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CCL REPORT NO. 231

INTERIM REPORT

WATER EQUILIBRATION STUDIES - BRAKE FLUIDS

BY

EDWARD A. MAINS

DECEMBER 1967

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U. S. ARMY COATING & CHEMICAL LABORATORY
Aberdeen Proving Ground
Maryland

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U. S. ARMY COATING AND CHEMICAL LABORATORY
ABERDEEN PROVING GROUND
MARYLAND

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ABSTRACT

The object of this investigation was to study the water absorption characteristics of various types of hydraulic brake fluids at controlled humidities.

Three series of tests were conducted using a wide scope of brake fluid types exposed to relative humidities ranging from 20% to 80%. Rates and amounts of water absorption were determined. Moisture equilibration percentages were obtained. Boiling point depression was correlated with water concentration.

All fluids tested were found to absorb water to varying degrees over the entire humidity range. Boiling points were drastically reduced with only small amounts of water. Fluids equilibrated at one relative humidity were found to desorb water when exposed to a lower humidity.

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I. INTRODUCTION

The U. S. Army Coating and Chemical Laboratory was authorized by AMC Directive AMC Code 5025.11.802, dated 1 August 1966, to conduct research on hydraulic brake fluids.

One area of investigation concerns the hygroscopic tendencies of all fluids. It was known the brake fluids absorb water at varying rates, and to varying degrees. This water absorption results in depression of the boiling point, often to a temperature well below that actually reached in operational systems. In addition, certain other properties of the fluid are altered (see CCL Report No. 198).

In three series of tests, several fluids were exposed to a range of controlled relative humidities. The water absorption was studied, and the results are the subject of this report.

II. DETAILS OF TEST

A. Tests conducted

1. Hygroscopicity test. Three 200 ml. samples of each fluid tested were placed in 100 x 50 mm. crystallizing dishes. These dishes were placed in desiccators maintained at 20, 50, and 80 percent relative humidities by means of saturated salt solutions. The initial water content was determined by the Karl Fischer method and subsequent samples were analyzed periodically for the duration of the test.

After equilibration of all the fluids, dishes from the 80% relative humidity atmosphere were moved to the 20% desiccators. Water desorption was measured, again by the Karl Fischer method.

2. Water determination. Water content was analyzed by Karl Fischer titration. A small sample (approximately 200 mg.) of brake fluid was introduced into a 50 ml. flask, and an excess of Karl Fischer reagent was added. This was then back titrated with standard water-methanol solution, containing one mg. of water per ml. of solution. This titration was compared to a blank and the water content was calculated from the equation: $\% \text{ water} = A-B/W \times 100$

where A is the volume (ml.) of standard to titrate the blank
B is the volume (ml.) of standard to titrate the sample, and
W is the weight (mg.) of the sample.

3. Boiling point

A 50 ml. sample of the brake fluid was placed in a 100 ml. round bottom flask, fitted with a thermometer and water cooled reflux condenser. The temperature of the fluid after refluxing for five minutes at a rate of one-two drops per second was recorded as the boiling point.

B. Fluids tested

Series 1. Five fluids were tested in this series: a commercial type 70R1 fluid with a modified castor oil base, commercial fluids of types 70R1 and 70R3 with polyoxy-glycol bases, a commercial fluid of proposed type 70R4, and a commercial low hygroscopic fluid. These fluids range in boiling point from 329°F. to 533°F.

These fluids were placed in the desiccators and water absorption was measured over a period of four weeks. At the end of each week, a 50 ml. sample was withdrawn for boiling point determinations. This resulted in a considerable change in volume in the test samples.

Series 2. Two fluids were included in this series; a second commercial low hygroscopic fluid, and a fluid meeting Composition 1 of MIL-P-46046. In order to minimize upsetting the only small 0.5 ml. samples were drawn once every 7 days for water determinations. After six weeks, the samples exposed to 80% relative humidity were moved to a 20% relative humidity atmosphere. The water content was then checked periodically for the next seventeen days.

Series 3. In series 3, four fluids ranging in boiling point from 330°F. to 420°F. were tested; a commercial polyoxy-glycol base 70R1 fluid, two commercial polyoxy-glycol base 70R3 fluids, and a fluid meeting Composition 3 of MIL-P-46046. Fluis were completely equilibrated in this series. Tests were run at the humidities used in the previous series. During the first thirty-five days, small samples were taken every two-three days to determine the rate of absorption. During the remainder of the test period, the fluids were stirred regularly to bring them to equilibrium, a small amount (0.5 to 1.0%) of water was added. If they were at equilibrium this added water would be lost to the atmosphere. This was continued until all fluids were found to be at equilibrium.

After equilibrium was reached, the samples from the 80% relative humidity desiccators were placed in the 20% atmosphere. Water content was analyzed periodically as in series 2.

Boiling point determinations were run on all equilibrated brake fluid samples.

III. RESULTS OF TESTS

A. Water absorption

All of the fluids tested were found to absorb water when exposed to a moist atmosphere. In series 1 and 2 the tests were not run to a point where it was possible to be sure equilibrium had been reached. The data is useful, however, in determining the relative rates and amounts absorbed by the different fluids (tables I-IX).

In series 3, all the fluids reached equilibrium, with the exception of Composition 3 which formed a sediment (reported in CCL Report No. 222) after 4.5% water was absorbed. The equilibrium amounts of water for the other fluids were 20.6%, 17.0% and 16.2%.

When the water absorption was plotted against time of exposure, a smooth curve was obtained (see graph 1-4). By plotting the same data versus the square root of time, a straight line resulted (graphs 5-8). The slope of this line will be the rate constant and will be dependent upon the physical properties of the fluid and the activity of the water (relative humidity).

After the fluids had reached equilibrium, those from the 80% relative humidity atmosphere when moved to the 20% desiccators. These were found to lose moisture to the atmosphere (table XI). A plot of water content versus time resulted in a smooth curve (graph 10).

B. Boiling points

Even very small amounts of water were found to lower the boiling points of the fluids tested. A comparison of original boiling points and depressed boiling points after equilibration can be found in table X.

The fluids tested cover a wide range of initial boiling points, however, after equilibration the range is narrowed considerably (graph 9). The only exceptions to this are the low hygroscopic brake fluids, which boil slightly higher than the others after equilibration.

IV. DISCUSSION AND RECOMMENDATIONS

From the data presented, it can be seen that absorption alone can account for sufficient water in a brake system to lower the fluid boiling point to a dangerous level. In fact, no fluid tested was found to maintain a boiling point above 300°F. when equilibrated at a relative humidity of 50% or higher.

Possible methods of dealing with this problem are to develop closed brake systems which would not be vented to the atmosphere; or to develop brake fluids which are less hygroscopic, or whose boiling points are less sensitive to water content.

V. REFERENCES

1. Authority: AMC Directive, AMC Code 5025.11.802 dated 24 July 1964.

2. Federal Specification VV-H-910a, Hydraulic Fluid, Non-Petroleum Base, Automotive.

3. Military Specification MIL-P-46046A, Preservative Fluid, Automotive Brake System and Components.

4. Specification 70R1, Society of Automotive Engineers, 485 Lexington Avenue, New York, New York.

5. Specification 70R3, IBID.

6. Proposed Specification 70R4, IBID.

7. CCL Report No. 198, Effect of Water on Hydraulic Brake Fluid, dated May 1966.

8. CCL Report No. 222, Study of Solubility of Ternary Mixtures of Water/3-Methoxybutanol-1/Sodium Tetra Borate-Diol Condensates, dated January 1967.

