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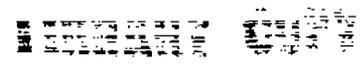
RESEARCH MEMORANDUM

LOW-TEMPERATURE CHEMICAL STARTING OF A 200-POUND-
THRUST JP-4 - NITRIC ACID ROCKET ENGINE USING A
THREE-FLUID PROPELLANT VALVE

By Glen Hennings and Gerald Morrell

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RESEARCH MEMORANDUMLOW-TEMPERATURE CHEMICAL STARTING OF A 200-POUND-THRUST JP-4 - NITRIC
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SUMMARY

Studies in a 200-pound-thrust rocket engine showed that starting of JP-4 - nitric acid propellants could be accomplished at temperatures as low as -65° to -60° F with hydrazine-water igniter fuels. Limiting igniter-fuel compositions for starting at -60° F were 55 and 69 percent hydrazine. An essential feature of the propellant system was a three-fluid flow control valve, which also scheduled the transition from igniter fuel to JP-4.

The same technique was used with an organophosphorus igniter fuel (RF 208) and with a blend of 70 percent triethylamine and 30 percent o-toluidine; starts were obtained at -80° F.

The tendency toward unstable combustion of JP-4 - nitric acid increased as temperature decreased.

INTRODUCTION

The jet fuel - nitric acid propellant system has been proposed or specified for a variety of tactical liquid rocket applications. Typical military specifications now require reliable ignition over a temperature range of -40° to 120° F (ref. 1). Future requirements are expected to lower the limit to -65° F or less; red fuming nitric acid containing 12 to 20 percent nitrogen dioxide will probably be used as an oxidant because of its low melting point (about -85° F) and good ignition characteristics (ref. 2). References 3 and 4, for example, show that the pressures developed in storage of such acids is low, and that a solution of the corrosion problem is in sight.

JP-4 and nitric acid do not ignite spontaneously at normal temperatures; one method for supplying ignition energy is to inject ahead of the hydrocarbon fuel a material that ignites on contact with the acid. Previous research (refs. 1 and 5) has shown that hydrazine-water mixtures

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(approximately 69 percent hydrazine) are suitable igniter fuels at -40° F and higher. In these studies, flow sequence and igniter composition appeared to be quite critical at the lower temperature. No systematic studies of permissible water content in hydrazine-water blends have been published. For field service especially, there are advantages to having as wide composition limits as possible.

Certain fuels such as dioxaphospholanes, alkylthiophosphites, and aromatic amine - aliphatic amine blends have short ignition lags with nitric acid at temperatures as low as -95° F (refs. 6 to 8); these should be suitable for igniting the jet fuel - acid system at temperatures well below -40° F.

This study was conducted to measure the temperature-composition limits of hydrazine-water blends as igniter fuels for the JP-4 - red fuming nitric acid system. A 200-pound-thrust rocket engine equipped with a three-fluid valve was used; the entire propellant system was immersed in a cold bath. The proportion of fuel and oxidant at any valve position was kept constant. Only the valve opening rate was varied in addition to temperature and igniter-fuel composition.

Another object of the study was to evaluate two representative extremely low-temperature igniter fuels using the same technique, tank, and rocket engine. These fuels were 4-methyl-2-dimethylamino-1,3,2-dioxaphospholane and a blend of 70 percent triethylamine and 30 percent o-toluidine. As a corollary of this objective, it was desired to learn whether the JP-4 - acid system could be ignited near -80° F by chemical means.

APPARATUS AND PROCEDURE

Propellant System and Engines

A diagram of the pressure-feed propellant system used for this study is shown in figure 1. The distinctive features of this system, as compared with that used in a previous study of engine starting (ref. 5), are the separate igniter fuel tank and the three-fluid propellant control valve. This valve, developed for the Navy Bureau of Aeronautics, has two functions: it controls the flow of propellants to the thrust cylinder, and it schedules the transfer from igniter fuel to running fuel (ref. 1).

For this investigation the fuel-flow sequence during starting was as follows:

(1) 100 percent igniter fuel was admitted during less than 5 percent of valve travel.

(2) 90 to 95 percent igniter fuel (5 to 10 percent JP-4) was admitted during approximately the next 60 to 65 percent of valve travel.

(3) A transition to 100 percent running fuel took place during the remaining 35 to 40 percent of valve travel.

All starting experiments were run in a nominal 200-pound-thrust rocket engine. Both aluminum and copper thrust cylinders were used; they were made by spinning tubing to the desired shape. The injector was an impinging-jet type with four sets of orifices spaced 90° apart. Each set consisted of one fuel orifice and two oxidant orifices with the fuel on the outside. Details of the thrust cylinder and injector design are shown in figure 2.

For low-temperature runs, the entire propellant system and engine was immersed in a dry ice - methylene chloride bath. A pump circulated the refrigerant in the bath and through the nozzle shroud (fig. 2); this equalized the bath temperature and cooled the nozzle during a run.

Instrumentation

Strain-gage-type pressure transducers were used to measure chamber pressure, fuel injection pressure, and oxidant injection pressure. Flow rates were measured by orifices fitted with strain-gage differential pressure transducers. Travel of the control valve pintles was measured by a linear potentiometer connected to the common yoke.

Outputs of the pressure transducers and position indicator were recorded by a multichannel oscillograph. Propellant and bath temperatures were measured by copper-constantan thermocouples and recorded by a multipoint self-balancing potentiometer.

Propellants

Oxidant. - Low-freezing-point red fuming nitric acid was the oxidant used for all runs. Two batches of acid were used; the compositions are shown in the following table:

	Compositions of oxidants, percent by weight	
	Batch 1	Batch 2
Nitric acid	75.40	75.40
Nitrogen dioxide	21.12	18.90
Water	3.48	5.70

Batch 2 was prepared for experiments below -80° F. No significant difference was observed in combustion behavior of the two oxidants.

Igniter fuels. - For most of the runs, the igniter fuels were hydrazine-water mixtures made by blending 93 percent hydrazine with distilled water. Mixture compositions were checked by measuring refractive index according to the method of reference 9. The following table shows the results of three tests made to compare the refractive index method with the more conventional acid titration method:

Sample	Hydrazine, percent by weight	
	Refractive index method	Titration method
1	92.6	92.6
2	74.1	73.9
3	65.6	65.6

Freezing-point data for hydrazine-water blends, obtained from references 10 and 11, are shown in figure 3.

Two other igniter fuels were used. One was undiluted 4-methyl-2-dimethylamino-1,3,2-dioxaphospholane (RF 208). The other was a blend of 70 percent triethylamine and 30 percent o-toluidine by volume, both of which were commercial grade.

Running fuel. - Jet fuel (JP-4) conforming to military specification MIL-F-5624B, amendment 1 was used as running fuel for all experiments. This material had the following properties:

Initial boiling point, °F	136
Final boiling point, °F	498
Hydrogen-carbon ratio	0.17
Net heating value, Btu/lb	18,700
Refractive index	1.4206
Aniline point, °F	132.4
Aromatics, percent by volume:	
Silica gel method	10.7
A.S.T.M. D875-46T	8.5

Procedure

The following procedure was used to fire the rocket engine. Refrigeration was started, after the propellant tanks were loaded, by filling the cooling bath with methylene chloride and adding dry ice. The liquid phase of the coolant was circulated through the nozzle shroud during cool-down and through the run. When all propellant temperatures were

within $\pm 3^{\circ}$ F of the desired test temperature, the tanks were pressurized and the rocket was fired. If more than one run was made from a single tank loading, sufficient time was allowed between runs for the entire system to return to the test temperature. When igniter fuels were changed, the starting fuel system was cleaned by draining, flushing repeatedly with a solvent such as acetone, and blowing dry with inert gas.

Accuracy of Data

The temperature data are estimated to have an error of $\pm 2^{\circ}$ F due primarily to the multipoint recorder. Calibrations of the chamber pressure and injection pressure transducers indicate an over-all accuracy of ± 10 pounds per square inch, or 2 to 3 percent error.

