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DETERMINATION OF WATER-GLYCOL COOLANT FLAMMABILITY

INTERNAL NOTE NO. MSC-IN-67-EP-10

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INTRODUCTION

The primary purpose of this test program was to determine the flammability of the water-glycol coolant used on board the Apollo spacecraft in order to ascertain to what extent, if any, this fluid constitutes a significant fire hazard under flight conditions. The secondary purpose was to determine the flammability limits of varying concentrations of water-glycol under a variety of temperature and pressure conditions and with various ignition sources. The glycol concentration was varied between 35% and 100% glycol. These percentages cover the concentration of the CSM (ECS) glycol as well as the LM glycol. Other glycol concentrations were used in order to establish the flammability curve at various temperature, pressure and ignition source conditions. In all cases, pure oxygen was used as the environmental gas into which the water-glycol mist was sprayed.

GENERAL TEST CONDITIONS

Glycol varying between 35% and 100% concentration was sprayed into the reactor which was maintained under pure oxygen conditions varying between 3 psia and 20 psia. The temperature of the inlet water-glycol spray was varied between ambient and 400°F. No attempt was made to control the reactor temperature. The oxygen was admitted into the top of the reactor at an approximate rate of 0.7 lb/min. Glycol flow was maintained at approximately 0.4 gpm. The ignition sources used were:

1. Electrical spark - 0.3125 joules energy
2. Exploding bridgewire - 6.25 joules energy
3. Hot platinum wire - 2500°F 117 watts energy
4. Fireball, rubber - 2250°F temperature

Ignition sources were positioned five feet below the water-glycol spray head.

DESCRIPTION OF TEST APPARATUS

The test apparatus consisted of a 6-inch diameter by 8-feet tall stainless steel column fully instrumented for pressure and temperature detection up and down the column interior. This test apparatus (reactor) was outfitted with several view ports through which camera coverage was achieved and also through which light was focused upon the action within the chamber. The chamber and spray head was constructed so that optimum conditions would occur for production of a fire within the system. The reactor, which was fitted with thermocouples at various positions, as well as pressure transducers, was monitored remotely by movie cameras, oscillographs and strip-chart recorders to indicate flammability conditions which might occur during the test.

Laminar flow of oxygen was achieved by pressurizing with oxygen which was introduced through four inlets into the top of the reactor. Oxygen flow was approximately 0.7 lbs/min. The pre-determined reactor pressure was maintained by use of a 300 cubic feet per minute capacity vacuum pump which was connected to a 6-foot diameter vacuum chamber; by this arrangement the pressures either below or above atmospheric conditions were precisely maintained. Note figures 1 and 2 for details of the flow reactor.

TEST PROCEDURE

Nominal operating conditions were established manually at the reactor prior to the actual test firing. Once the various flows had been established and the instrumentation was in proper working condition, the personnel retired to a safe area in the control room. Flows of both the oxygen and water-glycol were initiated by remote control from within the control room; the pressure in the system was also remotely controlled. By using television coverage the operators were able to determine whether spray and ignition source conditions were correct within the spray area. As soon as nominal spray and ignition source was properly achieved a countdown was initiated so that uniform conditions could be maintained between firings. In general, if an abnormally high flammability condition resulted from an initiated fire, an audible explosion would occur. Temperatures and pressures were recorded remotely. Visual coverage was achieved with movie cameras set at 200 to 400 frames per second. The reactor was protected from explosion damage by use of a metal burst disc with a 30psi rating. The greater portion of the tests were conducted with CSM (ECS) coolant. Numerous tests were made starting at ambient temperature and pressure conditions and proceeding upward to cover a wide variety of conditions; tests under reduced pressure were also performed.

RESULTS AND DISCUSSION

Series A - Spark and Exploding Bridgewire (EBW energy source).

It was determined that under proper conditions of glycol concentration and temperature that fire could be initiated with the EBW if the glycol percentage was at least 65%. The minimum inlet spray temperature was found to be 230°F for flammability to occur. Under no conditions could fire be initiated in the CSM (ECS) coolant. At 70-75% glycol concentration and temperature slightly above ambient (80-100°F) at the inlet of the spray nozzle, some fires occurred with the EBW. When 80-100% glycol was used the exploding bridgewire, almost in every case, initiated a bad flame condition regardless of the inlet temperature. There was no attempt made to heat anything except the incoming glycol-water solution. (It should be noted that the spark source never initiated any fires, even at 100% glycol concentration; all fires that occurred were initiated by the EBW).

Reduced pressures of 3-5 psia oxygen seemed to reduce the flammability as opposed to the 14.7 to 20 psia; the presence of greater oxygen concentration due to the higher pressures seemed to predominate over the other factors such as relatively high glycol vapor pressure under the reduced pressure conditions. Refer to Table I for details of the test with the EBW. Also, note Figure 3 showing the flammability curve with the EBW ignition source.

Series B - Heated Platinum Wire Ignition Source.