APPENDIX A

TABLE I

Series 1
Water Absorption at 20% Relative Humidity
% Water

Days Exposed	Commercial Modified Castor Oil Base (70R1)	Commercial Polyoxyglycol Base (70R1)	Commercial Polyoxyglycol Base (70R3)	Commercial Polyoxyglycol Base (Proposed 70R4)	Commercial LHF
0	0.06	0.34	0.11	0.07	0.31
1	0.81	0.88	0.44	0.27	0.40
2	0.97	0.39	0.59	0.46	0.27
3	1.12	1.18	0.67	0.59	0.24
4	1.27	1.18	0.99	0.83	0.54
7	1.50	1.35	1.07	0.85	0.46
14	1.53	1.90	1.76	1.58	0.58
21	1.80	2.57	2.05	1.91	0.36
28	2.01	2.40	2.30	1.96	0.54

TABLE II

Series 1
Water Absorption at 20% Relative Humidity
% Water

Days Exposed	Commercial Modified Castor Oil Base (70R1)	Commercial Polyoxyglycol Base (70R1)	Commercial Polyoxyglycol Base (70R3)	Commercial Polyoxyglycol Base (Proposed 70R4)	Commercial LHF
0	0.06	0.34	0.11	0.07	0.30
1	0.72	0.93	0.19	0.60	0.51
2	1.09	1.28	0.94	1.02	0.65
3	1.29	1.37	1.22	1.18	0.86
4	1.58	2.04	1.59	1.74	1.01
7	1.95	2.41	2.07	1.89	0.98
14	3.15	4.30	3.58	3.65	1.53
21	4.27	5.90	5.32	4.79	1.59
28	4.71	6.92	6.70	5.80	1.80
83	5.15	7.98	7.72	6.97	--
102	5.65	8.11	8.12	7.11	--

TABLE III

Series 1
Water Absorption at 80% Relative Humidity
% Water

Days Exposed	Commercial Modified Castor Oil Base (70R1)	Commercial Polyoxyglycol Base (70R1)	Commercial Polyoxyglycol Base (70R3)	Commercial Polyoxyglycol Base (Proposed 70R4)	Commercial LHF
0	0.06	0.34	0.11	0.07	0.23
1	0.97	0.88	0.86	0.75	--
2	1.40	1.46	1.49	1.13	--
3	1.96	2.04	2.24	1.64	--
4	2.42	2.65	3.06	2.15	--
7	3.38	4.12	3.71	3.49	2.37
14	5.64	8.46	7.00	5.30	3.36
21	6.98	9.13	8.52	6.75	3.80
28	9.02	14.21	11.10	9.77	3.93
42	--	--	--	--	--
49	13.20	16.90	14.50	12.82	--
83	--	18.16	--	--	--
102	12.92	20.94	--	14.91	--

TABLE IV

Series 1
Water Absorption at 20% Relative Humidity
% Water

Days Exposed	Composition 1 MIL-P-46046	Commercial LHF
0	0.05	0.05
7	0.59	0.56
14	1.37	0.82
21	1.82	1.01
28	--	0.88
35	1.87	--
42	1.68	0.39

TABLE V

Series 2
Water Absorption at 50% Relative Humidity
% Water

Days Exposed	Composition 1 MIL-P-46046	Commercial LHF
0	0.05	0.05
7	1.72	0.94
14	2.47	1.02
21	2.69	0.99
28	3.48	1.79
35	3.42	1.60
42	3.26	1.40

TABLE VI

Series 1
Water Absorption at 80% Relative Humidity
% Water

Days Exposed	Composition 1 MIL-P-46046	Commercial LHF
0	0.05	0.05
7	2.88	1.66
14	4.43	1.95
21	4.94	2.66
28	6.08	3.35
35	5.45	3.35
42	5.28	3.21

TABLE VII

Series 3
Water Absorption at 20% Relative Humidity
% Water

Days Exposure	$(Days)^{1/2}$	Commercial Polyoxyglycol Base (70R1)	Commercial Polyoxyglycol Base (70R3)	Commercial Polyoxyglycol Base (70R3)	Composition 3 MIL-P-46046
0	0	0.91	0.52	0.29	0.45
1	1.00	1.20	0.99	0.60	0.72
2	1.41	1.64	1.16	1.12	0.64
3	1.73	1.73	1.51	1.30	0.86
9	3.00	1.91	1.54	1.08	0.90
10	3.16	2.06	1.51	1.38	0.57
13	3.61	2.14	1.59	1.42	1.11
16	4.00	1.96	1.47	0.80	--
22	4.69	2.36	1.85	1.74	1.02
28	5.29	2.50	1.83	0.96	0.92
31	5.66	1.99	1.80	1.49	0.94
37	6.08	2.24	1.43	1.55	1.02
42	6.48	1.86	1.68	1.47	1.17
48	6.93	2.41	2.18	1.89	1.48
76	8.83	2.86	2.52	2.09	1.33
87	9.33	2.75	2.36	1.99	1.30
113	10.63	2.59	2.45	1.97	1.41
Equilibrium		2.8	2.4	2.1	1.40

TABLE VIII

Series 3
Water Absorption at 50% Relative Humidity
% Water

Days Exposure	$(Days)^{1/2}$	Commercial Polyoxyglycol Base (70R1)	Commercial Polyoxyglycol Base (70R2)	Commercial Polyoxyglycol Base (70R3)	Composition 3 MIL-P-46046
0	0	0.91	0.52	0.29	0.45
1	1.00	1.34	0.83	0.98	0.94
2	1.41	1.90	1.54	1.06	0.99
3	1.73	2.38	1.78	1.58	1.42
9	3.00	3.52	3.05	2.73	2.32
10	3.16	3.94	3.01	2.63	2.15
13	3.61	4.45	3.73	3.26	2.26
16	4.00	4.61	3.99	3.29	2.21
22	4.69	5.54	4.80	4.31	3.08
28	5.29	5.34	4.52	3.84	3.08
31	5.66	6.10	5.34	4.64	3.44
37	6.08	6.37	5.48	4.84	3.44
42	6.48	6.30	5.48	4.81	3.16
48	6.93	6.98	5.98	5.46	3.75
76	8.83	8.99	7.74	6.79	4.56
87	9.33	--	--	--	--
113	10.63	8.62	7.65	6.81	--
Equilibrium		8.9	7.6	7.1	4.2

TABLE IX

Series 3
Water Absorption at 80% Relative Humidity
% Water

Days Exposure	$(Days)^{1/2}$	Commercial Polyoxyglycol Base (70R1)	Commercial Polyoxyglycol Base (70R3)	Commercial Polyoxyglycol Base (70R3)	Composition 3 MIL-P-46046
0	0	0.91	0.52	0.29	0.45
1	1.00	1.70	1.44	0.78	1.24
2	1.41	2.82	1.70	1.52	1.52
3	1.73	3.04	2.31	1.80	1.71
9	3.00	6.88	5.98	4.12	3.96
10	3.16	7.15	5.55	4.36	3.88
13	3.61	8.81	7.15	5.34	4.55*
16	4.00	9.30	7.59	6.40	--
22	4.69	12.10	9.18	9.17	--
28	5.29	11.91	9.62	8.58	--
31	5.66	12.86	10.57	10.35	--
37	6.08	13.55	11.29	10.68	--
42	6.48	13.71	10.70	10.22	--
48	6.93	14.98	12.12	11.80	--
76	8.83	19.72	15.71	15.52	--
87	9.33	20.08	16.45	15.63	--
113	10.63	20.62	16.11	16.20	--
Equilibrium		20.6	17.0	16.2	--

*Separation occurs.

