

c. Private communication. Sanford Gordon, NASA, Lewis Research Center, Cleveland, Ohio

P_c = 600 psia

TABLE VI

JP-4 Fuel-Liquid Oxygen System

Run No.	Mass Flow Rate lb./sec. $\times 10^2$	O/F Ratio	Chamber Pressure psia	Thrust lb.	Heat Release Kcal/sec.	Heat Rejection Kcal/sec.	C* ft./sec.	% of Theor. C*	Isp Sec.	Corrected Isp Sec.	Isp % of Theor.
2	4.46	2.68	109.7	7.30	61.1	3.05	4770	86.0	163.5	182	88.5
3	4.28	4.30	76.7	4.90	39.4	1.05	3480	--	114.5	--	--
4	4.29	2.25	108.7	7.05	66.5	3.23	4930	87.2	164	184	89.0
5	4.51	1.64	106.7	7.00	--	--	4590	83.0	155	--	75.7
7a	4.14	0.82	95.7	6.50	--	--	4490	--	157	--	--
10	5.35	1.46	76.7	6.15	108.8	1.64	3830	76.0	115	123.8	66.0
11	4.57	1.98	86.7	7.40	77.5	2.18	5070	92.0	162.0	171.0	85.2
12	5.25	1.37	94.7	8.05	110.0	2.32	4830	98.2	153.5	163.5	86.0
13	5.08	2.07	92.7	8.00	83.6	2.05	4900	89.0	157.0	165.0	81.5
14	5.39	1.55	98.7	9.00	105.5	2.27	4900	94.3	167.0	175.5	88.0
15	5.21	1.60	101.7	9.15	101.0	2.27	5230	98.5	175.5	184.0	92.0
16	4.83	1.59	94.7	8.20	94.0	2.02	5250	99.5	170.0	178.5	90.2
17	3.54	1.43	78.7	6.00	72.5	1.55	5940	120.0	169.5	178.5	96.5
19	4.16	1.65	88.7	8.40	79.0	3.39	5730	106.5	202.0	216.0	110.0

TABLE VI
(Contd.)

JP-4 Fuel-Liquid Oxygen System

Run No.	Mass Flow Rate lb./sec. $\times 10^2$	O/F Ratio	Chamber Pressure psia	Thrust lb.	Heat Release Kcal/sec.	Heat Rejection Kcal/sec.	C* ft./sec.	% of Theor. C*	Isp Sec.	Corrected Isp Sec.	Isp % of Theor.
20	4.50	1.87	84.7	8.15	78.9	2.98	5050	91.8	181.0	193.0	97.5
21 ^b	4.71	2.04	84.7	7.90	78.3	2.41	4820	87.5	167.5	177.0	88.5
22 ^c	4.20	1.68	77.7	7.35	78.5	2.22	4950	91.7	175.0	185.0	96.5
24	4.40	2.49	78.7	6.70	63.4	2.57	4780	88.0	152.0	163.5	85.8
25	5.50	1.38	100.7	9.30	--	--	4900	100.0	169.0	--	90.0
26	5.25	1.61	99.7	9.40	101.5	2.34	5080	96.0	182.0	190.0	95.0
27	5.25	1.34	99.7	9.15	112.0	2.24	5080	102.0	174.0	183.0	94.5
28	5.05	1.30	98.7	8.50	109.0	2.16	5240	106.5	168.5	177.0	92.0
29	4.55	2.27	89.7	7.75	--	--	5280	96.4	170.0	--	80.8
30	3.79	2.63	80.7	7.00	--	--	5560	100.0	184.5	--	89.3
31	5.31	1.47	89.7	7.40	--	--	4520	89.4	139.5	--	71.8
32	5.53	1.51	106.7	9.70	--	--	5160	97.7	175.5	--	89.4
33 ^c	--	--	--	--	--	--	--	--	--	--	--

TABLE VI
(Contd.)

JP-4 Fuel-Liquid Oxygen System

Run No.	Mass Flow Rate lb./sec. $\times 10^2$	O/F Ratio	Chamber Pressure psia	Thrust lb.	Heat Release Kcal/sec.	Heat Rejection Kcal/sec.	C* ft./sec.	% of Theor. C*	I _{sp} Sec.	Corrected I _{sp} Sec.	I _{sp} % of Theor.
34	3.93	1.88	93.0	7.75	--	--	6330	111.0	197.5	--	93.6
35a	5.38	2.89	107.5	9.70	70.0	3.56	5340	97.5	180.0	191.0	94.0
36 ^a	5.05	2.38	103.7	9.15	75.3	3.25	5500	97.8	181.5	192.0	93.3

Notes: a - Hypergolic Ignition - 0.05% O₃F₂ in LOX

b - Hypergolic Ignition - 0.05% O₃F₂ and 1.0% O₃ in LOX

c - Hypergolic Ignition - 0.10% O₃F₂ in LOX

From the temperatures of the cooling water, it was found that 2-5% of the combustion heat was being rejected to the cooling water in the tests of the JP-4-LOX system. In order to correct the specific impulse, which was calculated on an adiabatic basis, the following equation was used:¹³

$$I_{sp(c)} = I_{exp.}^2 + \frac{2J}{g} Q_n$$

$$\text{where } n = 1 - \frac{T_e}{T_c}$$

$I_{sp(c)}$ = Specific impulse corrected for heat rejection.

