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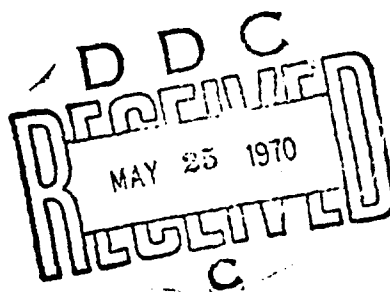
HYGROSCOPICITY
OF
AMMONIUM NITRATE SAMPLES

JOEL HARRIS

APRIL 1970



PICATINNY ARSENAL
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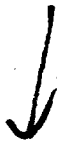
TECHNICAL REPORT 4052

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OF
AMMONIUM NITRATE SAMPLES

JOEL HARRIS

APRIL 1970

AMMUNITION ENGINEERING DIRECTORATE
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ACKNOWLEDGMENT

The author is grateful for the assistance of Gilbert G. Murray of Feltman Research Laboratories who conducted all the static hygroscopicity tests.

SUMMARY

Samples of ammonium nitrate were tested by the AED's Artillery Ammunition Laboratory for hygroscopicity at relative humidity (RH) levels of 60%, 70%, 80%, 90% and 95% and between 28°-30°C. Most of the sample of ammonium nitrate were coated; some were manufactured by a process producing prills; clay material was added in two cases. The results indicated that the ammonium nitrate which contained the least amount of impurities or additives also was the least hygroscopic.

CONCLUSIONS

An increase in the purity of ammonium nitrate causes a decrease in the hygroscopicity.

The addition of insoluble additives did not diminish the hygroscopicity of ammonium nitrate.

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RECOMMENDATIONS

A careful statistically designed test should be conducted in a room where conditions could be accurately monitored and air movements could be controlled to simulate those in a loading plant. Weighings could then be made on perfectly dried ammonium nitrate samples without disturbing the equilibrium conditions. The exact hygroscopicity of each sample simulated to loading plant conditions could then be determined.

Ammonium nitrate of high purity should be considered for use in explosives to minimize hygroscopicity and extend shelf life of explosives.

BACKGROUND

Ammonium nitrate is a component in many explosive system, but a disadvantage in its use is its hygroscopicity. To extend the shelf life of explosives containing ammonium nitrate, it is important to minimize its hygroscopicity.

The types of ammonium nitrate tested consisted of high purity reagent grade, a less pure type which meets government specification, and three different types of phase stabilized prills. The prills consist of small balls of ammonium nitrate coated with a water insoluble material. In addition, two types of insoluble clays were physically added to the specification grade ammonium nitrate and tested. The specification requirements for the ammonium nitrates tested are in Table 4. The purpose of these tests was to determine which of the various types of ammonium nitrate considered for use in explosives would be the least hygroscopic.

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RESULTS

This report deals with the rate of moisture gain and loss of ammonium nitrate samples. Table 1 and 3 contain data from a static test (samples in a controlled humidity dessicator) in which the percent hygroscopicity and percent dehydration are listed. Data of the hygroscopicity of ammonium nitrate from Table 1 are shown in Figures 1-3 for 0-6-1/2 hours and in Figures 4-7 for 0-24 hours. Table 2 contains the percent hygroscopicity data from a dynamic test in a chamber in which the desired humidity was maintained by blowers moving the moisture across the chamber and across the ammonium nitrate samples. This data is graphed and shown in Figures 8-10. Graphs of the hygroscopicity data from the static test listed in Table 3 are shown in Figures 11-15.

The graphs of the dehydration data in Table 1 are shown in Figure 16 and the same data from Table 3 is shown in Figure 17. Table 4 contains the available data on the chemical composition of each ammonium nitrate sample.

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DISCUSSION

Two Tests for Hygroscopicity: Static and Dynamic

Two types of tests were used to determine the hygroscopicity of ammonium nitrate for use as a component in high explosives. Test 1 consisted of small cups filled with ammonium nitrate samples which were placed in dessicators kept at a constant humidity and 30°C. A closed system was thus established with little air movement. This test may be considered a static test depending on the ability of ammonium nitrate to attract water molecules. Test 2 may be considered a dynamic test since a current of steam and air was continuously passed across the samples. The advantage of the dynamic test was that samples could be weighed without opening a dessicator and disturbing the equilibrium conditions. Thus the dynamic test could be useful for determining hygroscopicity for periods from 0-120 minutes which would not be feasible with the static test.

A third test consisted of drying the wet samples for short periods in a 100°C oven. This will be considered a dehydration test or a determination of drying time for ammonium nitrate samples at 100°C. At RH of 60%, 70%, 80%, 90% and 95% and 30°C, six samples of ammonium nitrate were tested for hygroscopicity in small cups placed within dessicators. For the times shown in Table 1 of 2-1/2 to 20 hours, specification grade ammonium nitrate was less hygroscopic than any of the other types. The one exception noted was that from 6-24 hours at 95% RH, Mississippi Ground Prills was the least hygroscopic material. The order of least hygroscopicity overall humidity levels for this static test was (Figures 1-7):

1. Specification Grade
2. Ketona Prills
3. Mississippi Ground Prills
4. Specification Grade with 0.6% Microcell E.
5. Gulf Prills
6. Specification Grade with 1.3% AC-10 Clay.