For these experiments, errors in flow measurement are due to errors in differential pressure readings, orifice flow coefficients, and propellant densities. Calibrations of the strain elements of the transducers and the recording system indicated a maximum variation of $\pm \frac{1}{2}$ percent. Flow calibrations showed that an orifice coefficient of 0.61 could be used with an error of ± 1 percent. In the range -80° to -40° F, propellant densities were found not to vary greatly, and the following average densities were used to simplify data reduction:

Acid, lb/cu ft	105
JP-4, lb/cu ft	47.5
Hydrazine-water blends, lb/cu ft	64.4
RF 208, lb/cu ft	68.7
<u>o</u> -Toluidine - triethylamine, lb/cu ft	51.0

This procedure introduced an error of about ± 1 percent. Therefore, the total error in flow measurement is about ± 2 percent, and the maximum error in characteristic velocity would be ± 5 percent.

RESULTS AND DISCUSSION

All the starting data are shown in tables I and II. Figure 4 shows the starting data for hydrazine-water blends superimposed on the freezing-point curve. Figures 5 to 9 are examples of flow, pressure, and valve position records obtained under various conditions. Only the starting portions of the oscillograms have been reproduced here.

For this study, a satisfactory start is defined as an ignition and transition from igniter fuel to JP-4 fuel that is free from over-pressure peaks or other abrupt chamber pressure variations.

Hydrazine-Water Blends

The starting data for hydrazine-water mixtures (table I and fig. 4) reveal that, for compositions containing more than 55 percent hydrazine, the lowest temperature for a satisfactory start was the freezing point of the igniter fuel. As hydrazine content was decreased, starting became erratic in a region extending from 47 to 54 percent at 0° F, and from 53 to 55 percent at the igniter-fuel freezing point. To the left of this region, energy release was apparently too low to ignite the JP-4. On the basis of the chamber pressure records, it appears that all the hydrazine compositions ignited with acid. It is possible, therefore, that more efficient utilization of heat released by the igniter fuel might shift the composition limits for satisfactory starts to lower values.

These data show that the present temperature limit of -40° F for JP-4 - white fuming nitric acid with eutectic hydrazine igniter fuel can easily be extended to -60° F, provided low-freezing red fuming nitric acid is used as the oxidant. Furthermore, it does not seem necessary to specify the eutectic composition (69 percent hydrazine), since mixtures containing as little as 55 percent hydrazine still gave satisfactory starts, as illustrated in figure 5. To prevent freezing of igniter fuel at -60° F, it would be necessary to limit the upper concentration to about 69 percent hydrazine.

Conditions that were maintained for the experiments corresponding to the unshaded data symbols of figure 4 can be described as follows:

(1) Valve opening time was long enough to prevent ignition over-pressure transients; that is, hydrazine accumulation during the ignition delay period was insufficient to cause explosions.

(2) Valve design together with the opening rates selected gave a relatively gradual transition from igniter fuel to JP-4 as compared with the transition obtained during so-called "slug" starts.

(3) Valve design promoted intimate mixing of JP-4 and igniter fuel throughout the transition period.

The results probably apply to other configurations and engine scales to the extent that the above conditions are maintained.

When condition (1) was eliminated by decreasing valve opening time to 0.3 second or less (table I(b), and shaded points, fig. 4) the starts were usually accompanied by over-pressure transients. An example is shown in figure 7. The starting limits seem to be about the same as for the long valve opening times (fig. 4).

Previous research, similar to that reported in reference 5 in which conditions (2) and (3) were not satisfied, resulted in a starting-temperature limit of about 20° F with hydrazine hydrate (64 percent hydrazine). When transition was made more gradual by suspending 5 percent of hydrocarbon in the hydrazine hydrate, starts could be obtained at -40° F with white fuming nitric acid.

Other Igniter Fuels

With low-freezing red fuming nitric acid at -85° F, 4-methyl-2-dimethylamino-1,3,2-dioxaphospholane (RF 208) has an ignition lag of 7 milliseconds (ref. 6), and a blend of 30 percent *o*-toluidine and 70 percent triethylamine by volume has an ignition lag of 28 milliseconds (ref. 7). These two fuels are examples of a series of organic materials which have been found to give very rapid ignition with nitric acid at extremely low temperatures.

Both fuels were evaluated by the same technique used for hydrazine-water blends, and both gave satisfactory starts at temperatures as low as -85° F (table II, and figs. 8 and 9(a)). Two of five *o*-toluidine runs resulted in abrupt flame-outs shortly after transition was completed (fig. 9(b)). Further investigation revealed that the *o*-toluidine blend when mixed with JP-4 at low temperatures deposited a sludge that did not completely redissolve on warming. Partial air-oxidation of the *o*-toluidine may have been the source of the sludge. A possible explanation of the flame-outs is that sludge deposited in the flow system broke loose and temporarily interrupted the flow; this could have been followed by a surge which caused flame-out. Figure 9(b) shows an interruption in flow and a slight surge in chamber pressure just before flame-out.

The one flame-out with RF 208 immediately followed a run with *o*-toluidine. It may be that the cleaning procedure did not remove sludge that had deposited during the previous run. Succeeding runs with RF 208 showed no evidence of flame-out, and it is believed that the single malfunction does not indicate a deficiency in this igniter fuel.

In all cases, ignition and transition were satisfactory, which indicates that the subsequent malfunctions cannot be related to the ignition-lag characteristics of these fuels.

It appears that the lower temperature limit for starting JP-4 - nitric acid can be -80° F, provided a low-freezing red fuming nitric acid is used, and the igniter is selected to have low viscosity and ignition lag. Care should also be taken that the igniter fuel and JP-4 do not precipitate insoluble materials when mixed. Since the alkylthiophosphites, for example, triethyltrithiophosphite, have physical and chemical properties similar to RF 208 (ref. 6), it may be expected that they too would be suitable for use at -80° F.

Combustion Pressure Oscillations

As shown in tables I and II, many of the runs exhibited combustion pressure oscillations after transition. Figures 5(b) and 8 show examples of this behavior. The increase in number of cases of pressure oscillations as temperature decreased suggests a significant increase in combustion time delay (ref. 12), which is the interval between propellant entry and conversion to combustion products. The primary causes are probably lowered mixing efficiency due to higher propellant viscosity and an injector design that is sensitive to pressure oscillations. Other research (refs. 1 and 13) has shown that triplet impinging jet injectors, such as the one used for this study, are prone to give unstable combustion of hydrocarbons and nitric acid over a wide range of mixture ratios.

Although the data reveal no apparent relation between temperature and oscillation frequency, the general level of frequencies (100 to 200 cps) implies a feed-system instability. The frequencies are thus in agreement with the idea that the rough burning is associated with poor propellant preparation. The occasional oscillation of 15 cps observed is probably a gas-regulator or propellant-line instability.

In every case, combustion of the igniter fuel was very stable (figs. 5, 6, 8, and 9) even at the lowest temperatures. Therefore, the subsequent instabilities were probably not connected with ignition transients and so did not affect the starting limits observed.

Igniter Combustion Efficiency

Theoretical combustion temperature and characteristic velocity for the igniter fuel were calculated for runs 14 and 18. No transition was obtained for run 14, which represents conditions at the lower boundary of the useful starting range; run 18 represents conditions well within the useful starting range. The products of combustion were assumed to be nitrogen dioxide, nitrogen, and water vapor; thermal data were taken from references 14, 15, and 16. For run 14 the theoretical values obtained were 948° F and 2360 feet per second, and for run 18 the values were 2540° F and 3656 feet per second. For these same runs, the experimental characteristic velocities and the characteristic-velocity efficiencies were 1495 feet per second and 63 percent, and 3410 feet per second and 93 percent, respectively.

The poor efficiency and low temperature obtained for run 14 show why transition could not be obtained with igniter fuels having less than 55 percent hydrazine. They also show that, for oxidant-fuel ratios far removed from stoichiometric, the main-stage injector gives poor mixture preparation for the igniter fuel. Consequently, if the igniter fuel could be burned more efficiently, it appears possible to extend the composition limits for hydrazine-water mixtures to values lower than those found in this investigation.