TABLE X

Brake Fluid	Boiling Point After Equilibration			
	Initial	20% R.H.	50% R.H.	80% R.H.
Series 1				
Commercial Modified Castor Oil Base (70R1)	329	270	234	221
Commercial Polyoxyglycol Base (70R1)	335	270	239	219
Commercial Polyoxyglycol Base (70R3)	410	298	240	224
Commercial Polyoxyglycol Base (Proposed 70R4)	533	300	236	219
Commercial LHF #1	496	402	295	235
Series 2				
Composition 1 MIL-P-46046	320	270	246	226
Commercial LHF #2	540	353	278	246
Series 3				
Commercial Polyoxyglycol Base (70R1)	331	276	241	226
Commercial Polyoxyglycol Base (70R3)	416	309	246	224
Commercial Polyoxyglycol Base (70R3)	470	308	249	225
Composition 3 MIL-P-46046	345	296	253	--

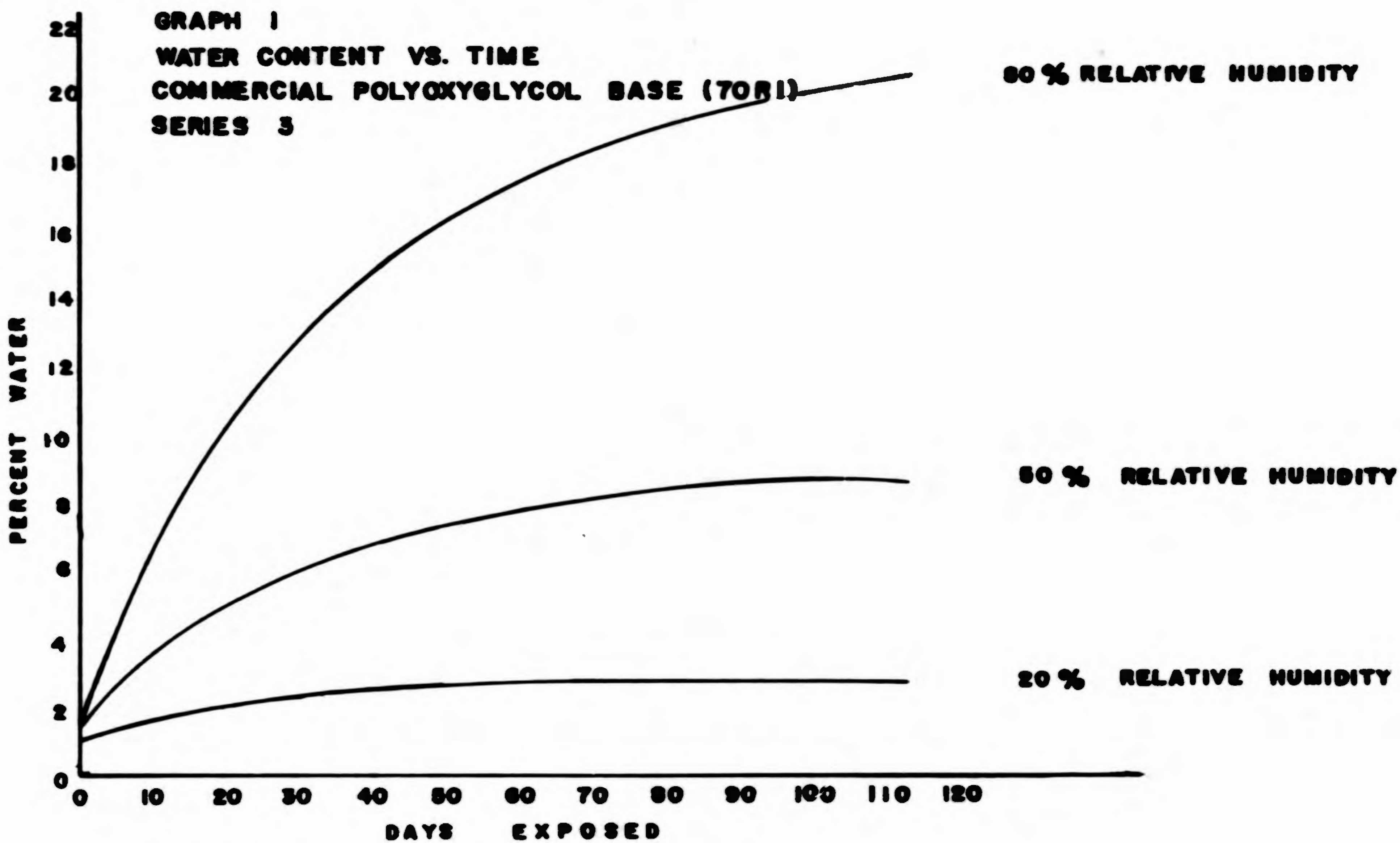
TABLE XI

Water Desorption at 20% Relative Humidity
% Water

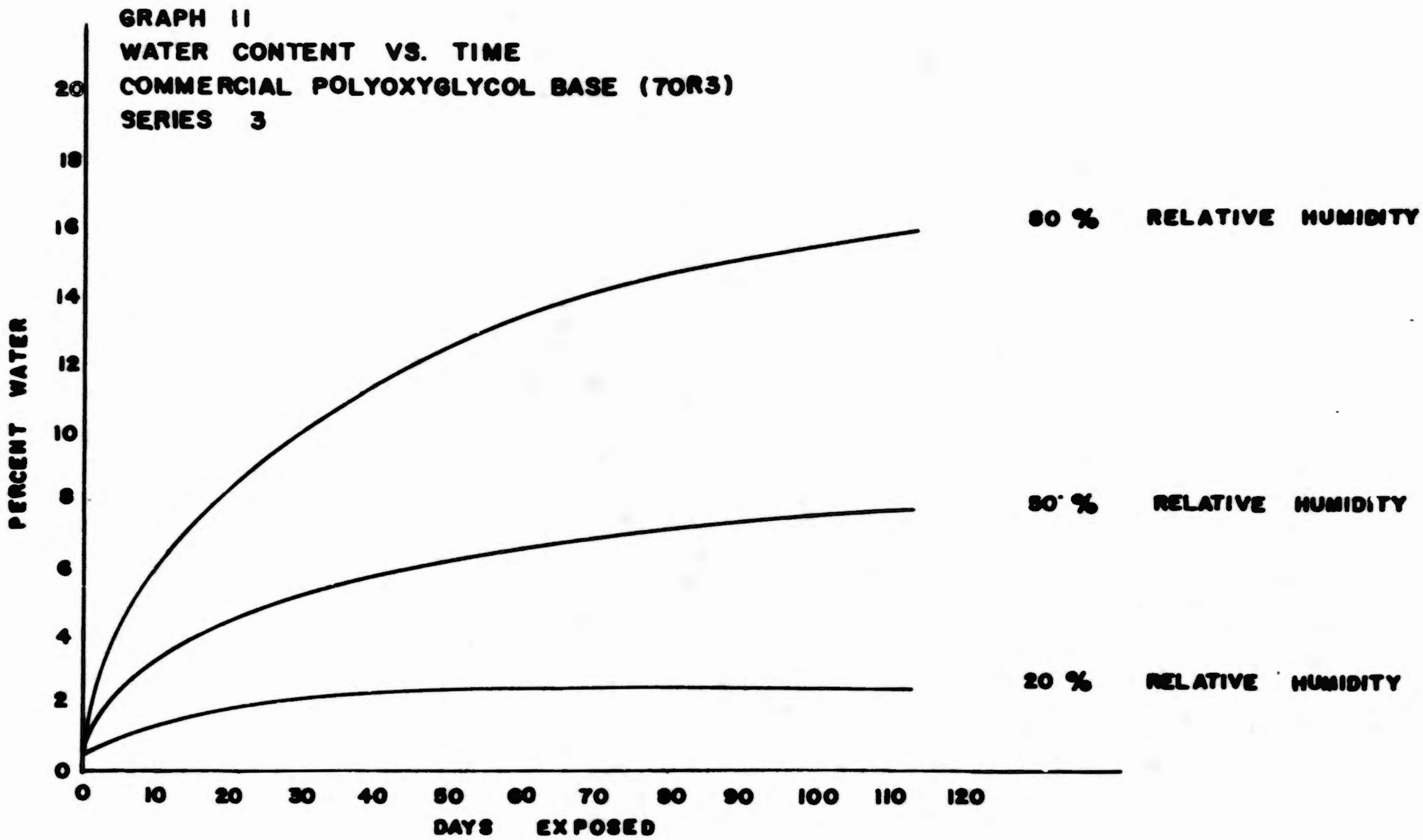
Days Exposed	Commercial Polyoxyglycol Base (70R1)	Composition 1 MIL-P-46046	Commercial LHF	Commercial Polyoxyglycol Base (70R1)	Commercial Polyoxyglycol Base (70R3)	Commercial Polyoxyglycol Base (70R3)
0	14.5*	5.3*	3.2*	20.6*	17.0*	16.1*
1	14.4	6.8	3.1	--	--	--
2	13.9	6.8	2.8	--	--	--
3	11.0	4.8	1.0	--	--	--
7	8.5	3.9	0.7	18.3	14.6	13.4
11	7.7	3.4	--	16.1	12.9	11.5
15	--	--	--	14.4	12.0	10.7
17	5.4	3.1	--	12.6	9.6	9.0
21	--	--	--	9.6	7.6	7.2
25	--	--	--	9.7	7.5	6.5
38	--	--	--	9.3	7.3	7.0
49	--	--	--	8.6	7.2	6.6