Following the EBW tests, testing was continued under the same general conditions of temperature, pressure, flow rates and concentrations but with a platinum wire ignition source heated to 2500°F. It was not deemed necessary, however, to test at concentrations above 70% since several ignitions (3 out of 10 runs) occurred with the CSM (ECS) coolant under heated conditions. The 70% glycol solution ignited at temperatures slightly above ambient (140°F). See Table I for a tabulation of the results using a heated platinum wire ignition source. Figure 4 represents the flammability curve with varying glycol percentages.

Series C - Fireball Ignition Source.

The heated platinum wire tests were followed by tests utilizing an ignition source consisting of a flaming 1-inch rubber cube which reached a maximum temperature of approximately 2250°F. Under these test conditions flammability occurred in every case with the CSM (ECS) glycol. No flammability occurred, however, with the 37.5% LM glycol. See Table I for a tabulation of results.

CONCLUSIONS

It is evident from the tests that the CSM (ECS) glycol coolant is flammable and will contribute to the intensity of a fire if it is sprayed onto an existing fire source. It is also apparent that considerable ignition energy must be present before burning will occur. This is illustrated by the fact that even 100% glycol did not burn in pure oxygen when a electrical spark of approximately 0.3 joules of energy was discharged into a very hot (300°F+) mist of the material. The exploding bridgewire, which produced approximately 6 joules of energy, consistently ignited hot solution of 65% to 100% glycol. This demonstrates the increased hazards with increased energy from the ignition source.

As the energy from the ignition source was increased through the application of a white hot platinum wire and followed by a burning rubber ball, flammability occurred in less concentrated glycol solutions. The obvious conclusion is that CSM (ECS) coolant containing 62.5% concentration of glycol is a fuel once it ignites. It is postulated that this ignition cannot occur so long as the water is present with the glycol. If the water is boiled off by a hot energy source, the glycol will then catch on fire and release sufficient energy to rapidly boil off more water thus propogating the fire until the glycol is completely exhausted.

ADDENDUM

The Stabilities of Glycols and Similar Coolant Fluids in Oxygen:

Ethylene glycol, propylene glycol and similar fluids are commonly used as coolant fluids but usually they are used in air rather than in pure oxygen and they normally would never be exposed to higher temperatures. Under these conditions, these liquids are safe to use and flammability is not a problem. Actually, it is well known that such coolant liquids do burn and one can calculate from available thermodynamic data that the combustion will be exceedingly exothermic. For example, the burning of one mole (62 grams) of ethylene glycol in oxygen will evolve 284,000 calories of heat, or about 4580 cal/gram. Other related compounds burn with similarly large evolution of energy. Flash points and other important properties for typical organic compounds which might be used as coolants are as follows:

Compound	Freezing Point	Norm. Boiling Point (unless indicated)	Flash Point	Source
Ethylene Glycol	-13°C	197°C	241-248°C	CCD,p.466
Propylene Glycol	-59°C	188°C	210°F	CCD,p.946
Dibutyl Phthalate	-35°C	227-235°C/37 mm	340°F	CCD,p.360
Dicapryl Phthalate	-	227-234°C/4.5 mm	395°F	CCD,p.362
Fluorochemical,FC-43	-	337-355°F	Inert	CCD,p.506
Fluorochemical,FC-75	-	210-225°F	Inert	CCD,p.506
Silicone Fluids	-	188°C and up	Inert	CCD,pp.399, 1019, 1021

(CCD = Condensed Chemical Dictionary, Reinhold, 1961)

It thus appears that ethylene glycol and propylene glycol, although commonly used as coolants in automobiles and other non-critical devices, are actually very poor choices from many viewpoints:

a. Glycols and most alcohols and esters burn exothermically with sufficient energy evolved to vaporize away considerable water even from solutions. Only in dilutions of 10-20% glycol by weight is one likely to have a reasonably flame-resistant coolant, and even these diluted solutions can be burned under extreme conditions.

b. Ethylene glycol is appreciably toxic to humans but propylene glycol is much safer.

Some other possible choices for coolants would include the following:

a. Dibutyl phthalate or dicapryl phthalate are about equally exothermic in combustion, but have much lower vapor pressures and pose much less fire hzard due to their definitely higher flash points. They are believed to be relatively non-toxic. Their viscosities are comparable with the glycols.

b. The fluorochemicals, FC-43 and FC-75 are marketed by 3M for use as "colorless, odorless, high temperature heat transfer media." Their viscosities are comparable with those of hydrocarbons and the liquids are "characterized by extreme chemical inertness, do not burn and are stable to temperatures of 500°F or more." They are typically used as fire extinguishing agents, lubricants, hydraulic fluids, coolants, etc.

c. Silicone fluids, for example, available from Dow-Corning Corporation, are characterized by heat stability, and resistance to oxidation and weathering. They are usable under severe operating conditions. When burned, there is a tendency to form a protective nonflammable layer of SiO_2 on a surface.