SUMMARY OF RESULTS

Chemical starting studies were made in a 200-pound-thrust rocket engine using JP-4 and a low-freezing red fuming nitric acid as main-stage propellants. The igniter fuels evaluated were hydrazine-water blends, 4-methyl-2-dimethylamino-1,3,2-dioxaphospholane (RF 208), and a blend of 70 percent triethylamine and 30 percent *o*-toluidine. A three-fluid propellant control valve was used, which also scheduled the transition from igniter fuel to main-stage fuel.

The results of these studies can be summarized as follows:

1. For hydrazine-water blends containing more than 55 percent hydrazine, starting was limited only by the freezing point of the mixtures. At -60° F, for example, limiting mixture compositions were 55 percent and 69 percent hydrazine.

2. With RF 208 and the triethylamine - *o*-toluidine igniter fuels, starts were obtained at temperatures near the freezing point of the acid (-85° to -80° F).

3. Below -80° F, several flame-outs were experienced after full main-stage pressure had been reached. For the *o*-toluidine igniter fuel, it was postulated that residual sludge, deposited at the low temperature, temporarily interrupted the flow.

4. The number of runs exhibiting low-frequency combustion instability (100 to 200 cps) increased as temperature was decreased. This was attributed to an increase in combustion-time delay due to poor propellant preparation with the injector used.

5. Calculated combustion efficiencies for the hydrazine-water mixtures were high at the hydrazine-rich limit and low at the hydrazine-lean limit.

Lewis Flight Propulsion Laboratory
National Advisory Committee for Aeronautics
Cleveland, Ohio, May 6, 1955

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TABLE I. - STARTING DATA FOR HYDRAZINE-WATER IGNITER PULSES

(a) Valve opening time, > 0.8 sec

Run	Temperature, °F	Hydrazine in igniter fuel, weight percent	Transition completed	Valve opening time, sec	Main-stage chamber pressure, lb/sq in. gage	Maximum chamber pressure transition failure, lb/sq in. gage	Main-stage flow rate, lb/sec	Main-stage oxidant-fuel ratio	Combustion characteristics		Transition type	Remarks
									Type	Frequency, cps		
									(a)	(b)		
1	80	74.1	✓	1.7	(c)		d1.08	d5.3				
2	40	48.1		1.3		170						
3	0	47.9	✓	1.5	438		1.16	5.0	S	210	Step	
4	0	48.1		1.3		170						
5	-26	50.3	✓	3.0	392		1.15	8.8	S	110-150	Step	15 cps chug
6	-26	47.9		2.0		218						
7	-37	50.5	✓	.8	408		.98	4.17	IR	140	Smooth	15 cps chug
8	-38	54.0	✓	(c)	412		.97	5.8	S	180	Step	
9	-39	46.1		1.4		188						
10	-39	55.0	✓	1.0	397		1.05	3.8	S	110	Step	
11	-40	65.6	✓	1.2	(c)		d1.08	d5.2	S	(c)	Smooth step	
12	-40	47.9		1.8		218						
13	-45	51.9	✓	2.2	370		1.10	4.8	R	200	Smooth step	
14	-45	54.0		1.7		208						
15	-48	72.3	✓	1.8	395		1.14	4.4	S	200-220	Smooth	
16	-50	54.8	✓	.8	445		1.02	5.5	S	100-110	Smooth step	
17	-52	70.3	✓	1.2	260		d1.48	d4.4	S	170	Smooth	Low combustion efficiency
18	-52	72.3	✓	2.0	456		1.01	4.4	R	210	Smooth	
19	-56	54.0		1.3		195						
20	-62	55.0	✓	1.0	384		1.05	6.1	S	110	Step	15 cps chug
21	-63	55.0	✓	1.1	408		1.04	4.8	S	170	Smooth step	
22	-63	68.0	✓	5.0	385		1.09	4.8	R	170-180	Smooth	
23	-64	55.0	✓	1.1	380		1.00	3.97	R	140	Smooth step	
24	-64	55.0	✓	1.7	394		.89	3.8	S		Smooth step	
25	-65	55.0	✓	1.2	(c)		1.13	4.5	R	140		
26	-66	55.0	✓	1.1	(c)		1.14	4.8	R	140		
27	-66	55.0	✓	1.1	(c)		1.02	5.4	R	136		
28	-65	55.8	✓	1.1	(c)		1.21	8.7	S			
29	-70	55.0	✓	1.1	348		1.10	4.7	R	140		
30	-71	61.8		1.8		208						
(b) Valve opening time, < 0.3 sec												
31	-20	74.1	✓	0.30	338		(c)		R	190	Sharp peak	
32	-22	74.1	✓	.25	354		(c)		R	150	Sharp peak	
33	-40	51.9		.20		178						
34	-40	51.9		.31		148						
35	-46	70.3	✓	-----	(a)							Exploded after 0.15 sec
36	-49	70.3	✓	.20	(c)		(c)		S	180	Small peak	
37	-56	65.6	✓	.22	317		(c)		R	180	Smooth	
38	-57	65.4	✓	.18	357		(c)		S	210		Sharp peak at start
39	-57	65.4	✓	.22	329		(c)		S	210		Sharp peak at start
40	-60	65.8	✓	.21	327		(c)		R	180		Sharp peak at start
41	-68	54.8	✓	.25	382		1.10	4.07	R	160	Sharp peak	Sharp peak at start
42	-68	54.8		.24		62						
43	-68	54.8	✓	.28	362	21						

^aS = smooth; R = rough; IR = intermittent roughness.

^bFrequencies noted for smooth combustion represent very low amplitude disturbances.

^cNo record.

^dApproximate value.

^eNo oxidant flow record.

TABLE II. - STARTING DATA FOR RF 208 AND TRIETHYLAMINE - o-TOLUIDINE IGNITER FUELS

(a) RF 208 (4-methyl-2-dimethylamino-1,3,2-dioxaphospholane)

Run	Temperature, °F	Transition completed	Valve opening time, sec	Main- stage chamber pressure, lb/sq in. gage	Main- stage flow rates, lb/sec	Main- stage oxidant- fuel ratio	Combustion characteristics		Transition type	Remarks
							Type (a)	Frequency, cps (b)		
44	-42	✓	1.35	371	1.23	6.17	R	210	Smooth with dip	Flame-out after 2.2 sec
45	-72	✓	1.60	409	1.04	4.24	S	170	Smooth	
46	-78	✓	1.44	400	1.02	4.37	R	140	Smooth	
47	-82	✓	1.40	397	.99	3.90	R	190	Smooth	
48	-82	✓	.60	381	1.03	5.92	R	185	Smooth	
49	-83	✓	.85	375	1.05	5.81	R	145	Smooth with dip	
(b) 70 Percent triethylamine - 30 percent <u>o</u> -toluidine										
50	-80	✓	1.2	400	1.04	4.48	R	140	Smooth	Flame-out after 1.9 sec
51	-80	✓	1.1	400	1.05	4.52	S	---	Smooth	Flame-out after 1.4 sec
52	-82	✓	.60	399	1.05	4.35	R	125	Smooth	
53	-82	✓	.90	432	1.03	4.11	S	150	Smooth	
54	-85	✓	1.60	(c)	1.01	6.07	S	---	-----	

^aS = smooth; R = rough.^bFrequencies for smooth runs are for very low amplitude disturbances.^cNo record.