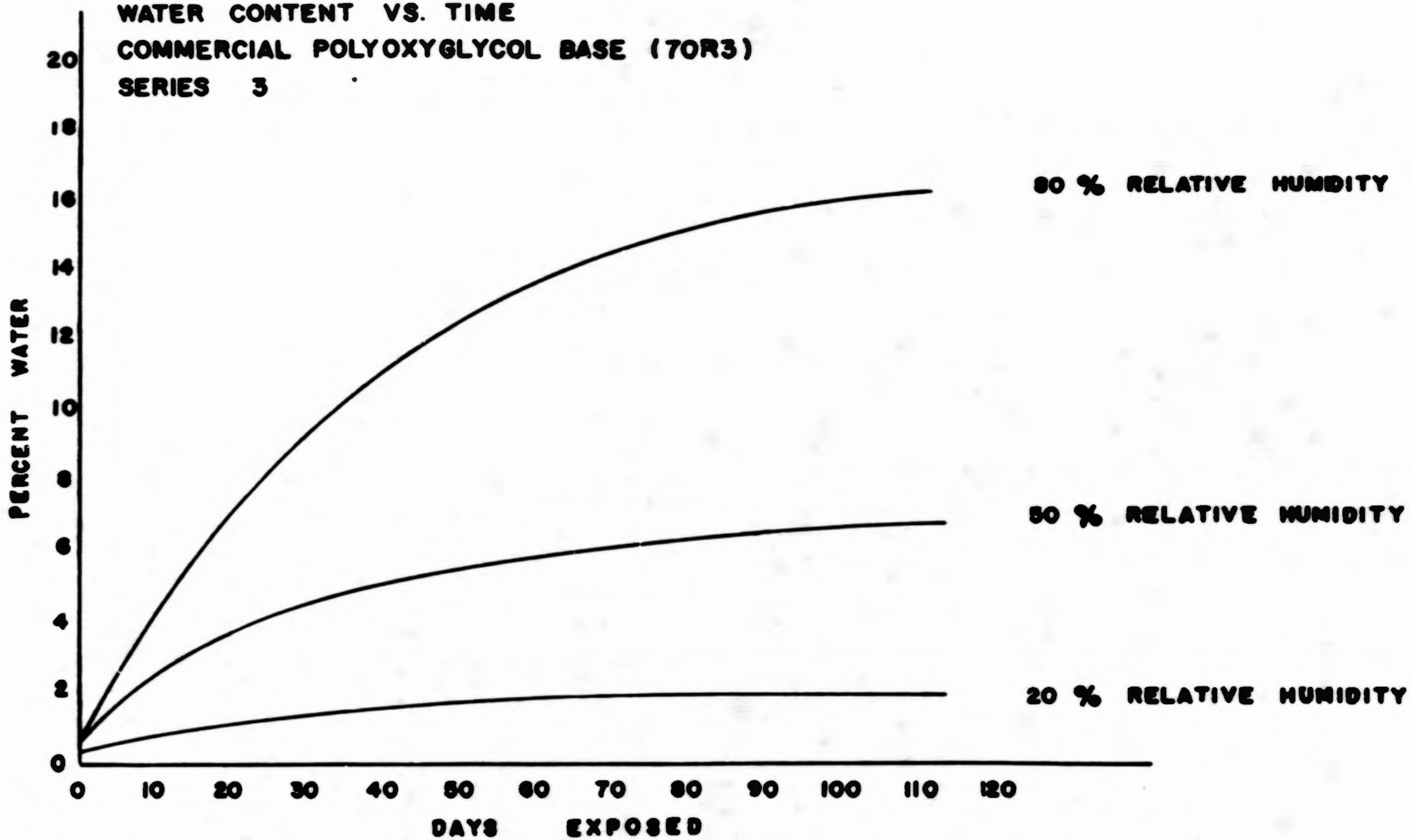
*Percent water in fluids at time of equilibration at 80% relative humidity.