Recommendations (If the ECS coolant is subject to elevated temperatures)

a. Reject the use of glycols as coolants since it appears that flammability will be a serious problem at all appreciable concentrations. In addition, ethylene glycol is well-known to cause a variety of undesirable physiological effects, including depression, nausea, vomiting, coma, respiratory failure, etc., (Merck Index, 1960, p.428).

b. Evaluate from engineering and flammability viewpoints, the commercially available esters of phthalic acid, like dibutyl phthalate and dioctyl (sometimes called dicapryl) phthalate. These low-vapor pressure liquids are much more difficult to ignite, are relatively non-toxic and should be reasonably satisfactory coolants.

c. Collect information on the commercially available Fluorochemicals from 3M and Silicone Liquids from Dow-Corning and/or General Electric and/or Union Carbide to see what these fluids have to offer. They are definitely more inert and in many cases are flame inhibitors. They can be prepared with a variety of viscosity and freezing point characteristics so that other requirements can be met.

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RUN NO.	GLYCOL %	GLYCOL INJ. TEMP. (°F)	O_2 PRESS. (PSI)	GLYCOL FLOW (GPM)	OX. FLOW (#/MIN)	T/C 1 MAX. (°F)	T/C 1 TIME TO MAX. (SEC)	T/C 2 MAX. (°F)	T/C 2 TIME TO MAX. (SEC)	T/C 3 MAX. (°F)	T/C 3 TIME TO MAX. (SEC)	T/C 4 MAX. (°F)	T/C 4 TIME TO MAX. (SEC)
1-A	85	120	15.2	0.48	0.71	92	0.15	1924	0.80	2136	0.62	2116	0.60
2-A	85	127	14.68	0.48	0.71	325	0.24	1873	0.90	2062	0.68	2067	0.68
3-A	80	141	14.68	0.48	0.71	169	0.96	1652	0.85	1796	0.61		
4-A	73	240	15.8	0.48	0.71	175	0.26	1428	2.06	1617	0.95		
5-A	62.5	150	3.8	0.49	0.71	NO REACTION							
6-A	62.5	155	[illegible].76	0.49	0.71	NO REACTION							
7-A	62.5	250	16.54	0.49	0.71	NO REACTION							
8-A	62.5	335	15.2	0.48	0.71	NO REACTION							
9-A	62.5	425	14.8	1.00	0.71	NO REACTION							
10-A	67.5	255	14.7	0.40	0.71	115	0.53	581	0.58	619	0.58		
11-A	73	255	14.7	0.40	0.71	1848	18.43	1305	1.30	1530	1.30	1489	1.30
12-A	67.5	250	14.7	0.44	0.71	NO REACTION							
13-A	62.5	256	3.6	0.44	0.3	NO REACTION							
14-A	67.5	267	2.7	0.50	0.3	NO REACTION							
15-A	67.5	252	14.7	0.48	0.71	2329	15.13	1274	1.28	1277	1.06	1345	1.28
16-A	65	324	14.7	0.40	0.71	1348	2.61	1569	2.48	1608	2.55	1489	2.61
17-A	67.5	188	14.7	0.45	0.71	NO REACTION							
18-A	65	193	14.7	0.44	0.71	NO REACTION							
19-A	65	234	15.7	0.48	0.71	NO REACTION							
20-A	67.5	193	14.7	0.48	0.71	NO REACTION							
21-A	65	278	14.7	0.48	0.71	2165	10.76	1000	2.41	1548	2.41	1681	2.41
22-A	70	187	14.7	0.48	0.71	1067	1.36	1256	0.67	1476	0.63	1560	0.64
23-A	65	250	14.6	0.48	0.71	NO REACTION							
24-A	65	267	14.6	0.48	0.71	2133	5.86	1085	1.17	1259	1.17	1322	1.17
25-A	70	172	15.8	0.46	0.71	1073	11.39	1250	1.23	1530	0.97	1518	0.95
26-A	75	131	14.7	0.46	0.71	1627	1.75	776	0.94	1419	0.57	1415	0.62
27-A	80	111	14.2	0.45	0.71	1646	8.17	1405	0.52	1599	0.54	1604	0.46
28-A	75	95	14.5	0.46	0.71	303	0.89	1551	1.13	1864	0.92	2000	0.79
29-A	70	90	15.1	0.46	0.71	NO REACTION							
30-A	62.5	352	22.3	0.48	0.71	NO REACTION							
31-A	70	140	14.7	0.48	0.71	258	1.91	1622	3.43	2049	1.77	2317	1.64
32-A	70	107	15.0	0.47	0.71	NO REACTION							
33-A	70	121	15.8	0.48	0.71	188	0	175	0.36	2099	2.06	2518	2.03
34-A	75	81	14.7	0.46	0.71	335	0.76	1590	1.38	1858	1.02	1921	0.97