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The same samples were tested from 0-2 hours in a humidity chamber. The first test was begun at 94% RH which slowly decreased over the two-hour test period to 87%. The temperature was constant at 28°C. Considering only the most hygroscopic of duplicate two-gram samples tested, the following order was established for least hygroscopicity at 87-94% (Table 2 and Figure 10):

1. Specification Grade with 1.3% AC-10 Clay.
2. Specification Grade with 0.6% Microcell E.
3. Mississippi Ground Prills
4. Specification Grade
5. Ketona Prills
6. Gulf Prills

A second dynamic test was conducted at 84% RH which decreased to 76% over a two-hour period at a constant 28°C. The following order was established for the most hygroscopic of duplicate two-gram samples (Table 2 and Figure 8 and 9) in order of least hygroscopicity:

1. Ketona Prills
2. Specification Grade
3. Mississippi Ground Prills
4. Gulf Prills
5. Specification Grade plus 1.3% AC-10 Clay.
6. Specification Grade plus Microcell E.

Before all these samples were obtained, a static test was performed by FRL on Specification Grade, Ketona Prills and Fisher Reagent Grade Ammonium Nitrate. These tests were performed like the above static test but for a longer period. The conditions of the test were RH 60-95%, temperature 30°C, time 163 hours. Fisher Reagent Grade was slightly less hygroscopic than was Specification Grade at 60%, 70%, 90% and 95%. The hygroscopicity of Specification Grade unaccountably increased more than that of the other two as the humidity level increased to 90% and 95% (Table 3, Figures 11-15).

Dehydration of the six samples in the first laboratory test at 100°C showed that Gulf Oil Prills dehydrated fastest and Ketona Prills the slowest (Table 1 and Figure 17). The following order was established for quickest dehydration:

1. Gulf Prills
2. Specification Grade with 1.3% AC-10 Clay.
3. Specification Grade
4. Specification Grade with 0.6% Microcell E.
5. Mississippi Ground Prills
6. Ketona High Density Prills

Dehydration of the samples from the earlier laboratory test showed that Fisher Reagent Grade ammonium nitrate dehydrated much quicker than did Specification Grade and Ketona Prills (Figure 17 and Table 3). All the data indicated that wet ammonium nitrate dehydrates in 2-4 hours at 100°C depending on the type and moisture content.

Fisher Reagent Grade ammonium nitrate is slightly less hygroscopic than other ammonium nitrates in the static test. Specification Grade was second best. It appears that the ability to attract moisture may be proportional to the impurities in ammonium nitrate. Fisher Reagent Grade also dehydrated faster than the other in one test and Gulf Oil Low Density Prills did in the other. The Fisher type was not tested in the dynamic test. The results for the dynamic test for hygroscopicity appear similar to those for the static test at an average hygroscopicity of 80%. However, at an average hygroscopicity of 90%, the Specification Grade with clays added absorbed less moisture over two hours than did all the others in the dynamic test. Mississippi Ground Prills also came close to the clays, but the other three AN types were decidedly more hygroscopic (Figure 16).

The dynamic test may differ from the static test via the mechanism of absorption. The static test depends on the ability of the ammonium nitrate to attract water molecules. The dynamic test depends on the amount of moisture which the surface of the ammonium nitrate can absorb, as the moisture-laden air is continuously passed over the surface of the chemical.

Experimental Procedure

Procedures followed in the dynamic test for 0-2 hours were:

Two-gram samples were weighed in jars which were 2 inches wide and 2 inches high. They were dried for two days at 100°C, cooled in a dessicator, weighed, and tightly closed; placed inside a chamber where temperature and humidity were controlled; opened, weighed and exposed to conditions in the chambers; weighed every 15-20 minutes thereafter inside this chamber.

Procedures followed in the static test are generally similar to those in Reference 1. 5-gram samples were weighed in cups 2-1/2 inches wide and 1/2 inches high. They were placed in dessicators in which chemical solutions controlled the RH. These samples were covered and removed from the dessicators at fixed periods of at least two hours, weighed, and returned to the dessicators.

The relative hygroscopicity was determined by dividing the weight increase by the original weight of ammonium nitrate sample.

$$\% \text{ Hygroscopicity} = \frac{\text{weight of water gained} \times 100}{\text{dry weight of ammonium nitrate sample}}$$

REFERENCE

Military Standard 650, Explosive Sampling, Inspection, and Testing,
U.S. Government Printing Office, Washington, 3 August 1962.

APPENDICES

APPENDIX A

Tables

TABLE 1

STATIC TEST I

% Hygroscopicity at 30°C &
% Dehydration at 100°C of Ammonium Nitrate
Samples

Relative Humidity %		<u>60%</u>	<u>70%</u>	<u>80%</u>	<u>90%</u>	<u>95%</u>
Exposure Time Hours						
A. Specification						
Grade with 0.6% Microcell E (Calcium Silicate)						
1. % Hygroscopicity	2½	0.39	0.46	0.64	0.81	0.79
	4½	0.51	0.64	0.99	1.52	1.58
	6½	0.60	0.78	1.22	2.08	2.31
	24	2.02	3.12	4.47	9.32	12.07
2. % Dehydration	½	0.26	0.40	0.66	2.52	3.46
	1	0.10	0.13	0.28	1.35	2.41
B. Mississippi Ground Prills						
1. % Hygroscopicity	2½	0.10	0.17	0.33	0.67	0.69
	4½	0.17	0.28	0.65	1.41	1.60
	6½	0.22	0.39	0.84	2.08	2.44
	24	1.58	2.83	1.13	10.65	10.34
2. % Dehydration	½	0.34	0.50	0.61	2.54	2.10
	1	-0.70	0.21	0.26	1.84	1.11
C. Specification Grade with 0.6% Clay (Aluminum Silicate)						
1. % Hygroscopicity	2½	0.30	0.21	0.42	0.79	0.86
	4½	0.49	0.30	0.79	1.61	2.02
	6½	0.61	0.37	1.83	2.51	3.13
	24	2.01	3.28	5.31	13.84	17.02
2. % Dehydration	½	0.19	0.43	0.49	0.58	0.32
	1	0.11	0.19	-0.20	0.12	0.01