APPENDIX B

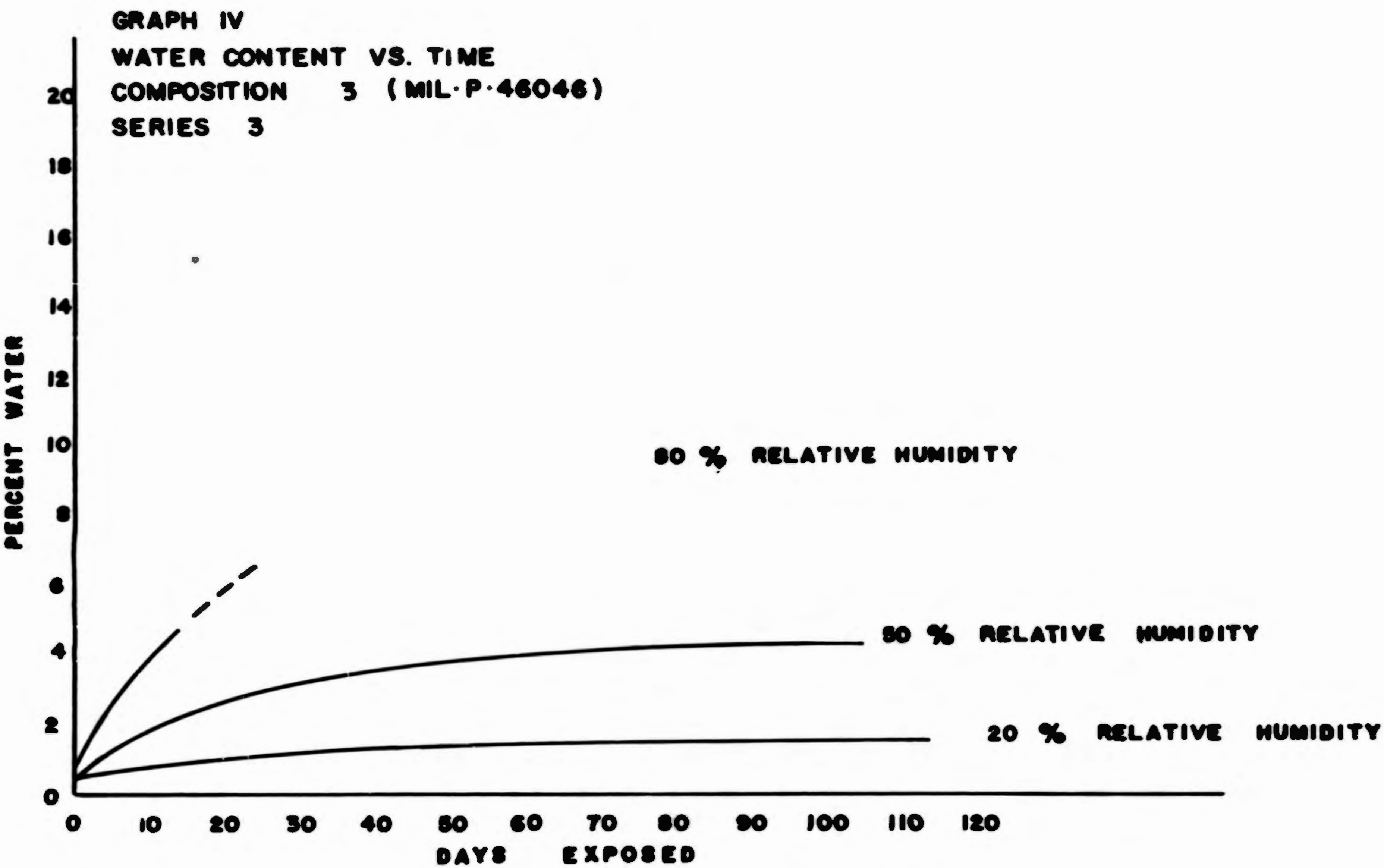


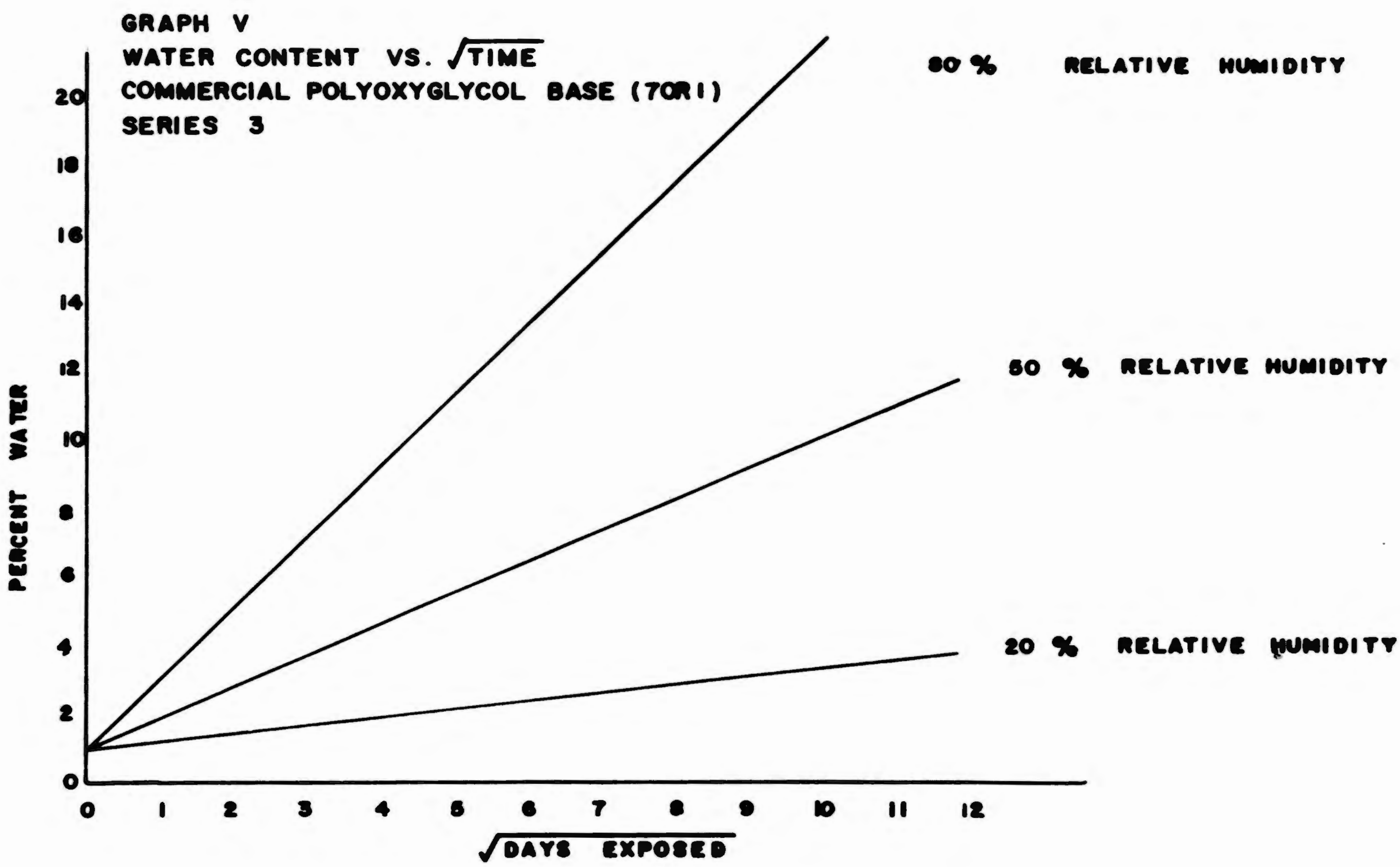
GRAPH 1
WATER CONTENT VS. TIME
COMMERCIAL POLYOXYGLYCOL BASE (70RI)
SERIES 3
80 % RELATIVE HUMIDITY
50 % RELATIVE HUMIDITY
20 % RELATIVE HUMIDITY
PERCENT WATER
0
2
4
6
8
10
12
14
16
18
20
22
0
10
20
30
40
50
60
70
80
90
100
110
120
DAYS EXPOSED