RUN NO.	T/C 5 MAX. (°F)	T/C 5 TIME TO MAX. (SEC)	P3 MAX. (PSI)	P3 TIME TO MAX. (SEC)	P4 MAX. (PSI)	P4 TIME TO MAX. (SEC)	P5 MAX. (PSI)	P5 TIME TO MAX. (SEC)	P6 MAX. (PSI)	P6 TIME TO MAX. (SEC)	P7 MAX. (PSI)	P7 TIME TO MAX. (SEC)	REACTION
1-A	191	0.48	58.53	0.11	56.19	0.11	54.72	0.11	53.75	0.11	53.53	0.11	x
2-A	191	0.65	73.14	0.21	71.80	0.21	67.79	0.21	67.78	0.21	[illegible]	0.21	x
3-A	156	0.61	52.84	0.16	51.55	0.16	48.35	0.16	48.50	0.16	47.40	0.16	x
4-A	110	4.93	46.01	0.23	46.27	0.23	42.89	0.23	42.65	0.23	41.27	0.23	x
5-A													NC
6-A													NC
7-A													NC
8-A													NC
9-A													NC
10-A		0.58	48.15	0.53	47.93	0.53	46.29	0.53	[illegible]	0.53	[illegible]	0.53	x
11-A	200	1.30	45.62	0.41	43.54	0.41	43.15	0.41	42.58	0.41	[illegible]	0.41	x
12-A													NC
13-A													NC
14-A													NC
15-A	181	1.05	47.24	0.49	NO TRACE		44.48	0.49	[illegible]	0.49	[illegible]	0.49	x
16-A	413	2.68	54.67	2.23	49.22	2.23	49.99	2.23	[illegible]	2.23	50.67	2.23	x
17-A													NC
18-A													NC
19-A													NC
20-A													NC
21-A	206	2.41	55.75	1.22	50.50	1.22	52.84	1.22	52.75	1.22	51.50	1.22	x
22-A	244	0.64	83.45	0.38	75.74		[illegible]	0.38	77.68	0.38	95.35	0.38	x
23-A													NC
24-A	219	1.13	46.70	0.59	42.08	0.59	[illegible]	0.59	[illegible]	0.59	43.23	0.59	x
25-A	188	0.88	45.62	0.35	40.80	0.35	43.34	0.35	[illegible]	0.35	40.95	0.35	x
26-A	193	0.54	ALL PRESSURES SATURATED										
27-A	523	0.56	83.86	0.27	81.83	0.27	[illegible]	0.27	[illegible]	0.27	73.35	0.27	x
28-A	535	0.81	34.26	0.25	34.93	0.25	30.26	0.25	[illegible]	0.25	30.03	0.25	x
29-A													NC
30-A													NC
31-A	593	1.52	59.77	0.39	60.00	0.39	53.56	0.39	[illegible]	0.39	54.55	0.39	x
32-A													NO
33-A	651	1.99	42.82	0.31	43.29	0.31	38.15	0.31	[illegible]	0.31	[illegible]	0.31	x
34-A	535	0.81	39.25	0.29	39.11	0.29	[illegible]	0.29	35.65	0.29	[illegible]	0.29	x