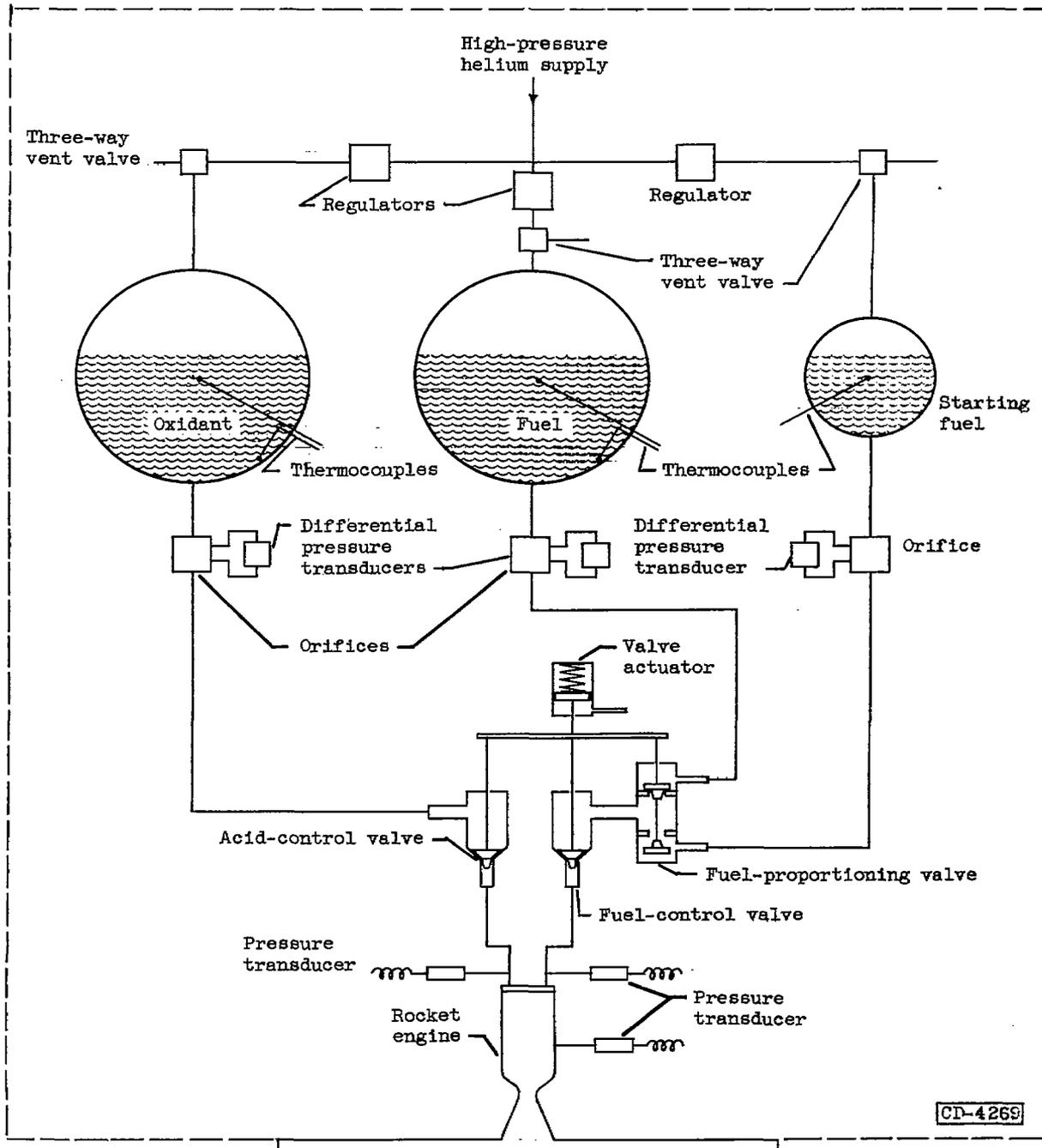


Figure 1. - Diagrammatic sketch of rocket engine flow system for starting experiments. Complete system immersed in refrigerant bath.

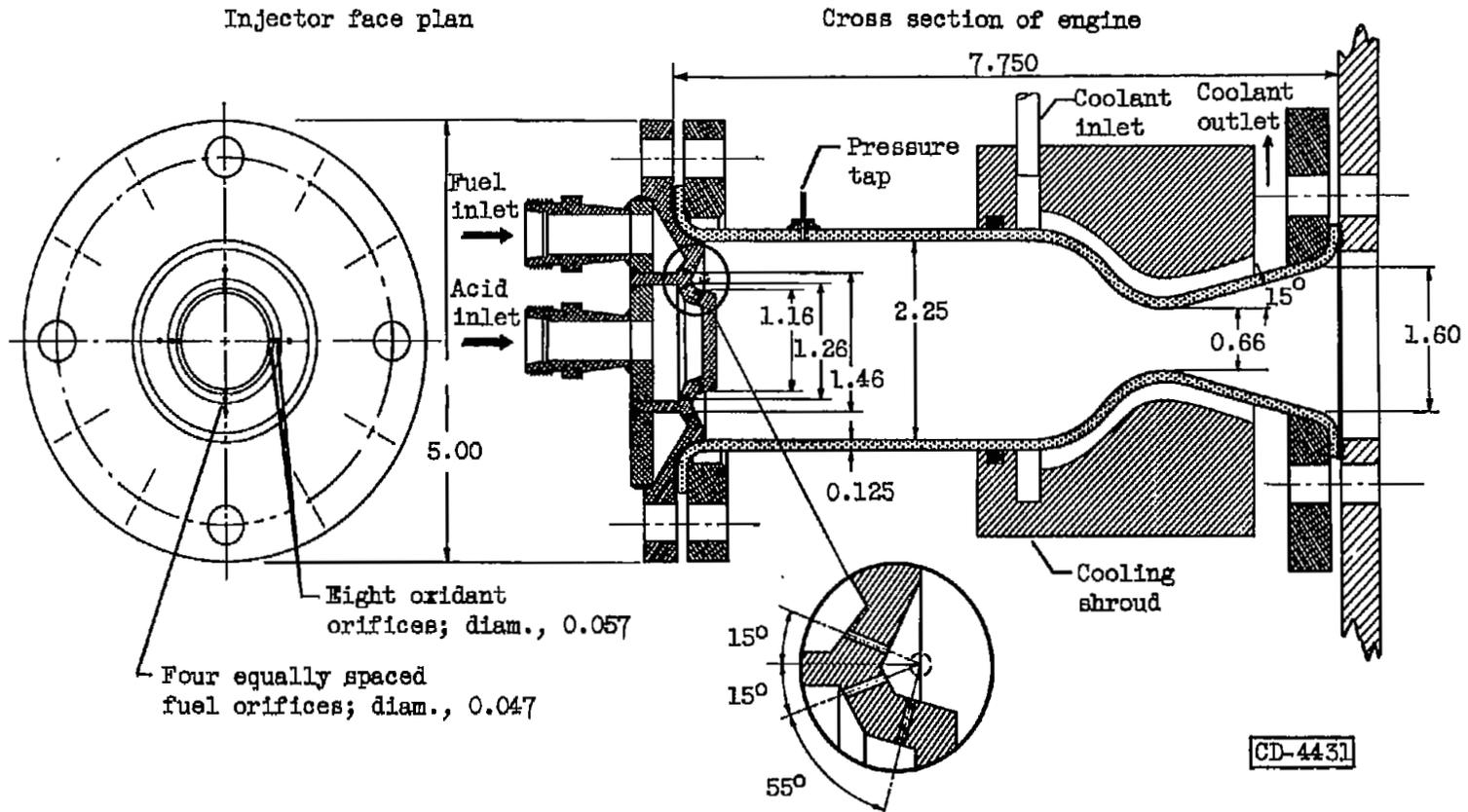


Figure 2. - Detailed views of 200-pound-thrust engine. Characteristic length, 50 inches.
(All dimensions in inches.)