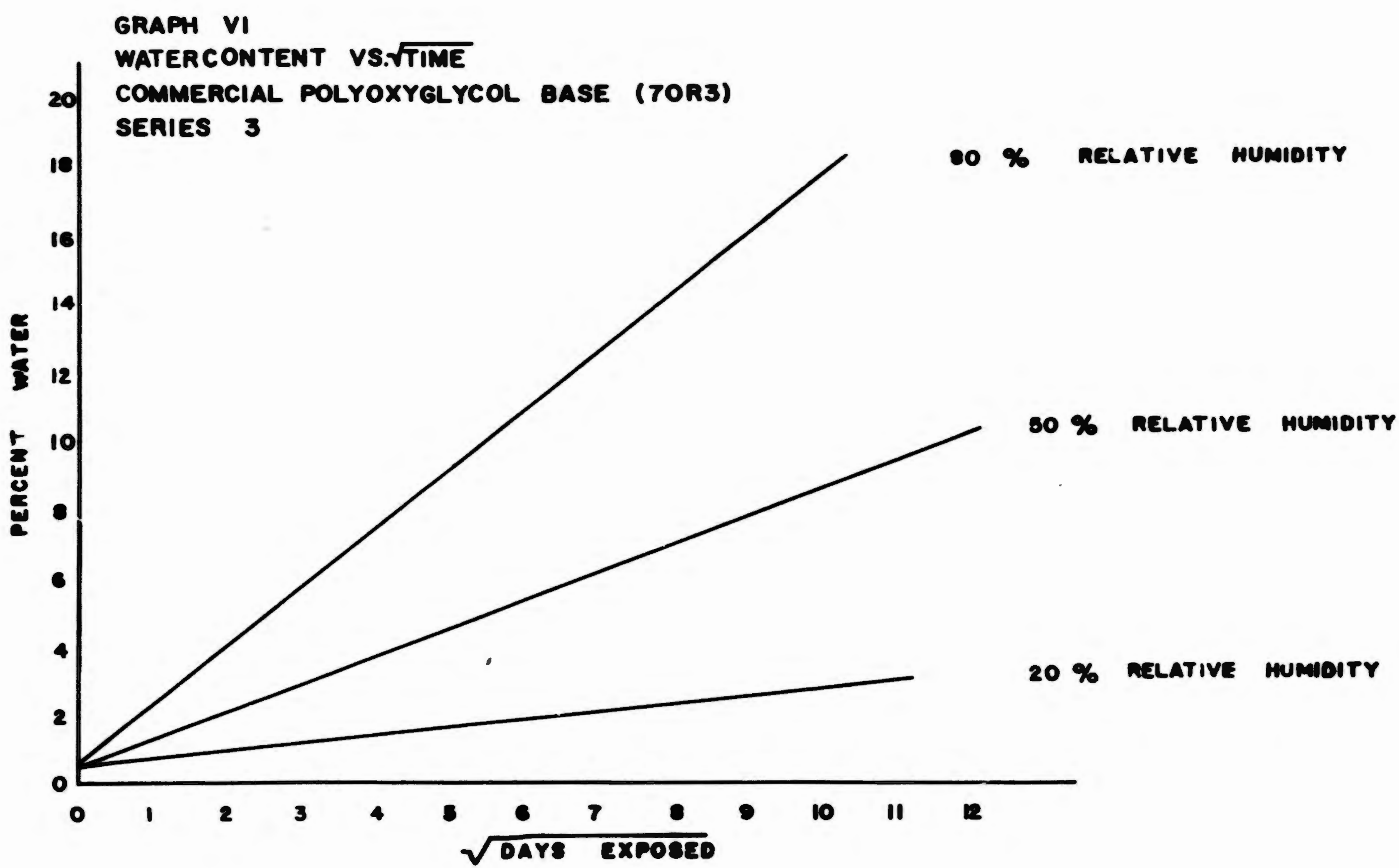

GRAPH 11
WATER CONTENT VS. TIME
COMMERCIAL POLYOXYGLYCOL BASE (70R3)
SERIES 3
PERCENT WATER
0
2
4
6
8
10
12
14
16
18
20
0
10
20
30
40
50
60
70
80
90
100
110
120
DAYS EXPOSED
80 % RELATIVE HUMIDITY
50 % RELATIVE HUMIDITY
20 % RELATIVE HUMIDITY