TABLE I

x - Fire
NC - No Fire

RUN NO.	GLYCOL %	GLYCOL INJ. TEMP. (°F)	O_2 PRESS. (PSI)	GLYCOL FLOW (GPM)	OX. FLOW (#/MIN)	T/C 1 MAX. (°F)	T/C 1 TIME TO MAX. (SEC)	T/C 2 MAX. (°F)	T/C 2 TIME TO MAX. (SEC)	T/C 3 MAX. (°F)	T/C 3 TIME TO MAX. (SEC)	T/C 4 MAX. (°F)	T/C 4 TIME TO MAX. (SEC)	T/C 5 MAX. (°F)	T/C 5 TIME TO MAX. (SEC)	P3 MAX. (PSI)	P3 TIME TO MAX. (SEC)	P4 MAX. (PSI)	P4 TIME TO MAX. (SEC)	P5 MAX. (PSI)	P5 TIME TO MAX. (SEC)	P6 MAX. (PSI)	P6 TIME TO MAX. (SEC)	P7 MAX. (PSI)	P7 TIME TO MAX. (SEC)	REACTION
1-B	62.5	378	14.6	0.58	0.71	918	10.57	868	10.88	1358	10.64	1529	10.68	1401	10.74	48.34	10.30	45.65	10.30	38.78	10.30	44.23	10.30	43.81	10.30	x
2-B	70.0	232	14.5	0.48	0.71	238	7.73	1427	6.21	1338	6.18	1529	6.10	1283	6.17	37.61	5.30	34.38	5.30	29.94	5.30	33.88	5.30	32.62	5.30	x
3-B	70.0	131	14.7	0.50	0.71	NO REACTION																				NO
4-B	62.5	234	14.8	0.50	0.71	123	1.82	315	1.85	269	1.86	332	1.95	178	1.96	33.98	1.82	31.86	1.82	27.56	1.82	31.25	1.82	30.54	1.82	x
5-B	70.0	146	14.7	0.48	0.71	194	12.51	1433	13.56	1521	13.24	1399	13.26	1276	13.05	62.50	12.48	54.20	12.48	46.10	12.48	52.89	12.48	52.35	12.48	[illegible]
6-B	62.5	190	14.8	0.57	0.71	252	8.20	1347	10.08	1419	9.44	1586	9.13	1454	9.16	55.22	8.17	47.98	8.17	40.82	8.17	46.68	8.17	46.09	8.17	x
7-B	62.5	117	14.7	0.52	0.71	NO REACTION																				NO
8-B	62.5	172	14.6	0.52	0.71	NO REACTION																				NO
9-B	62.5	194	15.8	0.50	0.71	NO REACTION																				NO
10-B	62.5	193	14.4	0.50	0.71	NO REACTION																				NO
11-B	62.5	204	14.7	0.50	0.71	NO REACTION																				NO
12-B	62.5	194	20.9	0.25	0.71	NO REACTION																				NO
13-B	62.5	199	16.5	0.52	0.71	NO REACTION																				NO
14-B	62.5	193	14.7	0.50	0.71	NO REACTION																				NO
15-B	62.5	260	15.6	0.50	0.71	NO REACTION																				NO
16-B	62.5	134	14.7	0.50	0.71	NO REACTION																				NO
17-B	62.5	145	14.7	0.50	0.71	NO REACTION																				NO
18-B	62.5	154	14.7	0.50	0.71	NO REACTION																				NO
1-C	62.5	-	14.7	0.50	0.71	130	1.35	1243	4.46	1586	3.13	1986	3.08	1040	3.01	47.23	1.32	46.88	1.32	46.08	1.32	46.21	1.32	44.38	1.32	x
2-C	62.5	125	14.7	0.50	0.71	95	3.55	1378	4.12	1607	2.74	1829	3.97	993	2.41	44.87	1.45	44.78	1.45	43.73	1.45	43.63	1.45	42.11	1.45	[illegible]
3-C	62.5	79	15.0	NO FLOW	0.71	BELOW 75	0	BELOW 75	0	1115	4.60	1900	5.43	929	6.51	24.32	2.83	25.44	2.83	22.81	2.83	22.75	2.83	21.59	2.83	NO
4-C	62.5	79	14.7	0.50	0.71	224	3.50	1542	5.03	1739	5.03	2147	4.64	1817	4.55	51.09	2.65	50.87	2.65	47.60	2.65	47.69	2.65	46.42	2.65	x
5-C	62.5	79	14.7	0.50	0.71	224	6.48	1480	6.26	1613	5.83	2053	5.44	1669	5.52	44.30	3.91	45.04	3.91	41.05	3.91	42.46	3.91	40.32	3.91	x
1-D	62.5	80	3.0	0.50	0.61	170	1.05	1236	5.02	1115	2.66	1207	1.75	353	1.81	10.37	0.88	15.64	0.88	11.11	0.88	11.01	0.88	10.51	0.88	x
2-D	62.5	80	20.6	0.50	0.71	224	3.07	1405	5.22	1704	5.12	1680	5.67	444	6.50	50.53	2.57	51.82	2.57	47.40	2.57	47.32	2.57	46.63	2.57	x
3-D	62.5	81	20.0	0.50	0.71	75	2.25	332	2.24	507	2.25	1807	13.20	1634	18.29	47.89	2.23	49.37	2.23	44.62	2.23	[illegible]	2.23	44.15	2.23	x
4-D	62.5	191	3.3	0.50	0.50	163	2.70	1135	3.48	1061	2.31	1627	1.92	1053	1.93	12.07	0.98	15.83	0.98	10.72	0.98	10.64	0.98	10.32	0.98	x
1-E	37.5	AMB.	14.7	0.50	0.71	NO REACTION																				NO
2-E	37.5	AMB.	14.0	0.50	0.71	NO REACTION																				NO

TABLE I (Cont'd)