$I_{exp.}$ = Specific impulse from experimental data.

T_e = Exit temperature (°K.).

T_c = Chamber temperature (°K.).

J = Mechanical equivalent of heat (778 ft.lb./BTU).

g = Acceleration due to gravity (32.17 ft./sec.²).

Q = Heat Rejection (BTU/lb. of propellant).

From runs in which the cooling water temperatures were taken, the corrected specific impulse was used to calculate the percent of theoretical specific impulse. The theoretical values of I_{sp} and C^* (characteristic velocity) were interpolated and extrapolated from a research memorandum by V. M. Huff and A. Fortini.¹⁴ The results of some of the runs will not be compared to the theoretical values since the oxidizer to fuel ratios were out of the range of interest.

B. RP-1-LOX

In the later calibration runs RP-1 hydrocarbon fuel was substituted for JP-4 as the fuel with liquid oxygen. RP-1 is slightly more difficult to ignite than JP-4 and has a higher density, but the performance data at 110 psia chamber pressure should be very nearly equivalent.

The petroleum fraction fuels were selected along with liquid oxygen as the oxidizer to be the basis of the calibration standard for propellant evaluation system of this contract. Ease of handling, excellent availability, and a wealth of existing theoretical performance data were the deciding factors in this selection.

The goal in the development of the propellant evaluation system was to perfect a micro-combustion unit which would be versatile enough to handle any liquid propellant in small quantities (50-200 ml.) and would yield performance data close to theoretical values. This goal has been attained in the case of the RP-1-LOX system. After eliminating the last of the major problems (very low gaseous flow measurement and recording) the propellant evaluator will now yield performance data on the RP-1-LOX system in the neighborhood of 85-95% of theoretical, Table VII.

TABLE VII

RP-1 Fuel-Liquid Oxygen System

Run No.	Mass Flow Rate lb./sec. $\times 10^2$	O/F Ratio	Chamber Pressure psia	Thrust lb.	Heat Release Kcal/sec.	Heat Rejection Kcal/sec.	C* ft./sec.	% of Theor. C*	Isp Sec.	Corrected Isp Sec.	Isp % of Theor.
46	5.43	2.46	97.7	8.70	174.2	4.32	4820	86.0	160.0	174.5	83.6
49	5.44	2.36	112.7	10.60	--	--	5720	101.5	195.5	--	93.5
52	5.54	2.04	102.7	9.20	--	--	5100	90.0	166.0	--	88.0
55 ^a	5.64	4.03	106.7	9.40	--	--	4790	92.2	166.5	--	87.8
56 ^a	6.66	2.99	106.7	9.80	--	--	4050	74.0	147.0	--	73.4
57 ^a	6.06	2.32	98.7	9.35	--	--	4130	73.5	154.1	--	75.0
58 ^a	5.91	4.95	90.7	8.75	--	--	3880	80.0	148.0	--	--
59 ^a	6.80	0.78	89.7	9.35	--	--	3340	76.0	137.5	--	76.5
60 ^a	4.55	2.07	88.7	9.20	--	--	4930	87.2	202	--	100.0
62	4.27	1.16	79.7	7.30	98.8	4.17	5150	112.0	171.2	184.4	106.5
63	3.54	2.07	67.7	6.33	58.0	3.58	5280	94.0	178.8	188.9	100.0
68	4.45	2.06	88.7	8.07	--	--	5500	97.2	181.5	--	90.2
69	4.62	2.47	92.7	8.60	--	--	5530	98.7	186.2	--	93.2

a. Calibration Test - Used with pentaborane nozzle.

C. Hydrogen-Oxygen

Three test runs, Table VIII, were made with the gaseous hydrogen-liquid oxygen system as a preliminary investigation and solely to provide injector calibration data, and handling characteristics of the system.

The data obtained were applied to the operation of this system with the addition of ozone fluoride to the liquid oxygen as a hypergolic ignition agent.

These later investigations were specifically requested by the contracting agency and are described in this report under Ozone Fluoride Investigations.

The gaseous hydrogen in these tests was cooled in 50 feet of coiled copper tubing immersed in liquid nitrogen. Under maximum flow conditions (0.003 lb./sec.) this cooling system was able to hold the exit hydrogen gas temperature between 85 and 90°K.

A water cooled injector, Type 4-3, was used for these combustion studies. This injector was not designed specifically for the hydrogen-oxygen system and cannot be considered an efficient injection device for a gaseous-liquid system. However, since compilation of accurate performance data was not the object of this investigation, inefficient combustion was of no consequence.

The tests provided the necessary information and displayed the typical hydrogen-oxygen flame with a low level of luminescence.