Relative Humidity %		<u>60%</u>	<u>70%</u>	<u>80%</u>	<u>90%</u>	<u>95%</u>
Exposure						
<u>Time Hours</u>						
D. Gulf Low	2½	0.11	0.27	0.38	0.68	0.82
Density Prills	4½	0.18	0.29	0.63	1.42	1.86
1. % Hygroscopicity	6½	0.14	0.36	0.84	2.23	2.97
	24	0.87	3.83	5.13	12.03	16.31
2. % Dehydration	½	.11	0.84	0.63	0.81	0.79
	1	0.06	0.43	0.23	0.20	0.17
E. Specification Grade						
1. % Hygroscopicity	2½	0.06	0.08	0.22	0.55	0.68
	4½	0.07	0.10	0.41	1.14	1.49
	6½	0.06	0.14	0.57	1.74	2.35
	24	0.52	2.43	3.91	9.06	13.21
2. % Dehydration	½	0.15	0.44	0.43	1.20	4.07
	1	0.09	0.18	0.18	0.54	1.32
F. Ketona High	2½	0.23	0.27	0.41	0.75	0.82
Density Prills	4½	0.30	0.32	0.63	1.37	1.44
1. % Hygroscopicity	6½	0.29	0.35	0.77	2.01	2.40
	24	1.27	2.74	4.15	10.51	13.02
2. % Dehydration	½	0.11	0.24	0.32	0.97	3.75
	1	0.06	0.00	0.12	0.45	1.92

TABLE 2

DYNAMIC TEST

% Hygroscopicity at 28°C
at Relative Humidity Levels of
76-84% C
87-94%

<u>Ammonium Nitrate Type</u>	<u>Exposure Time Minutes</u>	<u>Sample 1 RH 76-84%</u>	<u>Sample 2 RH 76-84%</u>	<u>Sample 3 RH 87-94%</u>	<u>Sample 4 RH 87-94%</u>
A. Mississippi Ground Prills % Hygroscopicity	15	1.10	1.42	2.14	0.95
	30	1.93	2.64	3.34	1.40
	45	2.93	3.65	4.17	1.93
	60	3.85	4.67	4.75	2.99
	75	4.48	5.53	5.83	3.18
	90	5.17	6.18	6.63	4.36
	105	5.72	6.79	7.25	4.94
	120	6.29	7.29	7.93	5.54
B. Specification Grade	15	1.20	1.06	1.16	2.15
	30	2.13	1.96	2.03	3.53
	45	3.14	2.92	3.05	4.81
	60	4.25	3.91	3.46	6.20
	75	5.10	4.68	4.09	6.80
	90	5.72	5.36	4.83	8.22
	105	6.31	5.90	5.29	8.87
	120	6.75	6.41	5.95	9.50
C. Ketona High Density Prills	15	0.98	1.06	1.04	2.00
	30	1.78	1.96	1.51	3.66
	45	2.51	2.92	2.74	4.96
	60	3.26	3.91	3.28	6.35
	75	3.95	4.68	3.83	7.53
	90	4.46	5.36	4.82	8.53
	105	5.01	5.90	5.46	9.28
	120	5.41	6.41	6.42	10.10

<u>Ammonium Nitrate Type</u>	<u>Exposure Time Minutes</u>	<u>Sample 1 RH 76-84%</u>	<u>Sample 2 RH 76-84%</u>	<u>Sample 3 RH 87-94%</u>	<u>Sample 4 RH 87-94%</u>
D. Gulf Oil Low Density Prills	15	0.88	1.05	0.93	2.51
	30	1.83	2.28	1.94	4.75
	45	2.63	3.23	2.75	7.24
	60	3.39	4.30	3.20	8.49
	75	4.13	5.08	3.99	9.76
	90	4.58	5.79	4.69	11.20
	105	5.06	6.38	5.18	13.10
	120	5.48	6.88	5.98	14.70
E. Specification Grade with 1.3% AG-10 Clay	15	1.31	1.18	1.43	1.01
	30	2.47	2.34	3.15	1.96
	45	3.56	3.54	3.97	2.90
	60	4.53	4.67	4.56	3.40
	75	5.20	5.37	5.47	4.45
	90	5.96	6.24	6.07	5.13
	105	6.53	6.91	6.66	5.76
	120	7.12	7.55	7.34	6.49
F. Specification Grade with 0.6% Microcell E	15	1.64	1.23	0.88	1.71
	30	2.62	2.14	1.71	2.76
	45	3.72	3.13	2.71	3.94
	60	4.80	3.97	3.17	4.75
	75	5.48	4.50	4.05	5.44
	90	6.23	5.20	4.58	6.47
	105	6.81	5.69	5.12	7.22
	120	7.48	6.29	5.90	8.21