x – Fire
NO – No fire

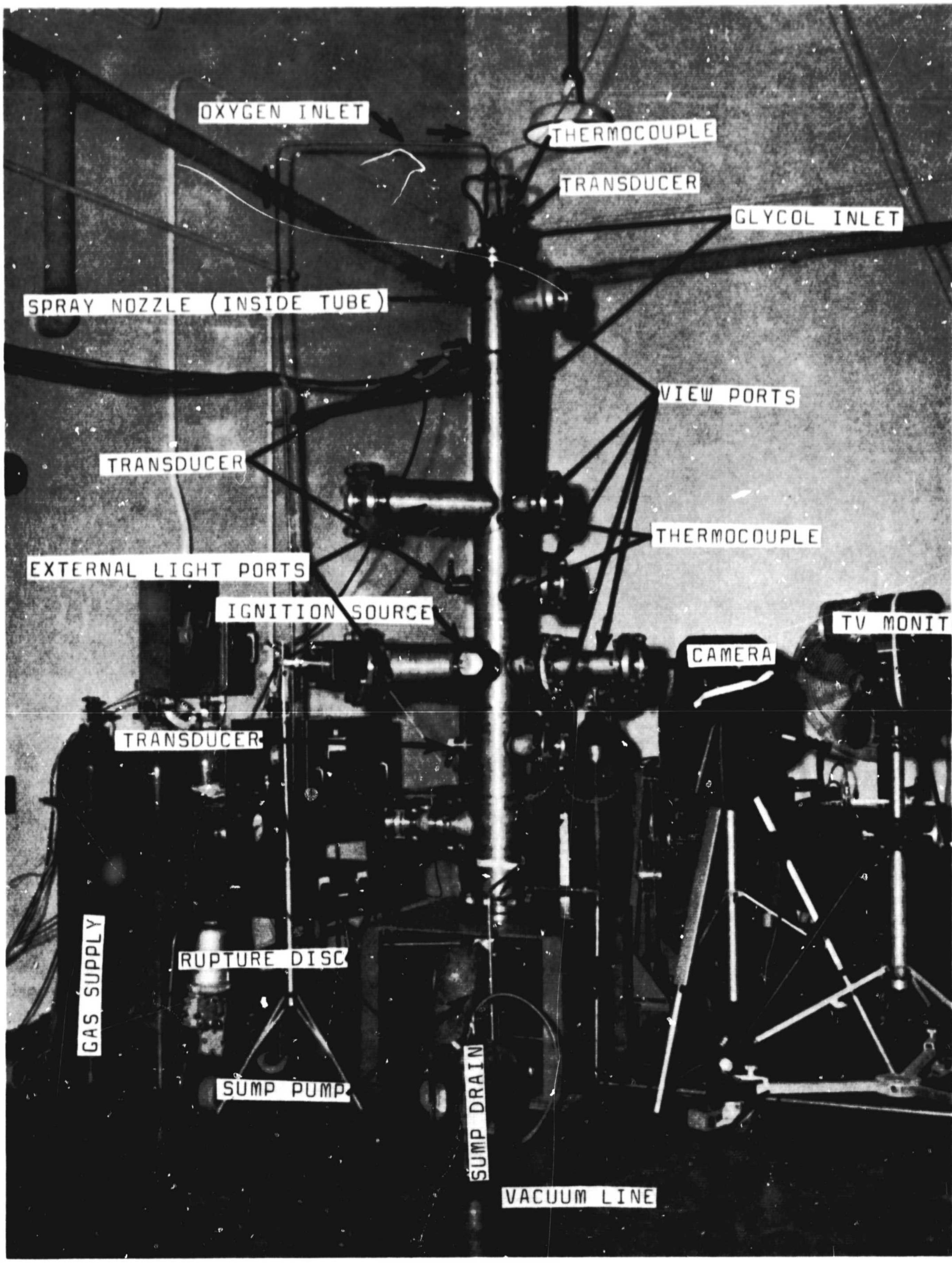

FIGURE 1

FLOW REACTOR SYSTEM