TABLE VIIIGaseous Hydrogen-Liquid Oxygen System

<u>Run No.</u>	<u>Mass Flow Rate lb./sec. x10²</u>	<u>O/F Ratio</u>	<u>Chamber Pressure psia</u>	<u>Thrust lb.</u>	<u>C* ft./sec.</u>	<u>I_{sp} Sec.</u>
38	O f f S c a l e		--	5.20	--	--
39	O f f S c a l e		--	5.30	--	--
40	3.19	7.98	71.0	5.30	5970	166.0
41*	3.84	11.50	74.0	5.55	5160	144.8
42*	3.82	10.30	76.7	5.95	5380	156.0

*Hypergolic ignition through use of 0.05% solution O₃F₂ in LOX.

D. n-Hexane-Oxygen

A number of test runs were made using n-hexane with liquid oxygen. These tests were conducted for the sole purpose of providing injector calibration data and handling characteristics of n-hexane before it was used as a starting fuel for the penta-borane-fluorine system, and as a solvent for the decaborane-fluorine system.

The highly volatile n-hexane was easily ignited and displayed exceptionally smooth combustion characteristics. Data recorded from these runs are listed in Table IX.

TABLE IXn-Hexane-Liquid Oxygen System

<u>Run No.</u>	<u>Mass Flow Rate lb./sec. x10²</u>	<u>O/F Ratio</u>	<u>Chamber Pressure psia</u>	<u>Thrust lb.</u>	<u>C* ft./sec.</u>	<u>I_{sp} Sec.</u>
64	5.55	5.81	100.7	9.43	5000	170.0
65	5.29	3.71	92.7	8.85	4840	167.5
66	5.64	2.55	88.7	8.50	4340	150.5
70	4.19	2.48	88.7	8.70	5840	207.5
71	3.95	2.83	90.7	8.35	6330	211.0
73	4.87	3.99	100.7	8.65	5710	177.5

E. Pentaborane-Fluorine

The pentaborane-fluorine system was investigated because of its high energy characteristics. At 110 psia, the operating chamber pressure of the propellant evaluator, this system at its optimum oxidizer/fuel ratio of 5.0 was calculated to have a specific impulse of 243 sec. and a density impulse of 21,200 lb.sec./ft.³

The RP-1-LOX system at its optimum oxidizer/fuel ratio of 2.0 gives a specific impulse of 212 sec. and a density impulse of 13,700 lb. sec./ft.³ At 600 psia the pentaborane-fluorine system gives a specific impulse of 305 sec. compared to 272 sec. for the RP-1-LOX system. Handling problems which would arise with

the use of this toxic and hypergolic system are obvious, but only the thermal decomposition characteristics of pentaborane provided any trouble in these investigations.

The first test of this system (Run No. 03-47, Table X) was made with a fuel cooled stainless steel injector (Type 6-1).

An instantaneous hypergolic ignition occurred with a very long brilliant greenish-white flame and clouds of dense white smoke. Approximately four seconds after the start of the run, a leak developed in the fluorine valve stem. The lowered pressure in the fluorine line caused a flashback and the fluorine line ruptured. The very fuel rich combustion and decomposition of pentaborane which then resulted quickly plugged up the injector holes with the thick polymerized products of decomposition of pentaborane.

The system was shut down and the remaining pentaborane was removed by evacuation of the system and condensation of the pentaborane vapors in a liquid nitrogen cooled trap.

The second test of the system (Run No. 03-61) was made with the Type 6-4 stainless steel injector. The run was started with the RP-1-LOX system and changed over to pentaborane and fluorine. Again the characteristic long greenish-white flame resulted, Fig. 19. The run was approximately 23 seconds long but