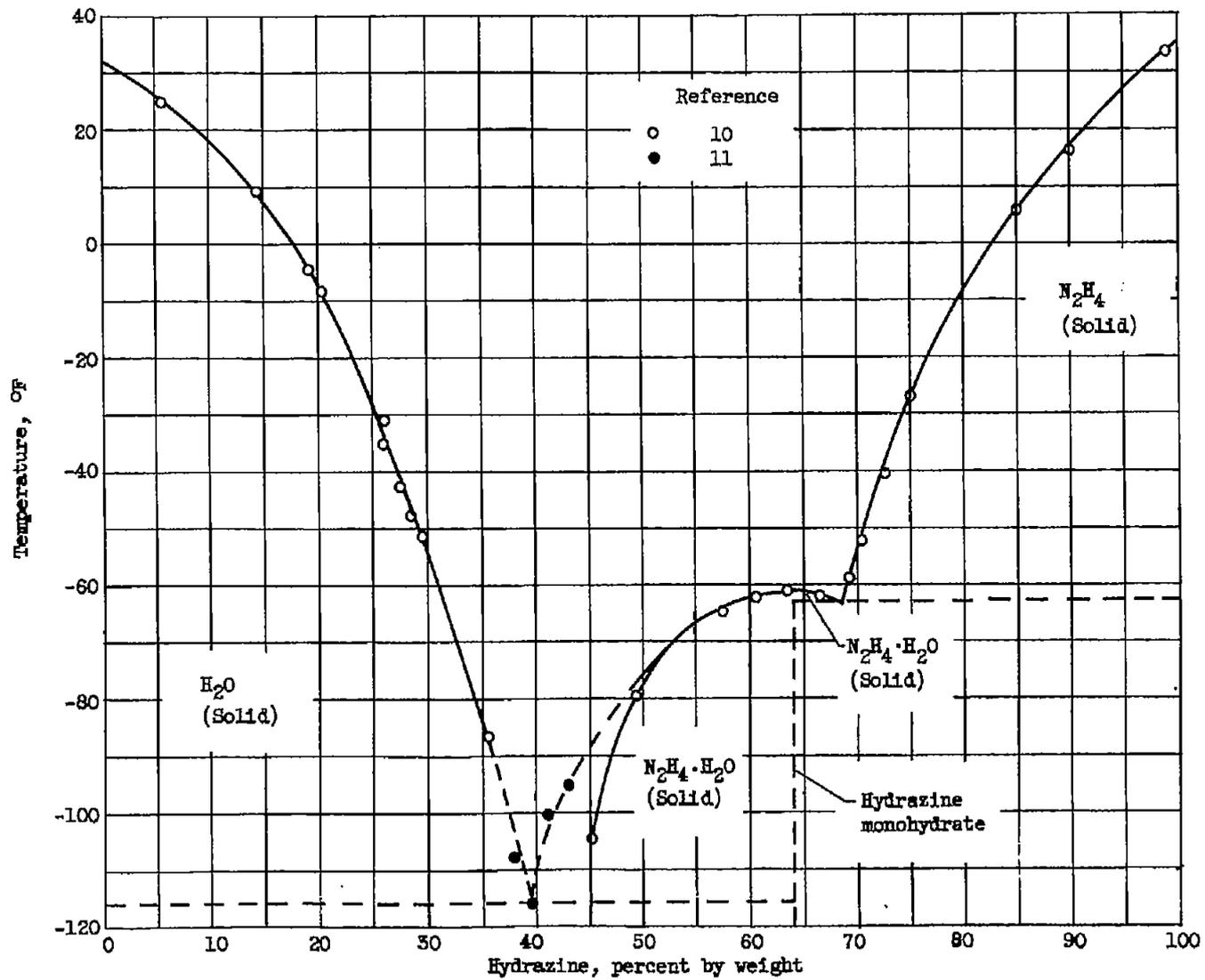


Figure 3. - Freezing-point diagram for hydrazine-water mixtures.