TABLE 3

STATIC TEST II

% Hygroscopicity at 30°C &
% Dehydration of Ammonium Nitrate at 100°C

		<u>Samples</u>				
Relative Humidity %		<u>60%</u>	<u>70%</u>	<u>80%</u>	<u>90%</u>	<u>95%</u>
Exposure Time Hours						
A. Ketona High						
Density Prills						
1. % Hygroscopicity	2	3.41	2.18	2.98	2.41	2.71
	24	11.91	7.33	12.94	13.99	18.25
	48	12.71	10.81	18.85	23.82	32.17
	72	13.33	14.27	24.86	33.82	46.29
	144	16.12	26.07	45.26	62.85	80.00-
2. % Dehydration	163	16.67	28.78	49.99	68.26	84.44
	1	1.12	1.63	3.05	6.85	10.85
	2	0.08	0.67	0.34	0.30	1.27
B. Specification						
Grade						
1. % Hygroscopicity	2	3.12	2.57	2.71	2.85	3.00
	24	10.17	7.84	13.53	16.76	19.49
	48	11.03	11.59	19.62	28.03	34.17
	72	11.80	15.26	25.93	39.35	48.94
	144	15.02	27.85	46.95	68.70	83.96
2. % Dehydration	163	15.62	30.80	51.69	74.20	91.15
	1	0.38	0.62	1.08	8.24	10.34
	2	0.00	-0.04	0.07	0.12	0.15
C. Fisher Reagent						
Grade						
1. % Hygroscopicity	2	3.58	2.19	2.78	2.33	2.67
	24	9.79	6.87	13.93	14.27	17.83
	48	10.38	16.20	20.02	23.66	31.27
	72	11.11	13.47	26.28	33.19	45.01
	144	13.83	24.59	46.99	61.45	80.28
2. % Dehydration	163	14.42	27.25	51.76	67.05	87.41
	1	0.29	0.52	0.34	1.42	2.60
	2	-0.02	-0.02	-0.07	0.15	0.80

TABLE 4

SPECIFICATION REQUIREMENTS OF
AMMONIUM NITRATE SAMPLES

	Gulf Oil Prills	Specification Grade	Fisher Reagent Grade	Hercules and Ketona Prills	Mississippi Prills
Chloroform soluble material %	.13	No test	No test*	.08	No analysis but it contains .2% boric acid, .2% am- monium acid phosphate and .01% diammonium sulfate
Ether soluble material %	No test*	.15 max	No test*	No	
Water insoluble material %	.48	.18 max	No test*	1.78	
Acidity as nitric acid %	.05	.02 max	*ph 8.5-60	.03	
Nitrites %	None	None	None	None	
Sulfates %	<.02	.02	.001	.02	
Chlorides %	<.02	.02 max	.0005	.02	
Ammonium Nitrate %	98.6	.99 min	.992 min	97.2	
Heavy Metal %	*	*	.005 max	*	
Diatomaceous Earth Coating %				1.73	
Sulfonate of Dimethyl Naphthalene Coating %	.06				
Density grams/cc	1.61	1.63 max	*	1.59	
Moisture %	.02	.15 max	*	.06	

Microcell E is composed of mostly of calcium silicate
AC-10 is composed mostly of aluminum silicate
were added to specification grade for test * No test