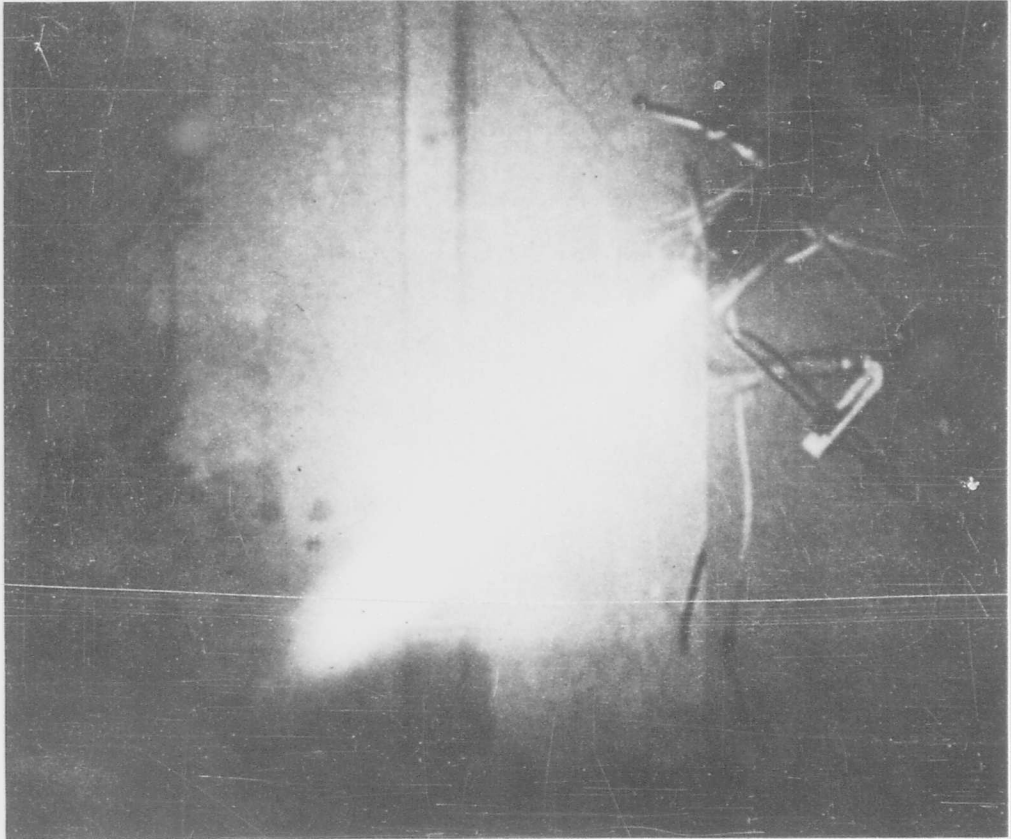


FIG. 19 - PENTABORANE - FLUORINE SYSTEM - RUN #03-61

was interrupted once when the fuel holes became clogged from the pre-combustion decomposition of pentaborane. It freed itself and the run continued. The oxidizer to fuel ratio of 4.78 was close to the desired 5.0, but the chamber pressure of 64.7 psia was low for the desired 110 psia. The calculated specific impulse was 184.5 sec. The specific impulse was considerably reduced by a high heat rejection to the cooling water. The heat rejection was approximately 6.9 Kcal/sec. which is about 4 Kcal greater than any value measured with the RP-1-LOX system. This is to be expected since the theoretical chamber temperature is at least 1000°K . higher in the pentaborane-fluorine system.

Results were also altered by a fluorine leakage around the low temperature valve stem. This leakage would give a higher actual specific impulse than the calculated value in Table X, but from extrapolated impulse-chamber pressure data the experimental value is approximately 83% of theoretical.

The third test run (Run No. 03-72) was similar to the second test, but the data should be invalidated due to a cooling water leakage into the chamber. It should be noted however, that the combustion was stable and no clogging occurred in the injector ports by use of a Type 7-1 water cooled injector.

TABLE XPentaborane-Liquid Fluorine System

<u>Run No.</u>	<u>Mass Flow Rate lb./sec. x10²</u>	<u>O/F Ratio</u>	<u>Chamber Pressure psia</u>	<u>Thrust lb.</u>	<u>Heat Rejection Kcal/sec.</u>	<u>C* ft./sec.</u>	<u>I_{sp} Sec.</u>
47	F l u o r i n e	L e a k	5.50	--	--	--	--
61	3.88	4.78	64.7	7.15	6.92	4227	184.5
72	4.75	2.95	92.7	7.87	--	4980	166.5

Another injector (Type 8-1) was designed and fabricated for a future test of pentaborane and fluorine. This latest injector was designed with a solid face to eliminate leakage and was machined from brass for high heat transfer to eliminate pre-combustion decomposition of pentaborane. Contract time did not allow the testing of this injector with its intended system but it was tested with the RP-1-LOX system and showed excellent heat transfer characteristics. These tests indicate that good results should be obtained with pentaborane and fluorine and there is no reason why results should not be obtained on the order of 90% of theoretical as in the RP-1-LOX system.

IV. OZONE FLUORIDE INVESTIGATIONS

An investigation into the use of O_3F_2 as an additive to liquid oxygen for hypergolic ignition properties was initiated.

A separate report was issued on this subject.¹⁵

A. Properties of O_3F_2 ^{2,15,16,17}

Ozone fluoride is a blood-red viscous liquid solidifying at 83°K. and decomposing quantitatively with heat evolution at about 115°K. according to the equation:



At about 200°K. O_2F_2 dissociates with heat evolution to $O_2 + F_2$.

O_3F_2 is a strong oxidizer, and, although extremely reactive, it may be evaporated rapidly, refluxed or thermally decomposed without exploding. However, in contact with any organic or other oxidizable material it will burn or detonate immediately.

B. Preparation of O_3F_2 ^{2,15,17}

O_3F_2 was prepared by passing a high voltage electrical discharge through a reaction vessel containing a $3O_2:2F_2$ mixture at 77°K., and about 12 mm. total pressure, Fig. 20. The electrical discharge varied between 2100-2400 volts and 20 - 30 milliamperes.