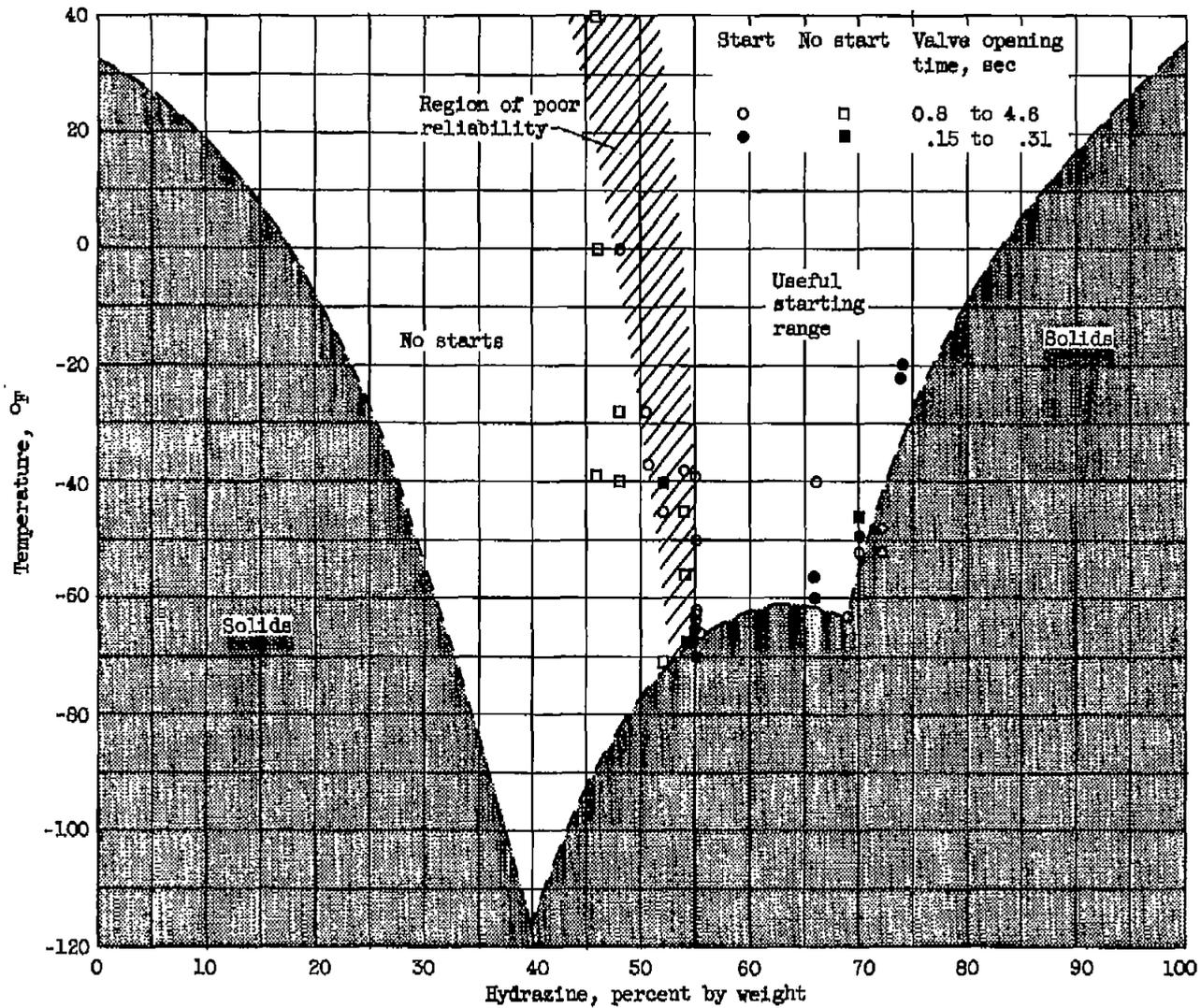
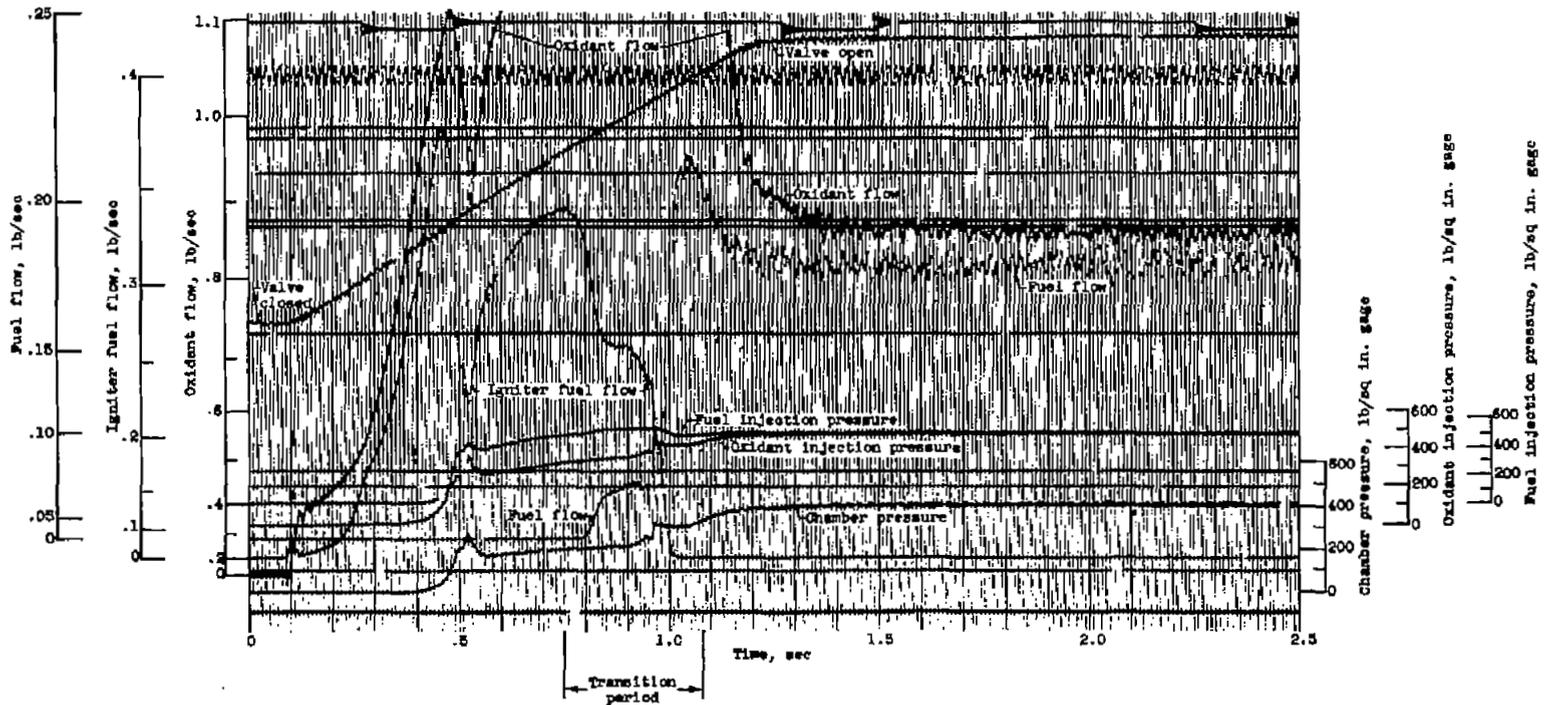
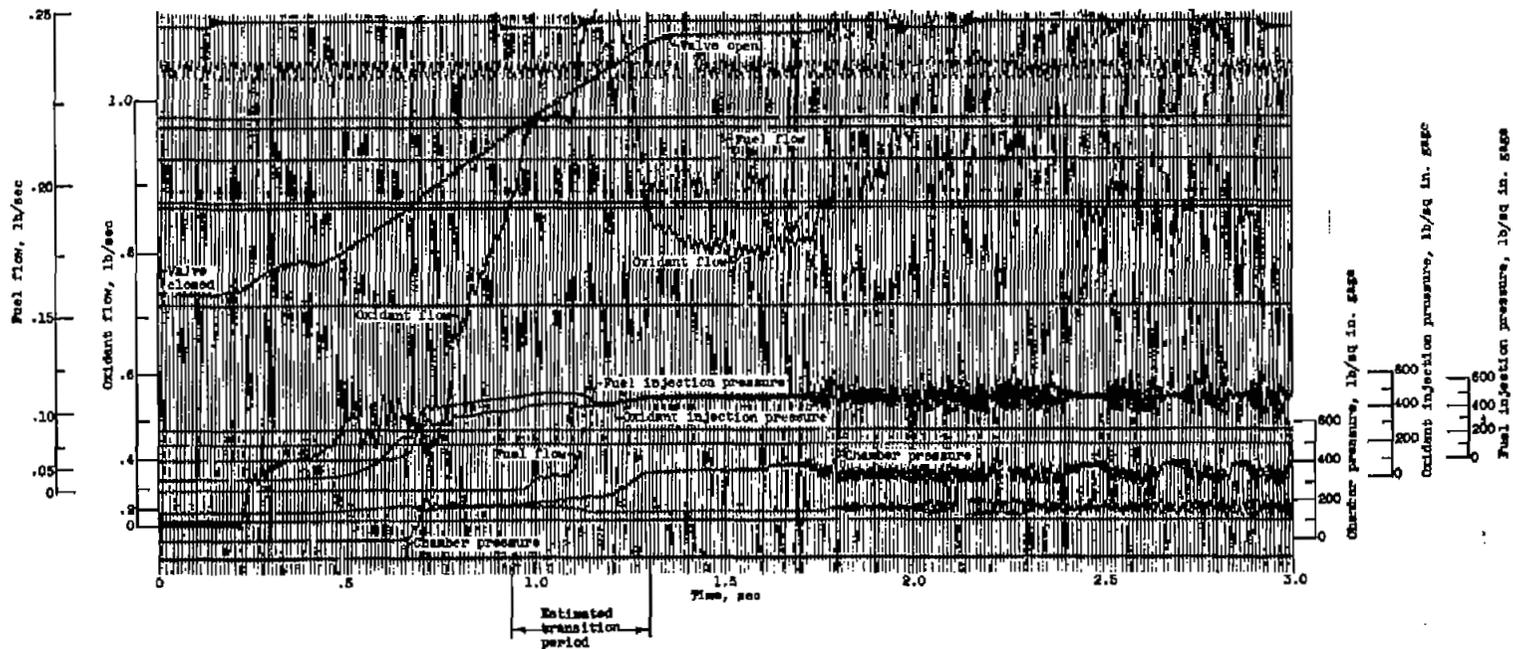


Figure 4. - Temperature-composition limits on hydrazine-water mixtures for starting JP-4 - nitric acid system in 200-pound-thrust rocket engine.



(a) Smooth combustion following transition. Run 21; -63° F.

Figure 5. - Pressure and flow curves during engine starts with 55 percent hydrazine - water igniter fuel.



(b) Rough combustion following transition. Run 23; -64°F ; no igniter fuel flow record.

Figure 5. - Concluded. Pressure and flow curves during engine starts with 55 percent hydrazine - water igniter fuel.