APPENDIX B

Figures

FIGURE 1

RELATIVE HUMIDITY STATIC TEST 60% & 90%
HYGROSCOPICITY OF AMMONIUM NITRATE AT 50°C

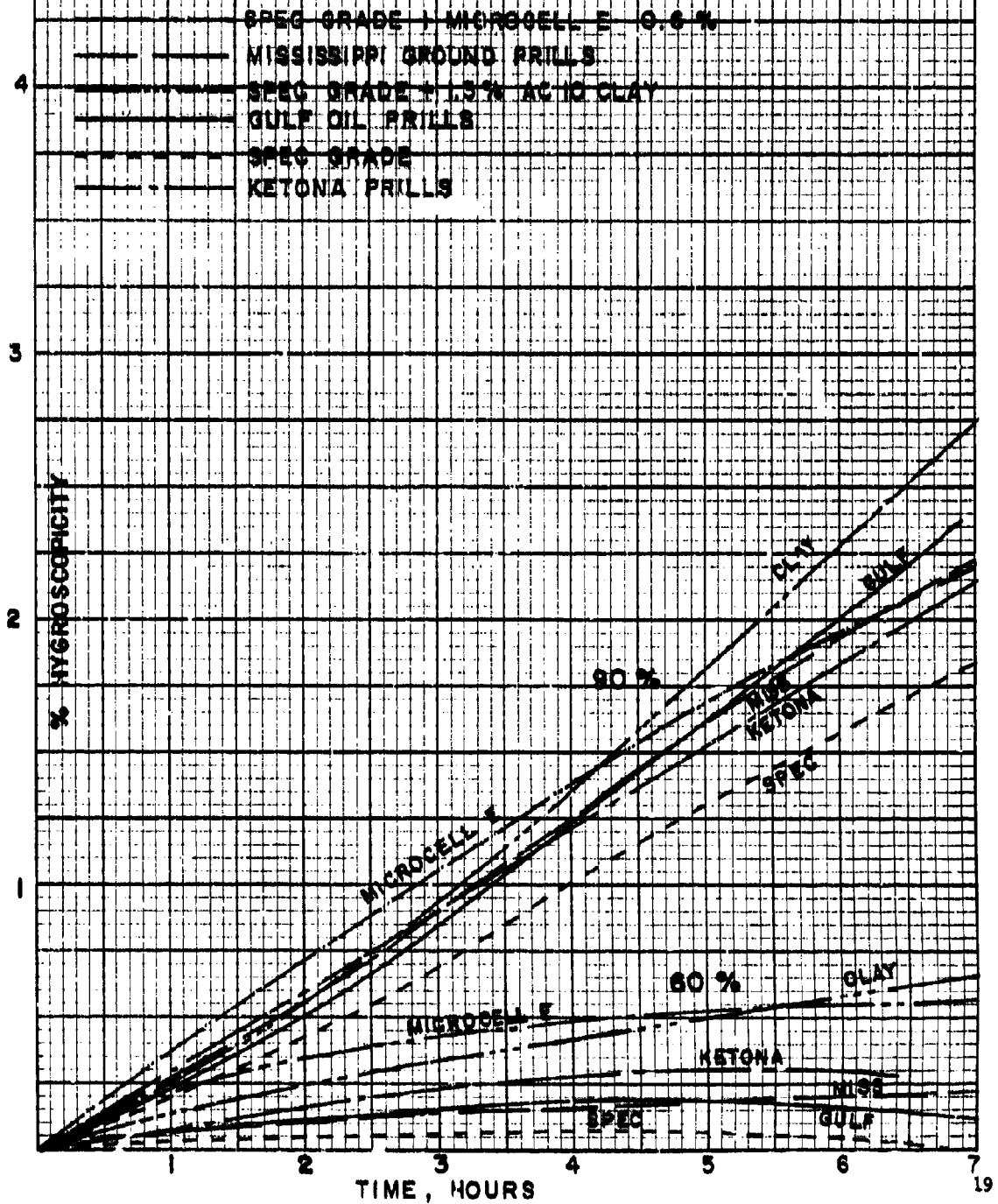
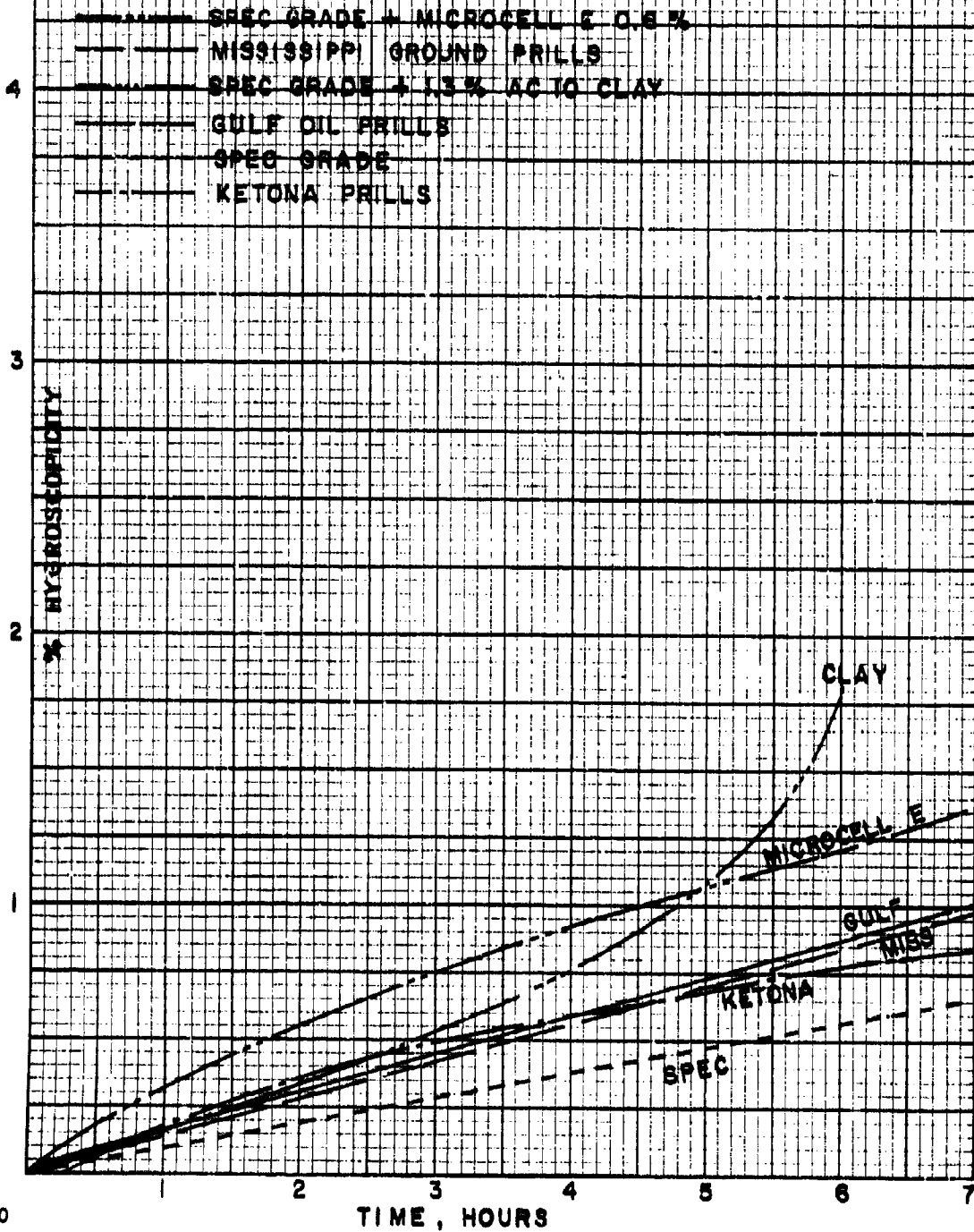