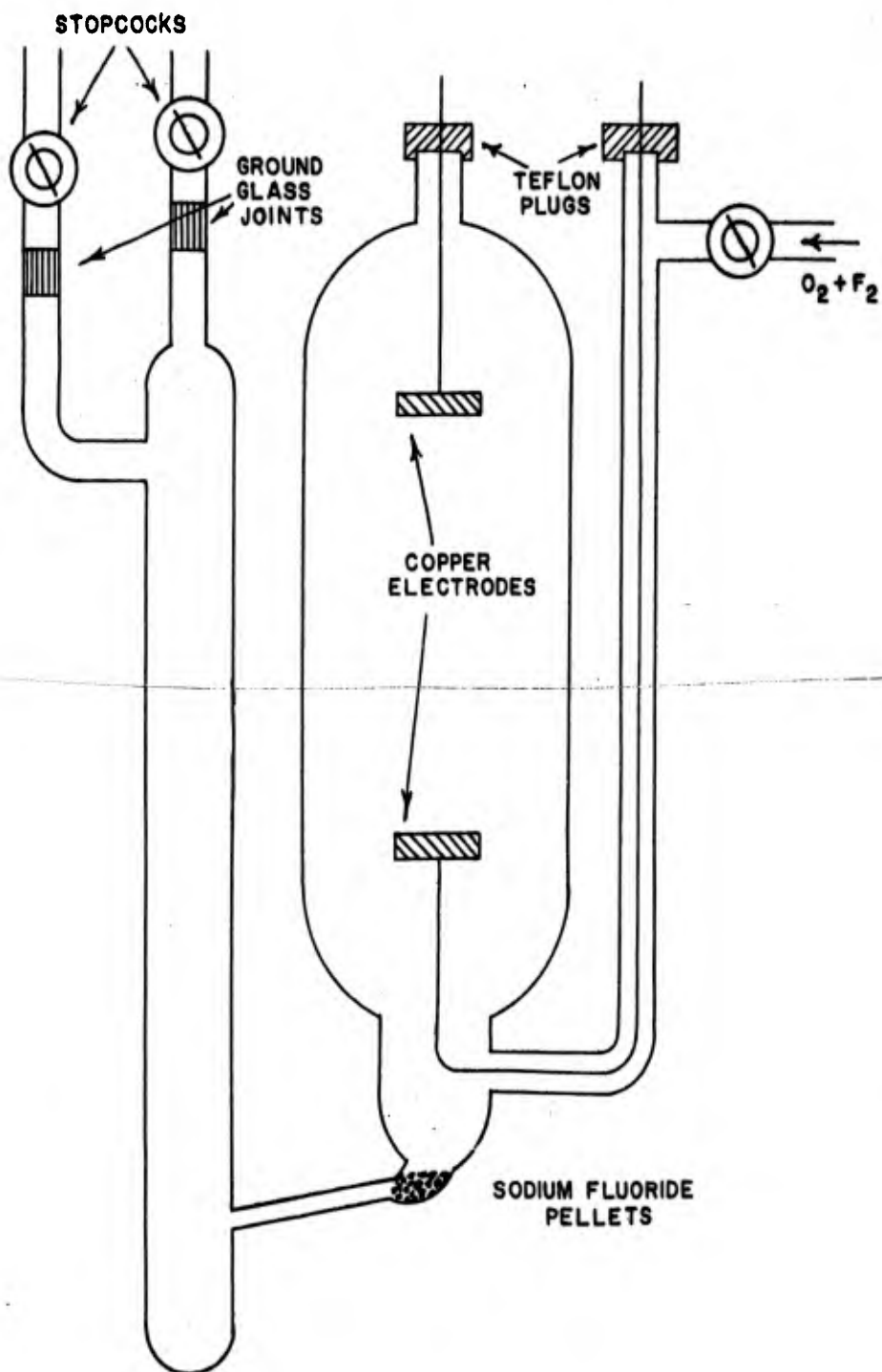


FIG. 20 - REACTION VESSEL FOR PREPARATION
OF O_3F_2

All stopcocks on the reaction vessel were ground glass with Kel-F grease as a lubricant. The fluorine-oxygen mixture combined quantitatively to form O_3F_2 at the rate of about 10 cm^3 per 8 hr. day, while the pressure remained constant. The use of two parallel reaction vessels doubled this daily yield.

Before use the gaseous fluorine-oxygen mixture was passed through a trap at 77°K . to remove any hydrogen fluoride impurities in the fluorine. Sodium fluoride pellets were placed in the reaction vessel to remove any SiF_4 formed.

C. Handling Procedure of O_3F_2 ²

Each day's production of O_3F_2 was removed from the reaction vessels and temporarily stored in a glass storage vessel.

The solid O_3F_2 which formed on the walls of the reaction vessel was allowed to liquefy by immersing the reaction vessel in a Dewar flask containing liquid oxygen.

The O_3F_2 -LOX mixture was made by vigorously stirring 100 cc. of liquid oxygen with an excess (5 cc.) of O_3F_2 in a small glass test tube until saturation occurred. Then the O_3F_2 -LOX mixture was transferred to a 500 cc. storage flask. This procedure was repeated until about 400 cc. of the mixture was obtained. Transfer of the mixture to the micro-rocket oxidizer tank was effected by the use of a jacketed glass siphon cooled by liquid oxygen. The transfer was accomplished under a helium atmosphere and helium pressure was used to force the O_3F_2 into the receiving vessel.