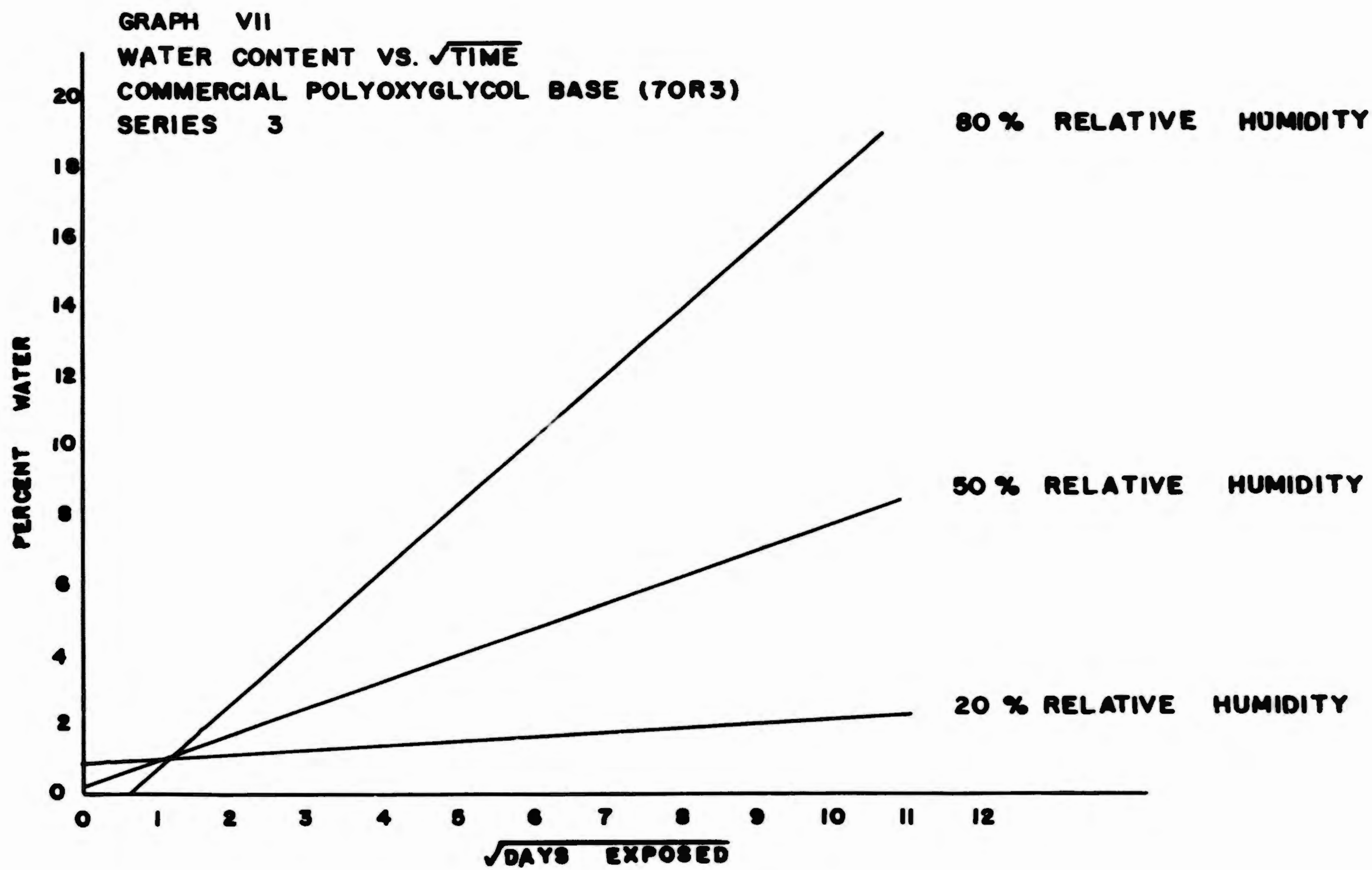


GRAPH III
WATER CONTENT VS. TIME
COMMERCIAL POLYOXYGLYCOL BASE (70R3)
SERIES 3

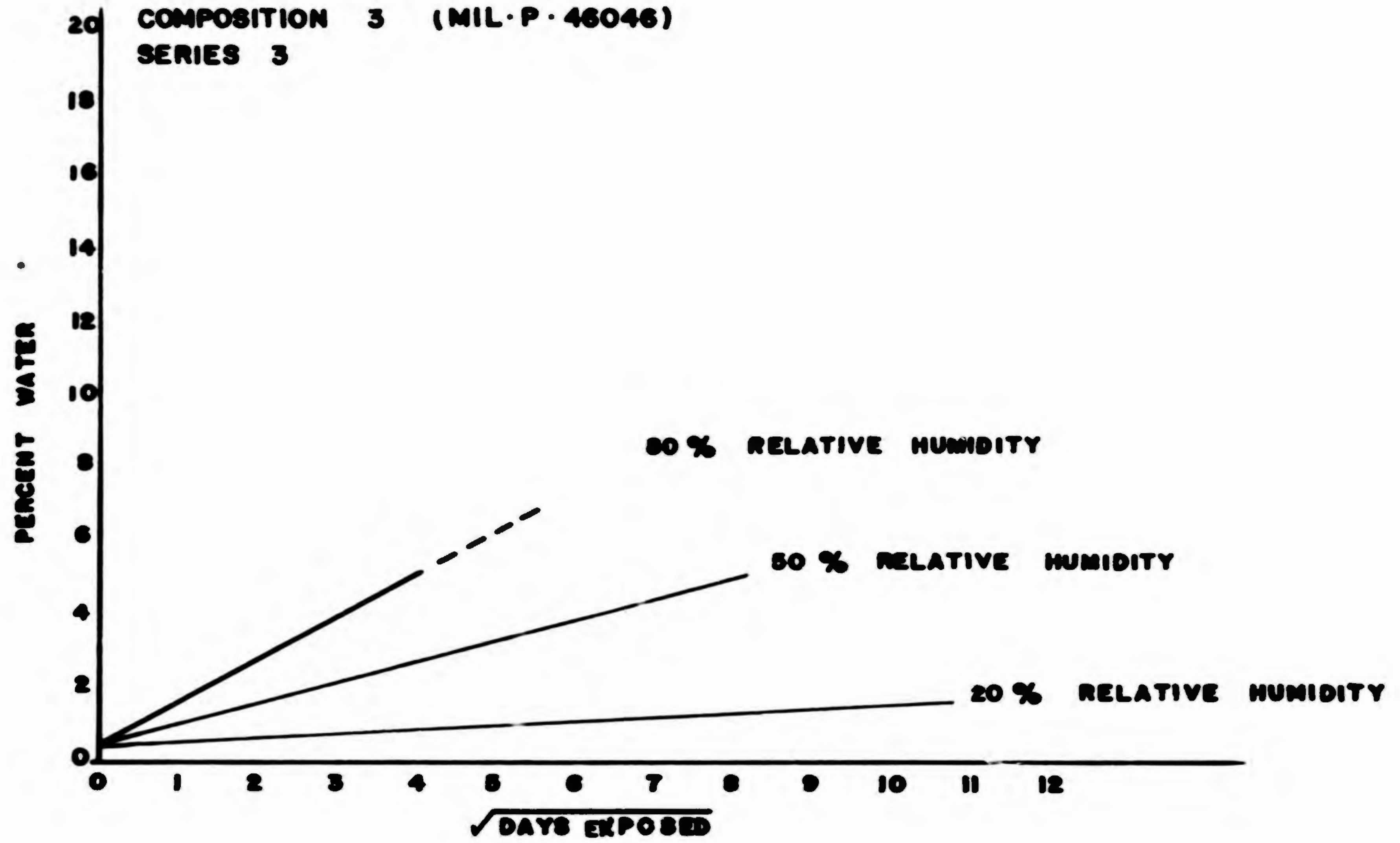

GRAPH IV
WATER CONTENT VS. TIME
COMPOSITION 3 (MIL·P·46046)
SERIES 3
PERCENT WATER
0
2
4
6
8
10
12
14
16
18
20
80 % RELATIVE HUMIDITY
50 % RELATIVE HUMIDITY
20 % RELATIVE HUMIDITY
0 10 20 30 40 50 60 70 80 90 100 110 120
DAYS EXPOSED


GRAPH V
WATER CONTENT VS. √TIME
COMMERCIAL POLYOXYGLYCOL BASE (70R1)
SERIES 3
80 % RELATIVE HUMIDITY
50 % RELATIVE HUMIDITY
20 % RELATIVE HUMIDITY
PERCENT WATER
0
2
4
6
8
10
12
14
16
18
20
0 1 2 3 4 5 6 7 8 9 10 11 12
√DAYS EXPOSED