FIGURE 2

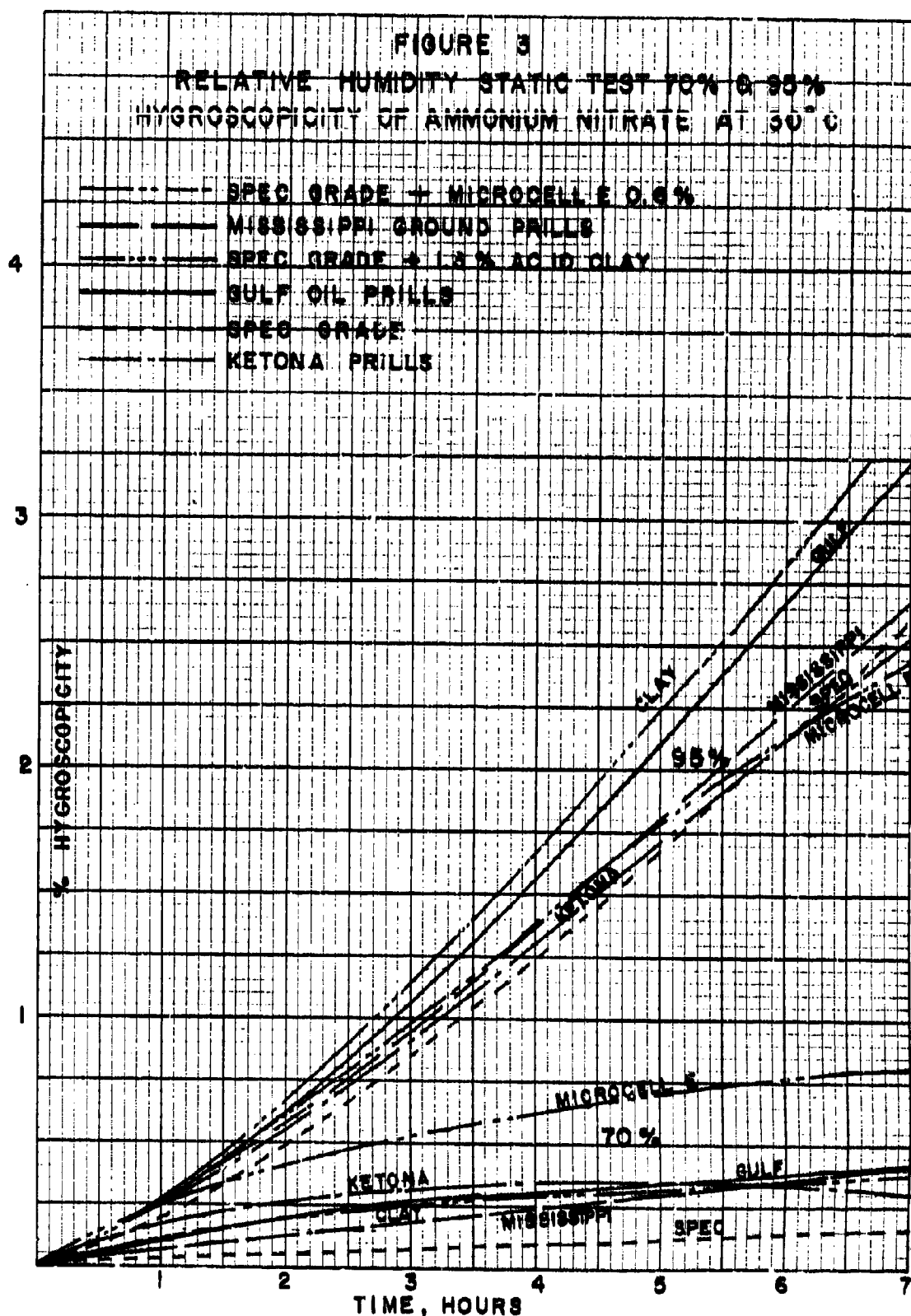
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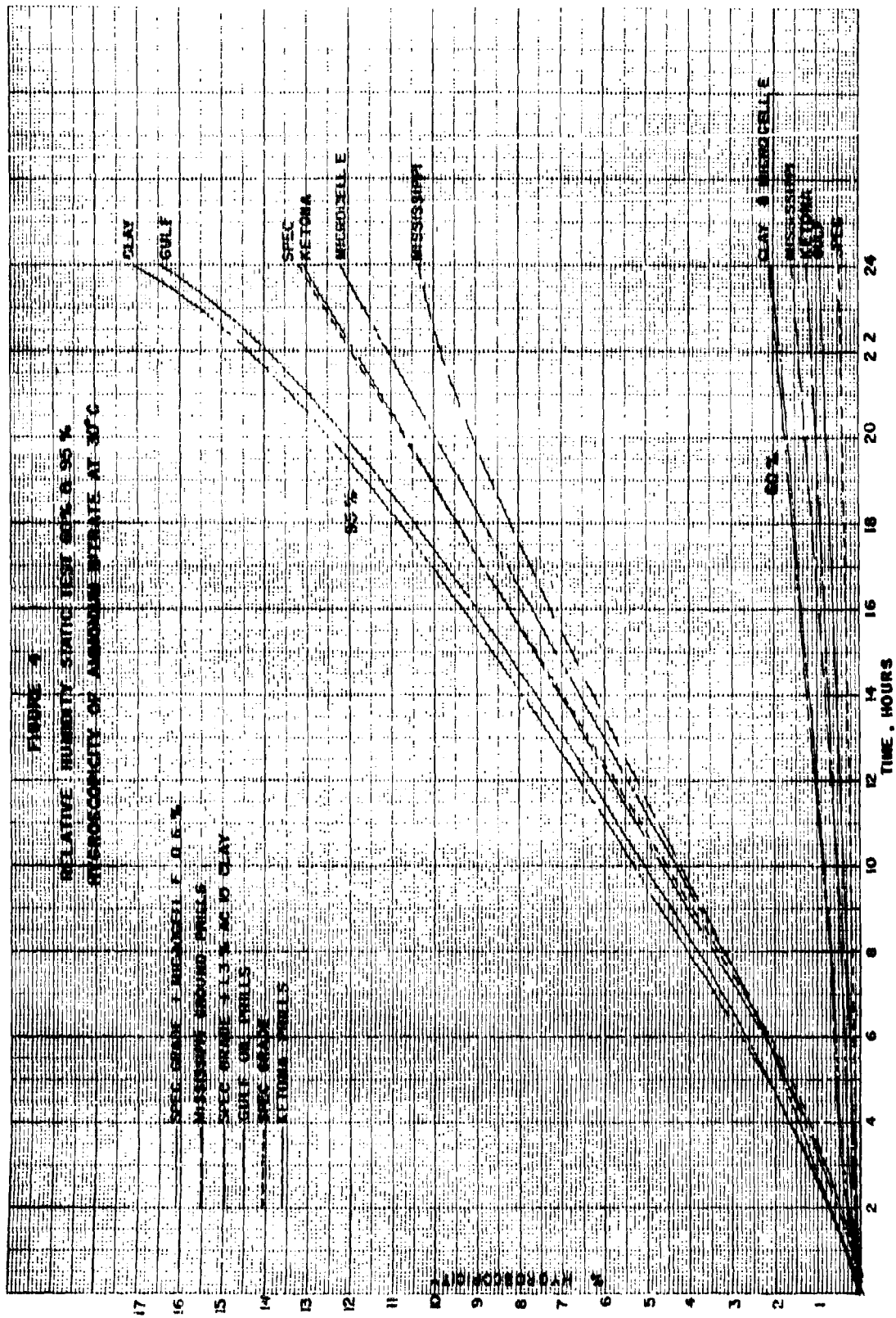
HYGROSCOPICITY OF AMMONIUM NITRATE AT 30°C