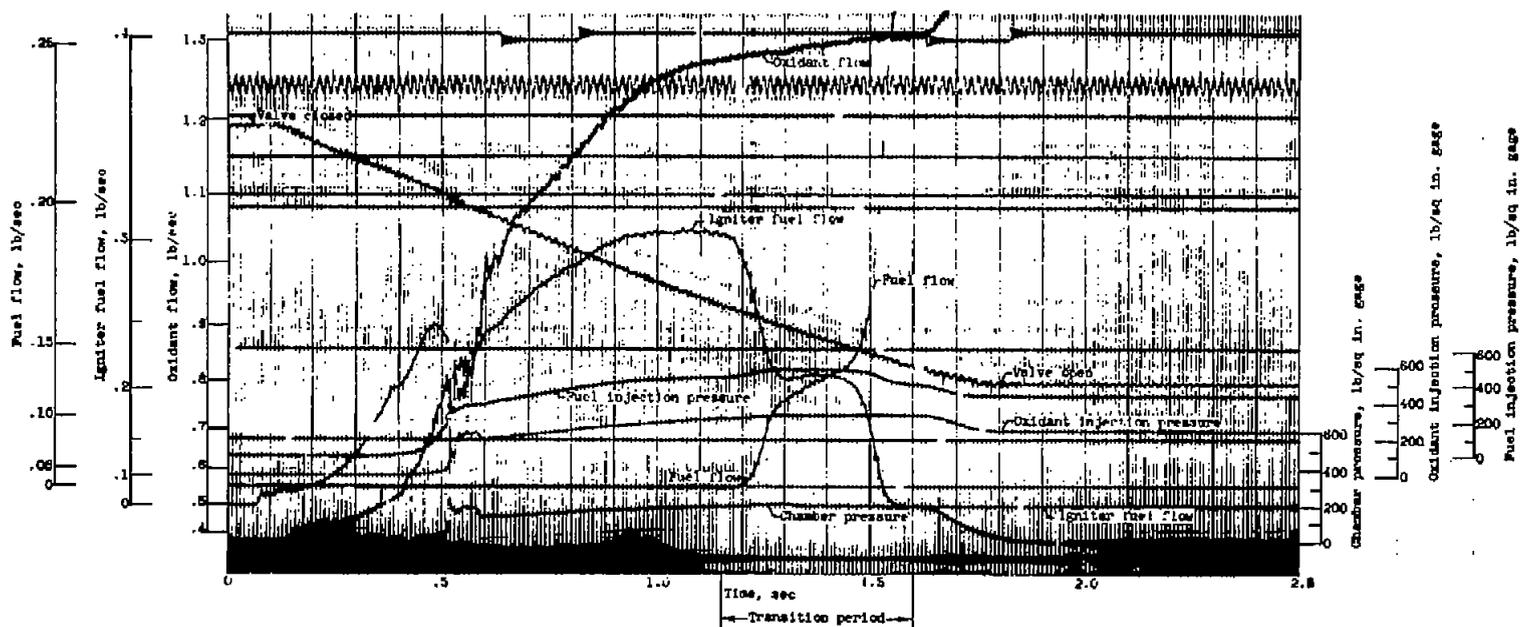


Figure 6. - Typical pressure and flow curves during unsuccessful engine start with hydrazine-water igniter fuel. Run 14; 54 percent hydrazine; -45° F.

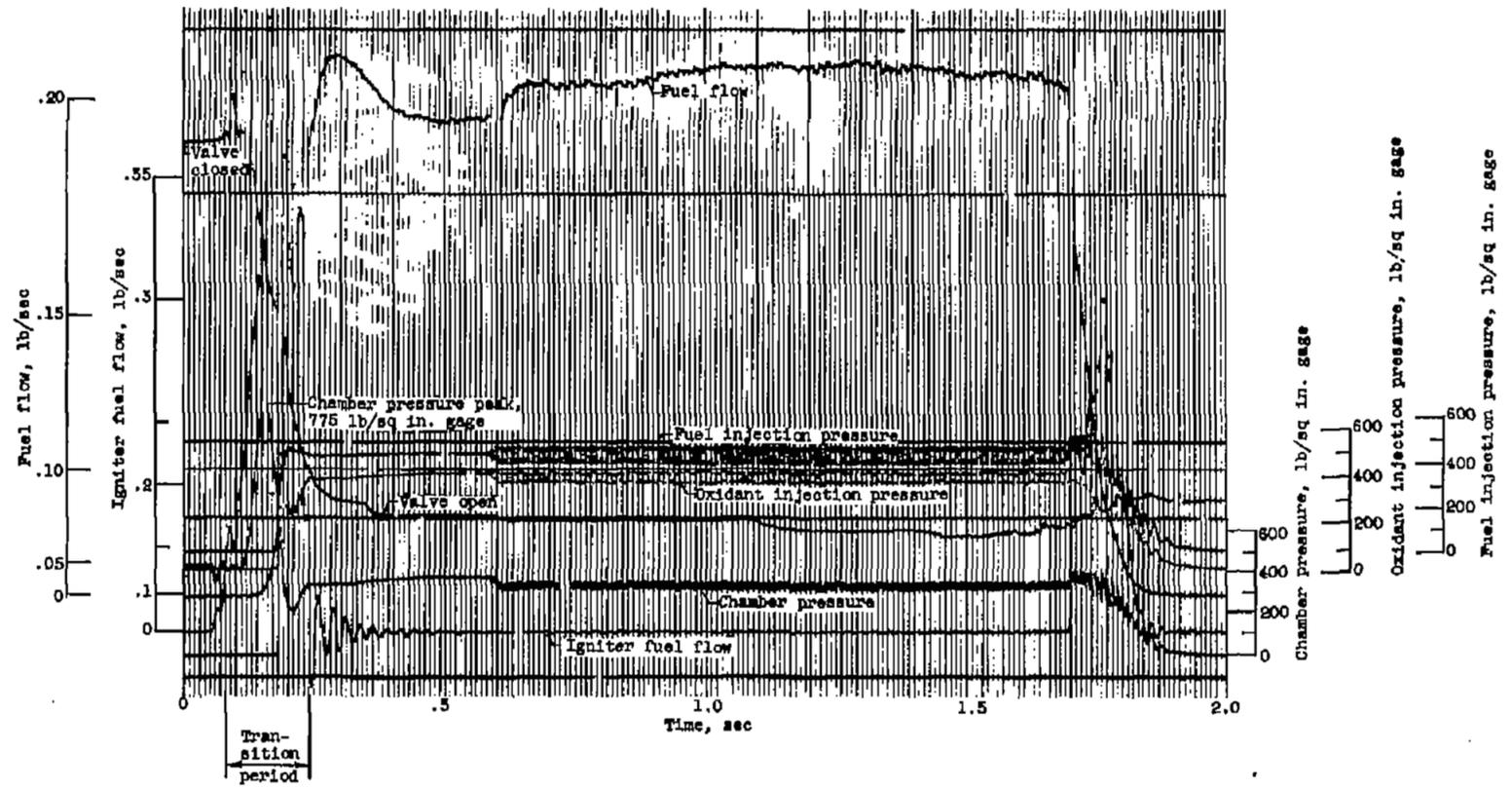


Figure 7. - Pressure and flow curves during engine start with hydrazine-water igniter fuel and with extremely short valve opening time. Run 31; 74percent hydrazine; -20° F; no oxidant flow record.