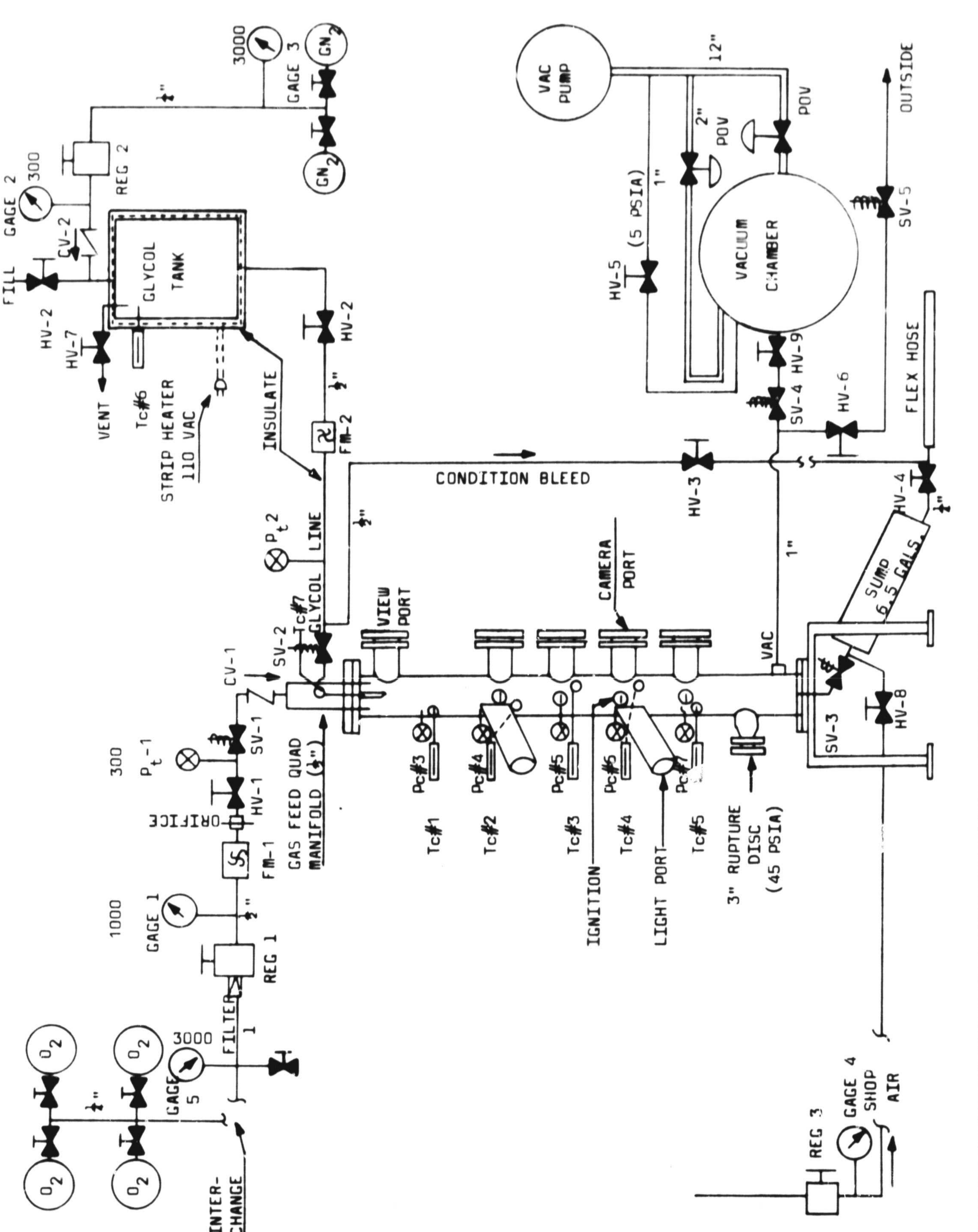

FIGURE 2

FLOW REACTOR SCHEMATIC

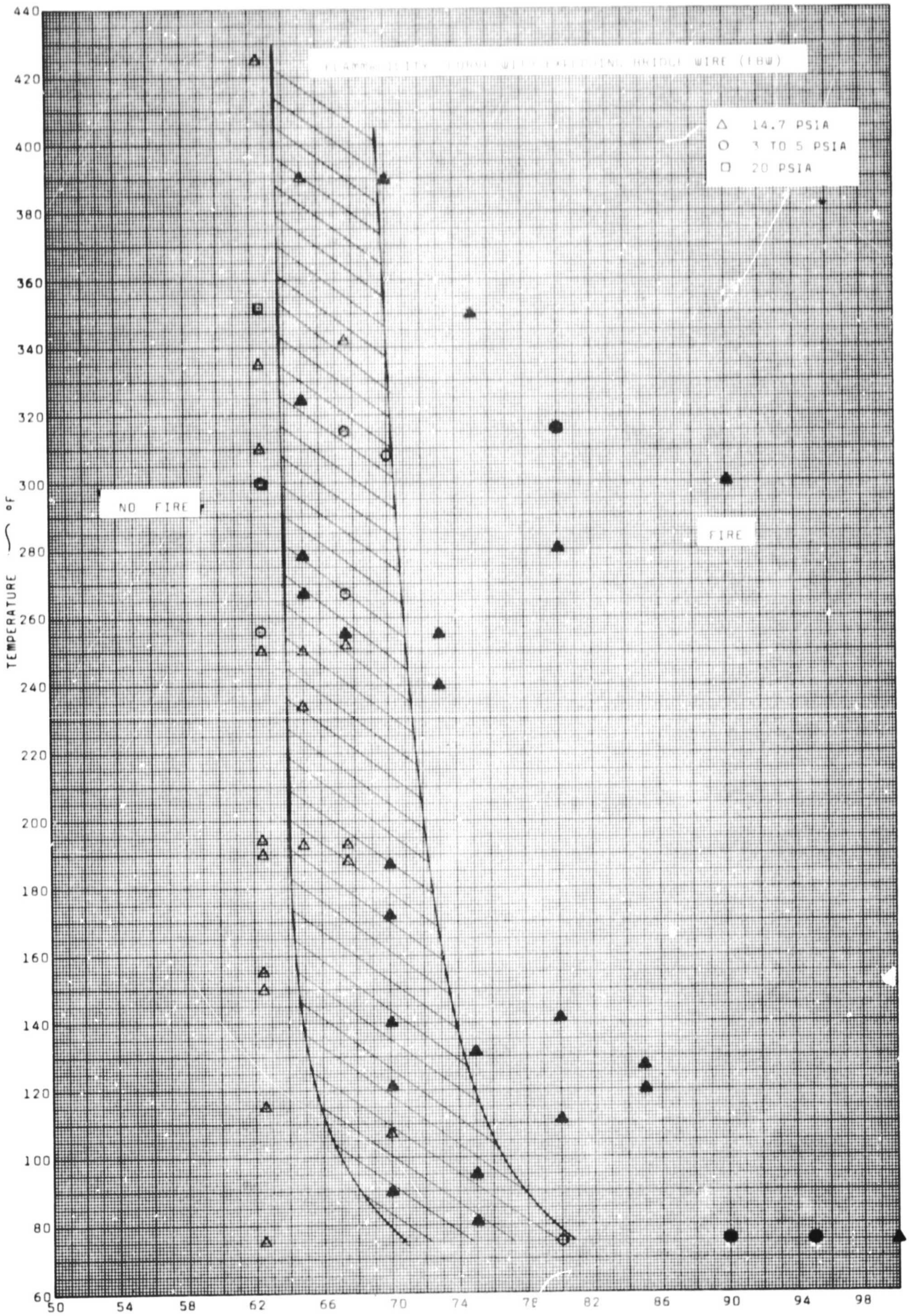

FIGURE 3