D. Solubility of O_3F_2 in LOX

Solubility measurements of O_3F_2 in LOX were made at two temperatures, 90°K. (b.p. of O_2) and 77°K. (b.p. of N_2).² The results are listed in Table XI and are shown in Fig. 21

TABLE XI
 O_3F_2 Solubility in LOX

<u>T°K.</u>	<u>Solubility ppm.</u>	<u>Saturation Wt. %</u>
90	1100	0.110
77	460	0.046

Experiments have shown that if a saturated solution of O_3F_2 in LOX at 90°K. is cooled to 77°K., O_3F_2 will precipitate from solution. However, a 0.05% solution of O_3F_2 in LOX would allow a 50% boil-off of LOX without any precipitation of O_3F_2 . Data given later in this report show that this mixture (0.05% O_3F_2 in LOX) is hypergolic with every fuel tested thus far.

E. Initial Studies Using 0.05% O_3F_2 in LOX - Open Cup Tests

A series of open cup tests were performed to establish ignition time of various fuels with 0.05% O_3F_2 in LOX. Five cc's of the fuel to be tested were poured into an aluminum cup and a few

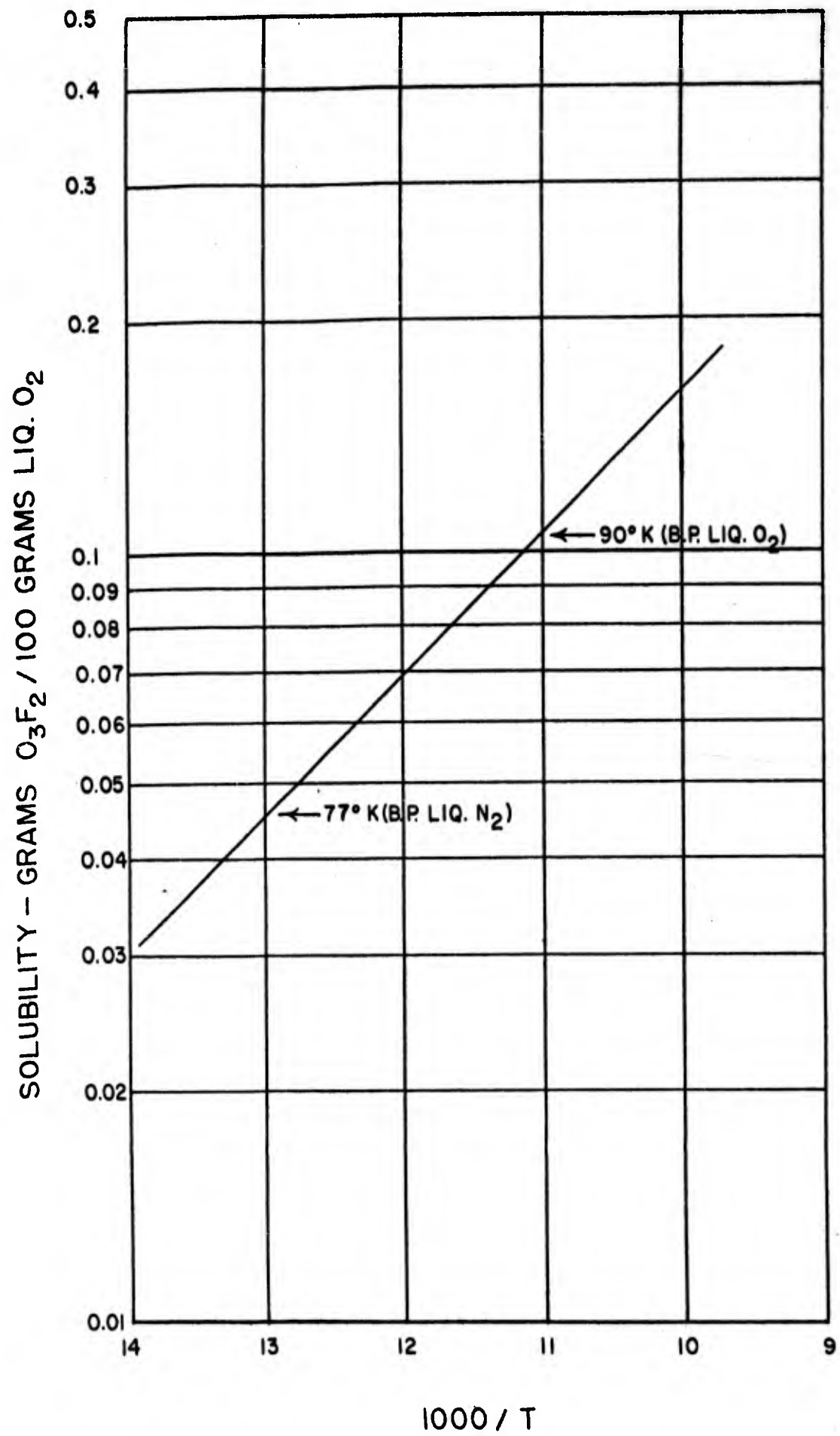


FIG. 21

ml. of 0.05% O_3F_2 in LOX added. The time required for ignition to take place was noted. Results are given in Table XII.