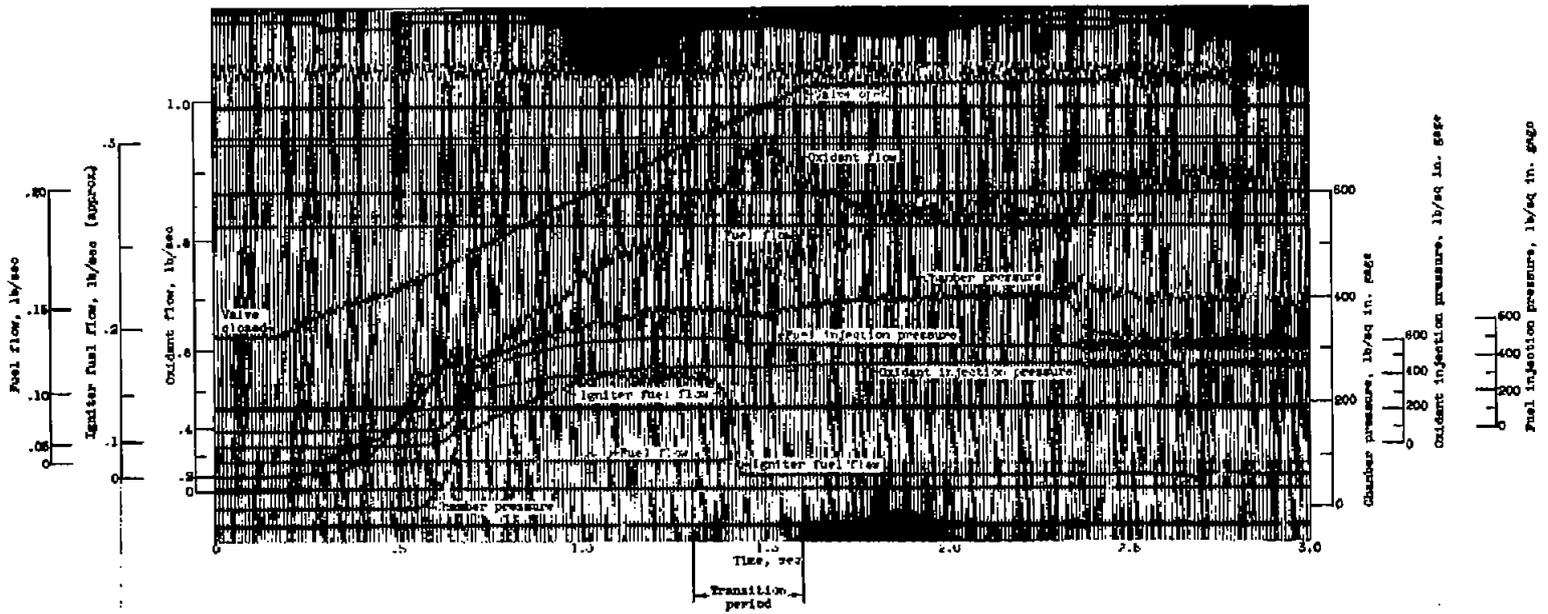
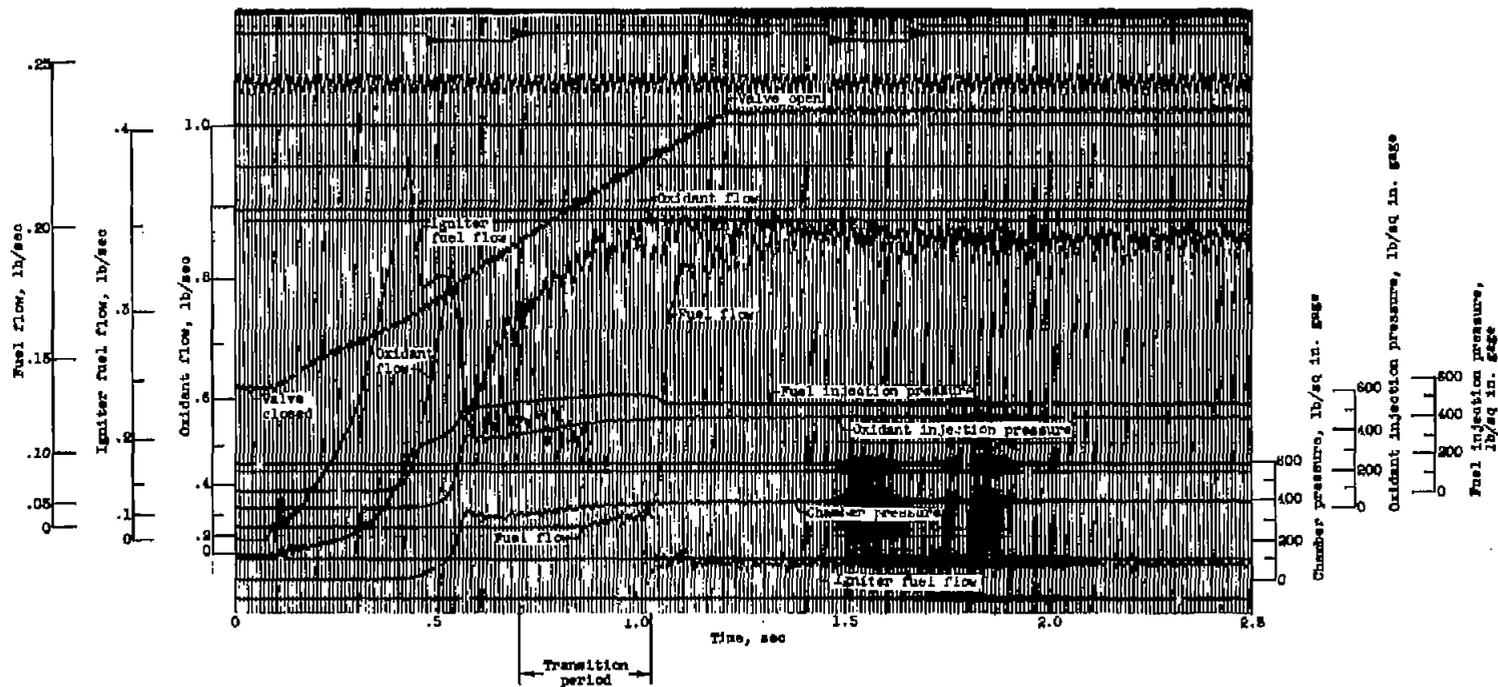
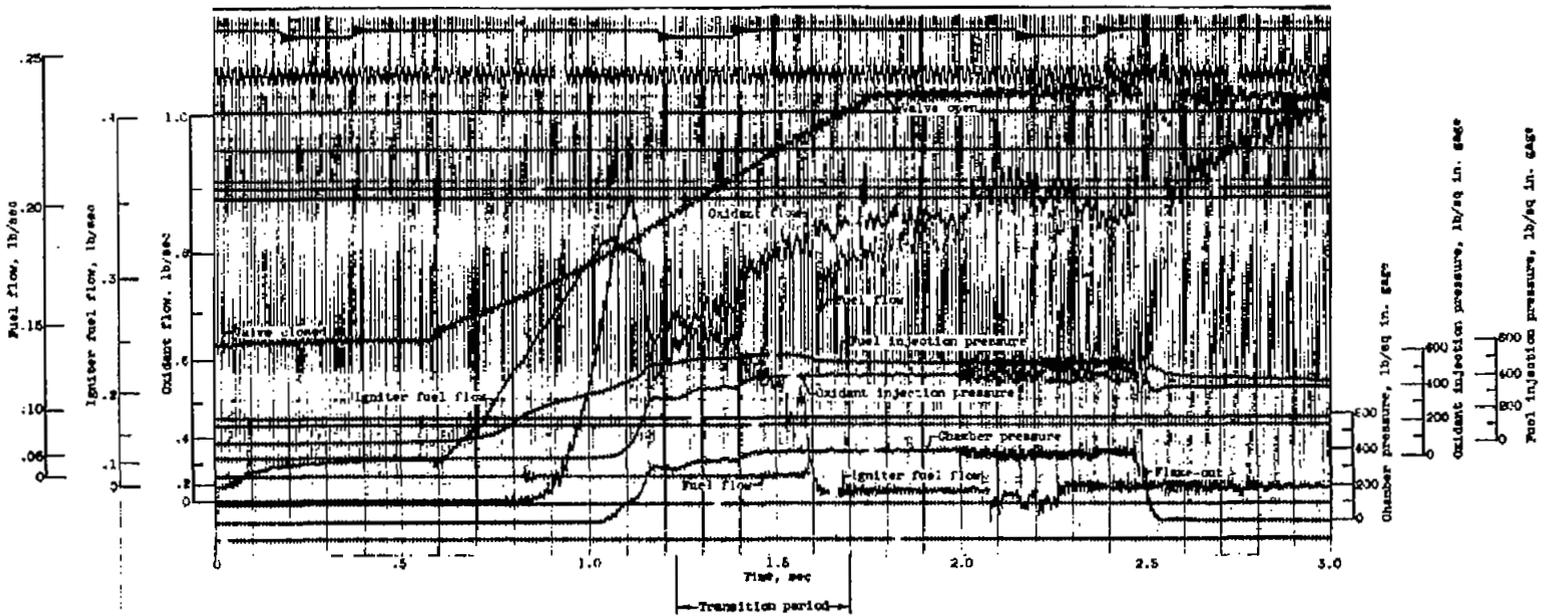


Figure 8. - Pressure and flow curves during engine start at -78°F with RF 208 igniter fuel. Run 46.



(a) Normal start. Run 51.

Figure 9. - Pressure and flow curves during engine starts with o-toluidine - triethylamine igniter fuel at -80° F.



(b) Flame-out following transition. Run 50.

Figure 9. - Concluded. Pressure and flow curves during engine starts with o-toluidine - triethylamine igniter fuel at -80° F.



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