FLAMMABILITY VS. PERCENT GLYCOL

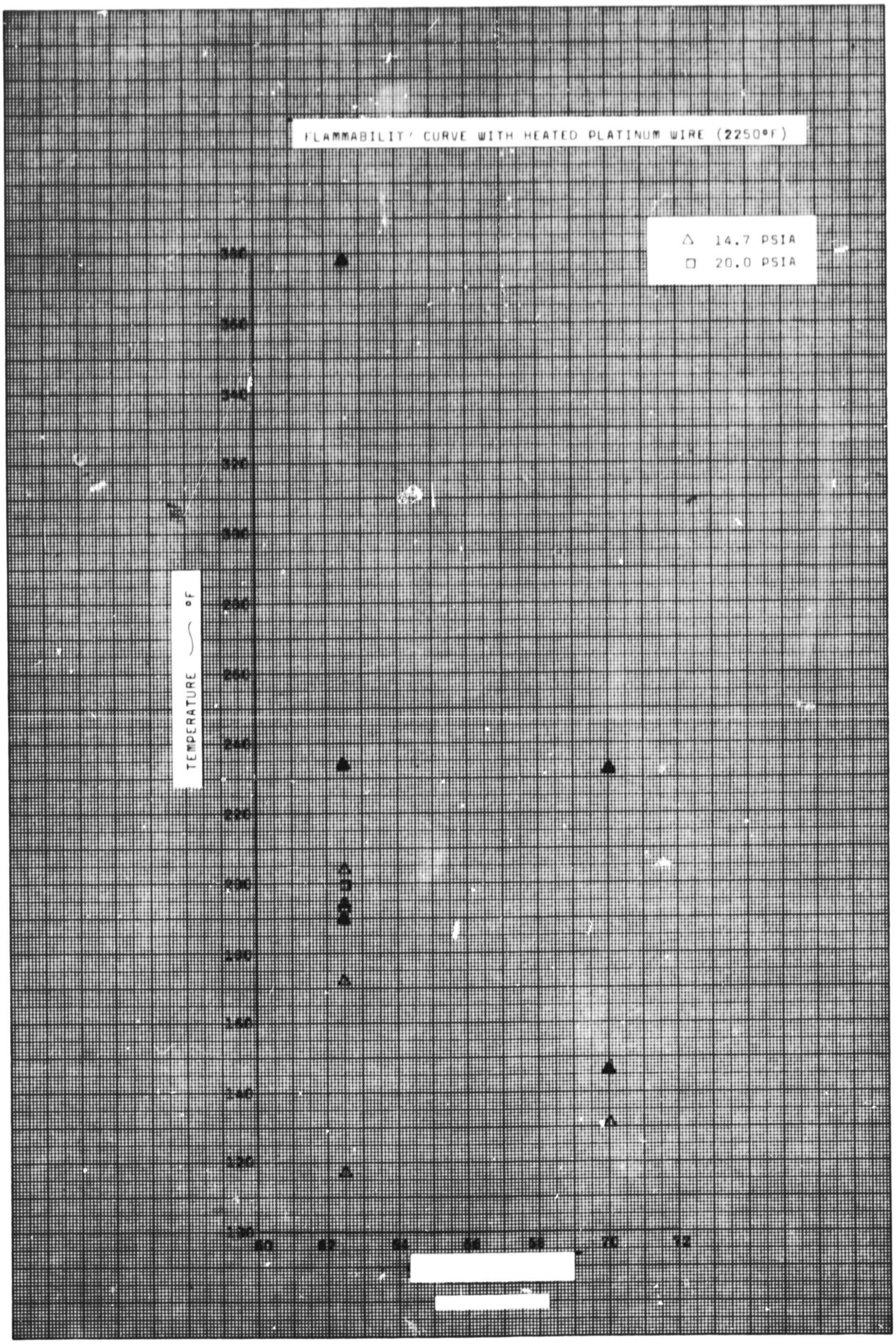

FIGURE 4

FLAMMABILITY VS. PERCENT GLYCOL