TABLE XII

O_3F_2 Open Cup Ignition Tests

<u>Fuel</u>	<u>Ml.</u>	<u>Oxidizer</u>	<u>Ml.</u>	<u>Reaction Time - Sec.</u>
JP-4	5	0.05 wt. % O_3F_2 in LOX	5	3.4 (ignition)
JP-4	5	" "	10	12.4 (detonation)
UDMH	5	" "	5	Instantaneous detonation
U-DETA	5	" "	1	Instantaneous ignition
50 vol. % UDMH 50 vol. % JP-4	5	" "	3	" "
10 vol. % UDMH 90 vol. % JP-4	5	" "	3	" "
5 vol. % UDMH 95 vol. % JP-4	5	" "	5	" "
1 vol. % UDMH 99 vol. % JP-4	5	" "	5	" "

F. Micro-Rocket Tests Using 0.1 Wt. % O_3F_2 in LOX

O_3F_2 in LOX and various fuels were tested in a 7.5 atm. micro-rocket motor. Results are summarized in Table XIII. Regardless of the concentration of O_3F_2 in LOX used, injector design plays an important role in the ignition results. The

TABLE XIIIExperiments Using 0.1 Wt. % O_3F_2 in LOX

<u>Fuel</u>	<u>Starting Pressure</u>		<u>Injector*</u>	<u>Remarks</u>
	<u>Fuel</u>	<u>Oxidizer</u>		
Ethyl alcohol (100%)	10	70	3 Fuel on 1 Oxidizer	Immediate ignition
Propane (at -78°C.)	10	70	" "	Ignition outside chamber
JP-4	4	20	3 Fuel on 3 Oxidizer	Immediate ignition
U-DETA	4	20	" "	" "

* All injectors were fuel cooled

injector must allow maximum evaporation of the O_2 from the O_3F_2 - O_2 mixtures, with resulting concentration of O_3F_2 , and also intimate mixing of the fuel with the O_3F_2 - O_2 mixture.

After several failures to obtain hypergolic ignition using 0.1% O_3F_2 in LOX with JP-4, the injector design was varied. An injector with 3 fuel on 3 oxidizer, 1 on 1 impingement (Type 6-1) proved most successful.

G. Micro-Rocket Test Using 0.05 Wt. % O_3F_2 in LOX

The problem of O_3F_2 precipitation from LOX solution exists in the use of 0.1% O_3F_2 in LOX, since any boil-off of O_2 would cause some O_3F_2 to come out of solution. The use of 0.05% O_3F_2 in LOX

allows a 50% boil-off of O_2 without any precipitation of O_3F_2 . Experiments were carried out in the micro-rocket using 0.05% O_3F_2 in LOX with ethyl alcohol, JP-4, U-DETA, and hydrogen gas. The results are given in Table XIV.

TABLE XIV

0.05% O_3F_2 in LOX

<u>Fuel</u>	<u>Starting Pressure</u>		<u>Injector*</u>	<u>Remarks</u>
	<u>Fuel</u>	<u>Oxidizer</u>		
Ethyl alcohol	4	20	3 Fuel on 1 Oxidizer	Instantaneous ignition
JP-4	4	20	" "	Instantaneous ignition in each of 3 runs
U-DETA	4	20	" "	" "
H_2 (gas at 90°K.)	75	20	1 Fuel on 1 Oxidizer water- cooled	Instantaneous ignition in both of 2 runs

*Unless otherwise noted all injectors were fuel cooled

In the hydrogen runs the gaseous hydrogen was cooled to approximately 90°K. as described earlier in the report. Since the injector used was not specifically designed for efficient combustion, the instantaneous ignition in both runs would indicate that the hydrogen-oxygen system is an especially good application for the hypergolic ignition properties of ozone fluoride.

H. O₃F₂ Solution Compatibility Tests

Compatibility tests of O₃F₂-liquid oxygen solutions with various organic and inorganic materials were requested by the Structures and Mechanics Division of the George C. Marshall Space Flight Center, Huntsville, Alabama.

Two methods of testing compatibility were used. The first method was a very simple test but was also a very severe exposure. The material to be tested was placed in a shallow aluminum cup. Using remote control apparatus behind a barrier, 10 ml. of O₃F₂ solution in liquid oxygen was then poured over the test sample. Two different concentrations were used; the saturated solution at the boiling point of liquid oxygen (0.10%) and the saturated solution at the boiling point of liquid nitrogen (0.05%). The solution was allowed to vaporize completely and if any reaction occurred, the relative reaction times were noted. Materials tested in this manner were thus exposed not only to direct contact with the cryogenic O₃F₂ solution, but also with all the products of the stepwise decomposition of O₃F₂, which include O₂F₂ and eventually fluorine. The results of these tests are reported in Table XV and Table XVI.

The second method of compatibility testing allowed contact of the test materials with the cryogenic solution only.