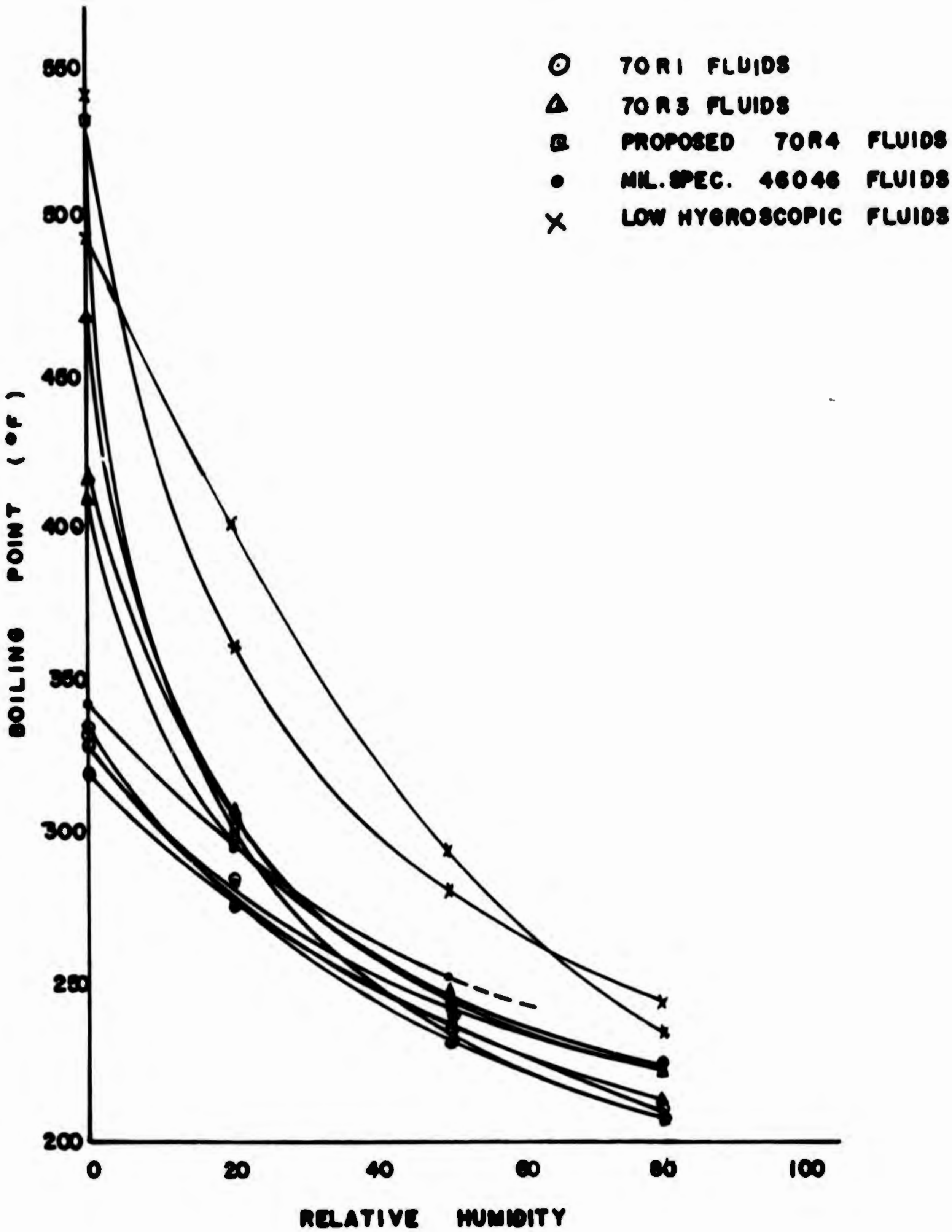

GRAPH VI
WATERCONTENT VS. √TIME
COMMERCIAL POLYOXYGLYCOL BASE (7OR3)
SERIES 3
80 % RELATIVE HUMIDITY
50 % RELATIVE HUMIDITY
20 % RELATIVE HUMIDITY
PERCENT WATER
0
2
4
6
8
10
12
14
16
18
20
0
1
2
3
4
5
6
7
8
9
10
11
12
√DAYS EXPOSED

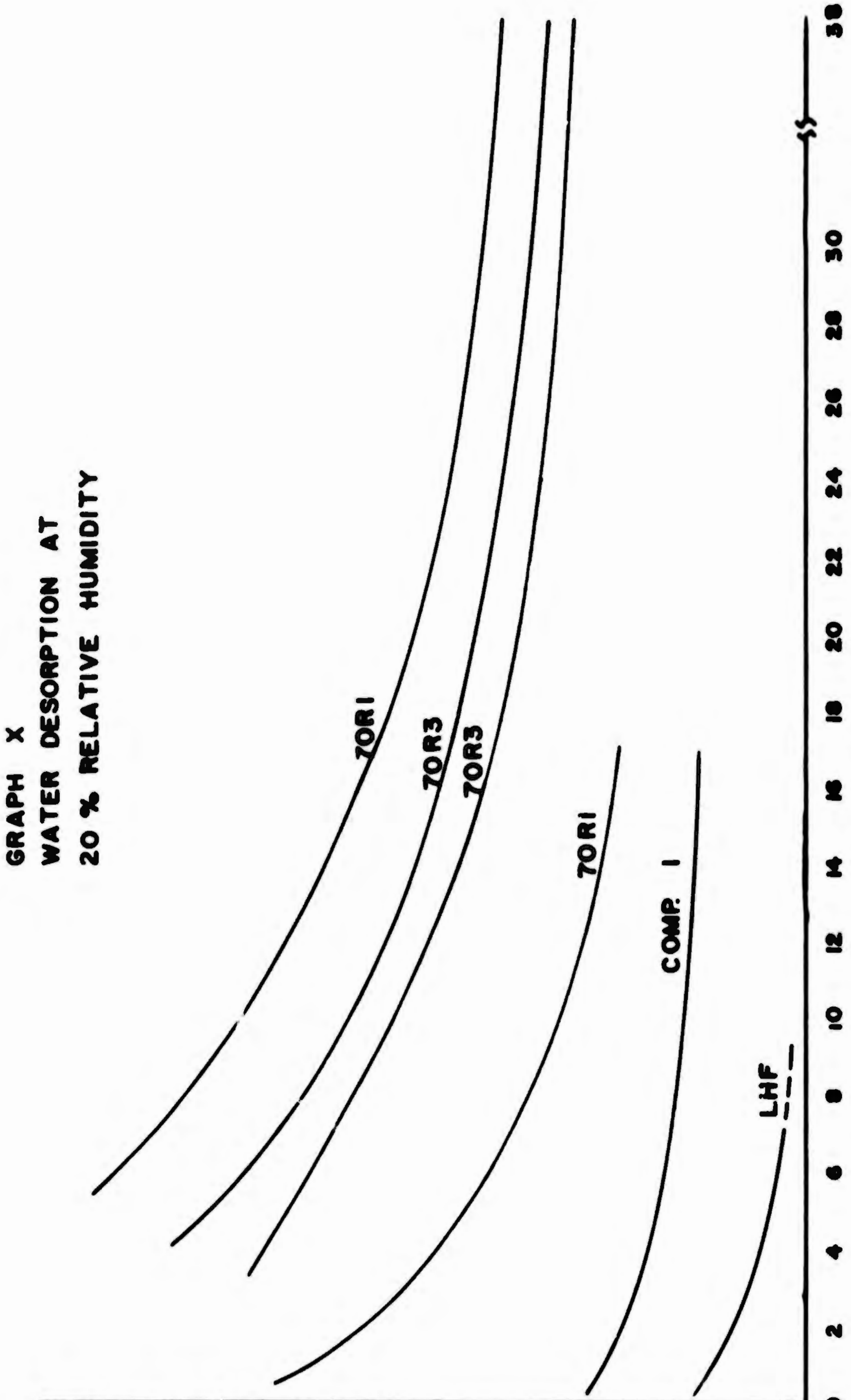

GRAPH VII
WATER CONTENT VS. √TIME
COMMERCIAL POLYOXYGLYCOL BASE (70R3)
SERIES 3
80 % RELATIVE HUMIDITY
50 % RELATIVE HUMIDITY
20 % RELATIVE HUMIDITY
PERCENT WATER
0
2
4
6
8
10
12
14
16
18
20
0
1
2
3
4
5
6
7
8
9
10
11
12
√DAYS EXPOSED

GRAPH VIII
WATER CONTENT VS. $\sqrt{TIME}$
COMPOSITION 3 (MIL·P·46046)
SERIES 3



GRAPH IX
BOILING POINT OF
EQUILIBRATED FLUID
VS. RELATIVE HUMIDITY




GRAPH X
WATER DESORPTION AT
20 % RELATIVE HUMIDITY
70R1
70R3
70R3
70R1
COMP. I
LHF
PERCENT WATER
DAYS EXPOSURE
0
2
4
6
8
10
12
14
16
18
20
22
24
26
28
30
38

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	U. S. Army Materiel Command Washington, D. C. 20310

13. ABSTRACT

The object of this investigation was to study the water absorption characteristics of various types of hydraulic brake fluids at controlled humidities.

Three series of tests were conducted using a wide scope of brake fluid types exposed to relative humidities ranging from 20% to 80%. Rates and amounts of water absorption were determined. Moisture equilibration percentages were obtained. Boiling point depression was correlated with water concentration.

All fluids tested were found to absorb water to varying degrees over the entire humidity range. Boiling points were drastically reduced with only small amounts of water. Fluids equilibrated at one relative humidity were found to desorb water when exposed to a lower humidity.

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14. KEY WORDS	LINK A		LINK B		LINK C	
	ROLE	WT	ROLE	WT	ROLE	WT
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