TABLE XVVaporization Compatibility Tests - 0.05% O₃F₂ Solution

<u>Sample No.</u>	<u>Material</u>	<u>Size</u>	<u>Results</u>
1	Ethanol	5 ml.	Instant ignition
2	Polyethylene	1" x 1"	Delayed ignition - 60 sec.
3	Lox Lube AR-1F	2 ml.	Delayed slight reaction
4	Halocarbon Oil- Series 11-14 Halocarbon Prod. Co.	2 ml.	No reaction
5	Kel-F Oil	2 ml.	" "
6	Kel-F Alkane 464, M. W. Kellogg Co.	2 ml.	" "
7	JM-76 Gasket Matl.	1" x 1" x 1/8"	" "
8	Teflon	1/2" thick ring	" "
9	Kel-F	1/2" thick ring	" "
10	Fluorolube CP-93- 49, Hooker Chem. Co.	1 ml.	" "
11	Molycote-Z	3 grams	" "

TABLE XVIVaporization Compatibility Tests - 0.10% O₃F₂ Solution

<u>Sample No.</u>	<u>Material</u>	<u>Size</u>	<u>Results</u>
1	Ethanol	5 ml.	Instant ignition
2	Polyethylene	1" x 1"	Delayed ignition
3*	Lox Lube AR-1F	2 ml.	Delayed slight reaction
4	Halocarbon Oil- Series 11-14 Halocarbon Prod. Co.	2 ml.	" " "
5	Kel-F Oil, 3M Co.	2 ml.	No Reaction
6	Kel-F Alkane 464 M. W. Kellogg Co.	2 ml.	" "
7*	JM-76 Gasket Matl.	1"x1"x1/8"	" "
8	Teflon	1/2" thick ring	" "
9	Kel-F	1/2" thick ring	" "
10	Fluorolube, CP-93- 49, Hooker Chem. Co.	1 ml.	" "
11*	Molycote-Z	3 grams	" "
12*	Stainless Steel- Carpenter 20Cb	Filings (5 gr.)	" "
13*	Magnesium Lithium (14.1%) Alloy	" "	" "
14*	Magnesium Thorium Alloy	" "	" "
15*	Titanium Alloy	" "	" "

TABLE XVI
(Contd.)

Vaporization Compatibility Tests - 0.10% O₃F₂ Solution

<u>Sample No.</u>	<u>Material</u>	<u>Size</u>	<u>Results</u>
16*	JM "Lo-Flo" Gasket Matl.	1/16"x1/2" strip	No Reaction
17*	Duroid 3400 Gasket Matl.	" " "	" "
18*	Oxylube 702	Liquid (2 ml.)	" "

*Submitted for test by:
Structures & Mechanics Division
George C. Marshall Space Flight Center
Huntsville, Alabama

This method of testing would more nearly simulate the conditions of large scale commercial handling.

A saturated solution (0.10%) of O₃F₂ in liquid oxygen at liquid nitrogen temperature (77.2°K.) was contained in a vessel surrounded by a liquid nitrogen coolant. The sample to be tested was cooled by immersion in liquid nitrogen and was then dropped into the O₃F₂ solution. The materials tested are listed in Table XVII. No reaction was caused by any one of the samples tested.

TABLE XVIIImmersion Compatibility Tests - 0.10% O₃F₂ Solution

<u>Sample No.</u>	<u>Material</u>	<u>Type of Sample</u>	<u>Results</u>
1*	Stainless Steel - Carpenter 20Cb	Filings (5 grams)	No Reaction
2*	Magnesium-Lithium (14.1%) Alloy	" "	" "
3*	Magnesium-Thorium Alloy - XK31	" "	" "
4*	Titanium-Alloy - Beta-120-VCA	" "	" "
5*	Johns-Manville "Lo Flo" Gasket Material	1/16"x1/2" strip	" "
6*	Duroid 3400 Gasket Material	" " "	" "
7*	Oxylube 702	Liquid (2 ml.)	" "
8*	Molycote "Z"	Powder (3 grams)	" "
9	Polyethylene	Film 1" x 1"	" "

*Submitted for test by:
Structures & Mechanics Division
George C. Marshall Space Flight Center
Huntsville, Alabama

CONCLUSIONS AND RECOMMENDATIONS

The test results of the propellant evaluator, particularly those for the standard RP-1-LOX system which were so close to theoretical (90%), prove the feasibility of this apparatus as a research tool in the development of high energy propellants. The small quantities of propellants (50-200 ml.) required to provide accurate experimental data is an outstanding feature which limits the hazards encountered in the handling of high energy propellants. The Institute recommends the further use of this evaluator to provide actual firing data on all of the new propellants constantly being synthesized.

The results of the tests of dilute solutions of O_3F_2 in liquid oxygen as a hypergolic oxidizer, show this material to be hypergolic with all of the fuels tested, and at the same time to be non-reactive on simple contact with most materials of construction commonly used in LOX systems. The most significant practical application of this development is immediately recognized to be the use of small amounts (0.05%) of O_3F_2 to render the important hydrogen-oxygen system hypergolic. A stop-start space vehicle relying completely on O_3F_2 based ignition may not presently be realistic, however, the addition of O_3F_2 to the oxygen would at least be an added safety factor to the standard spark ignition.

The Institute advises the further, larger scale investigations of O_3F_2 for use in hypergolic ignition.

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