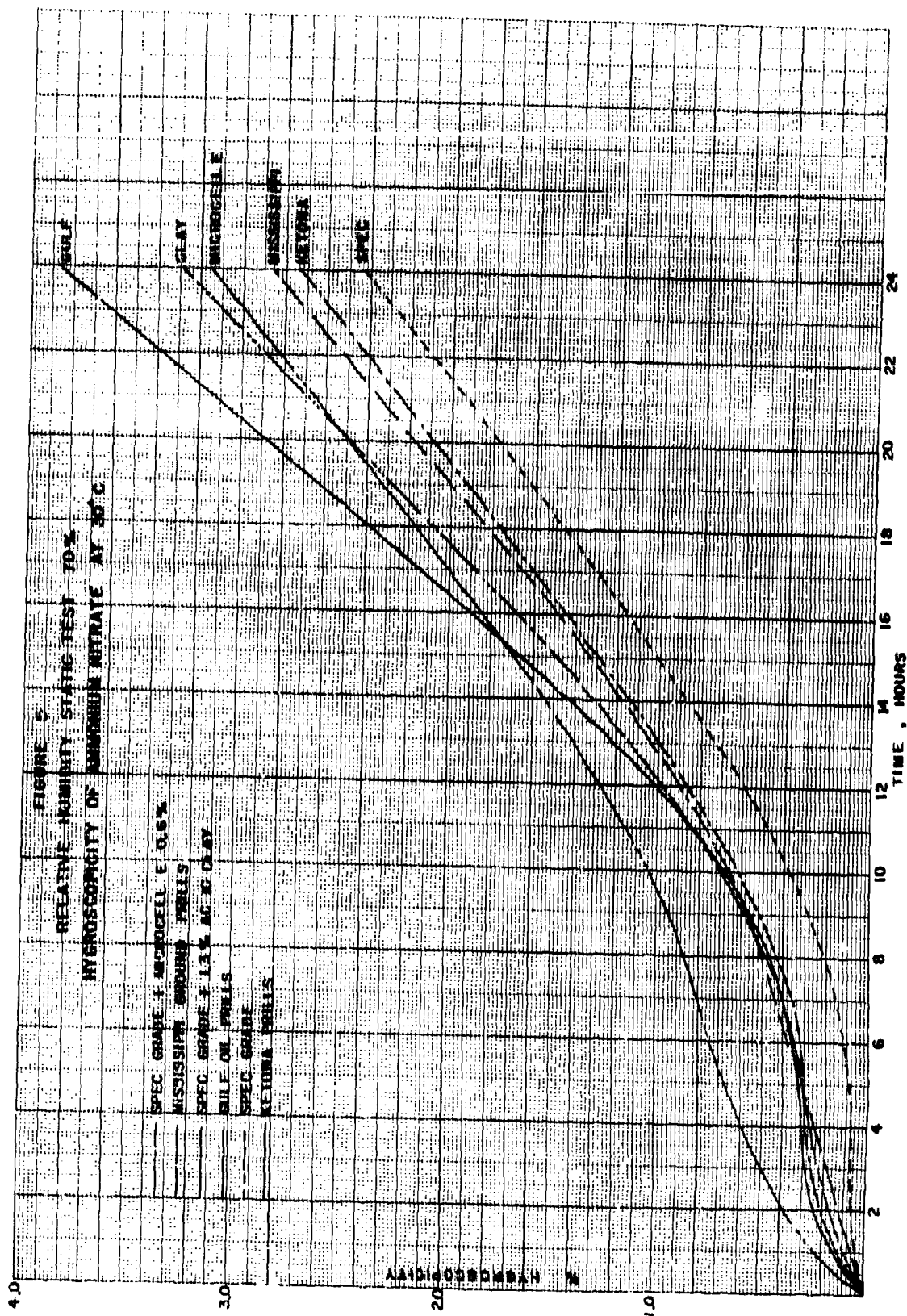


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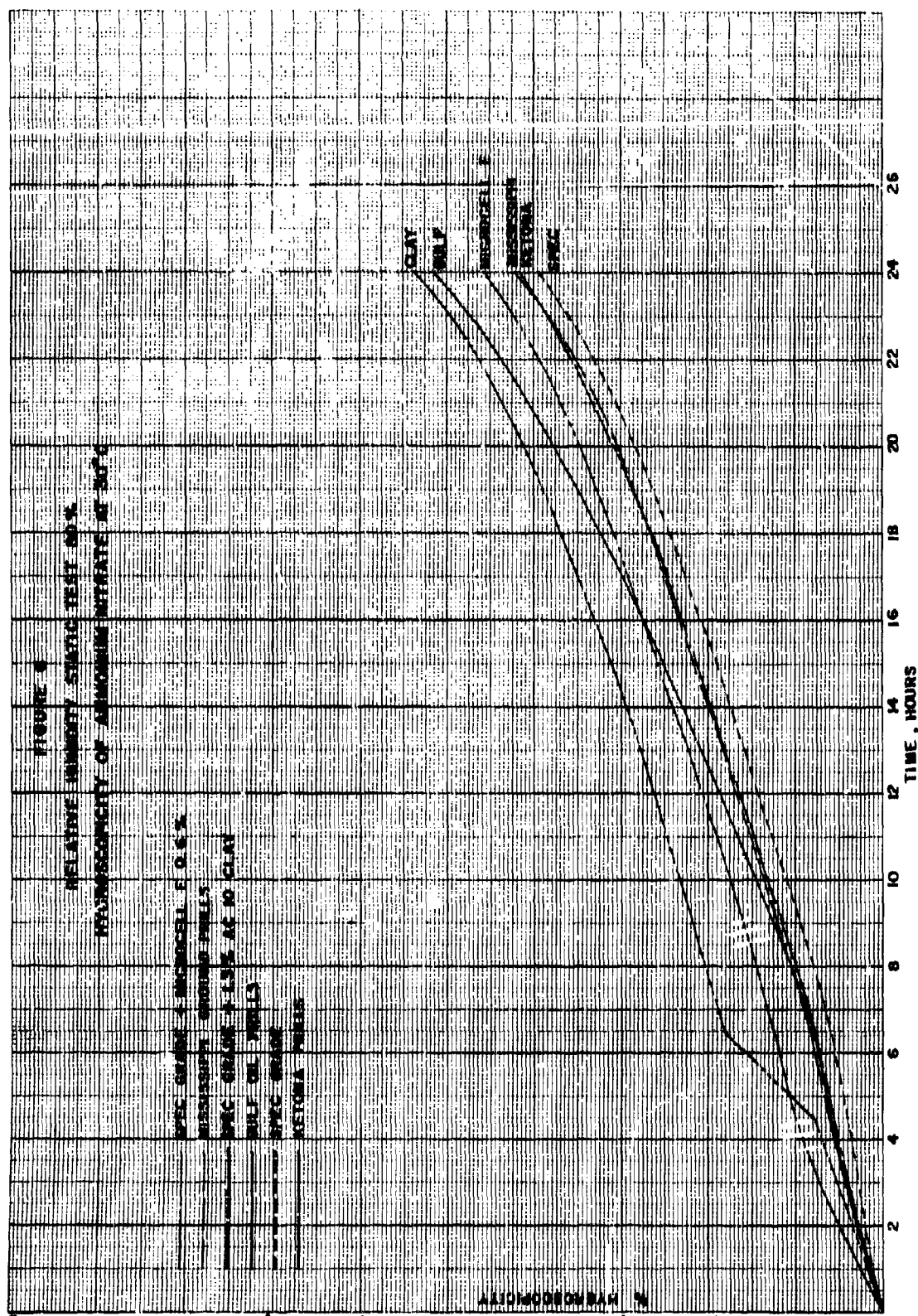
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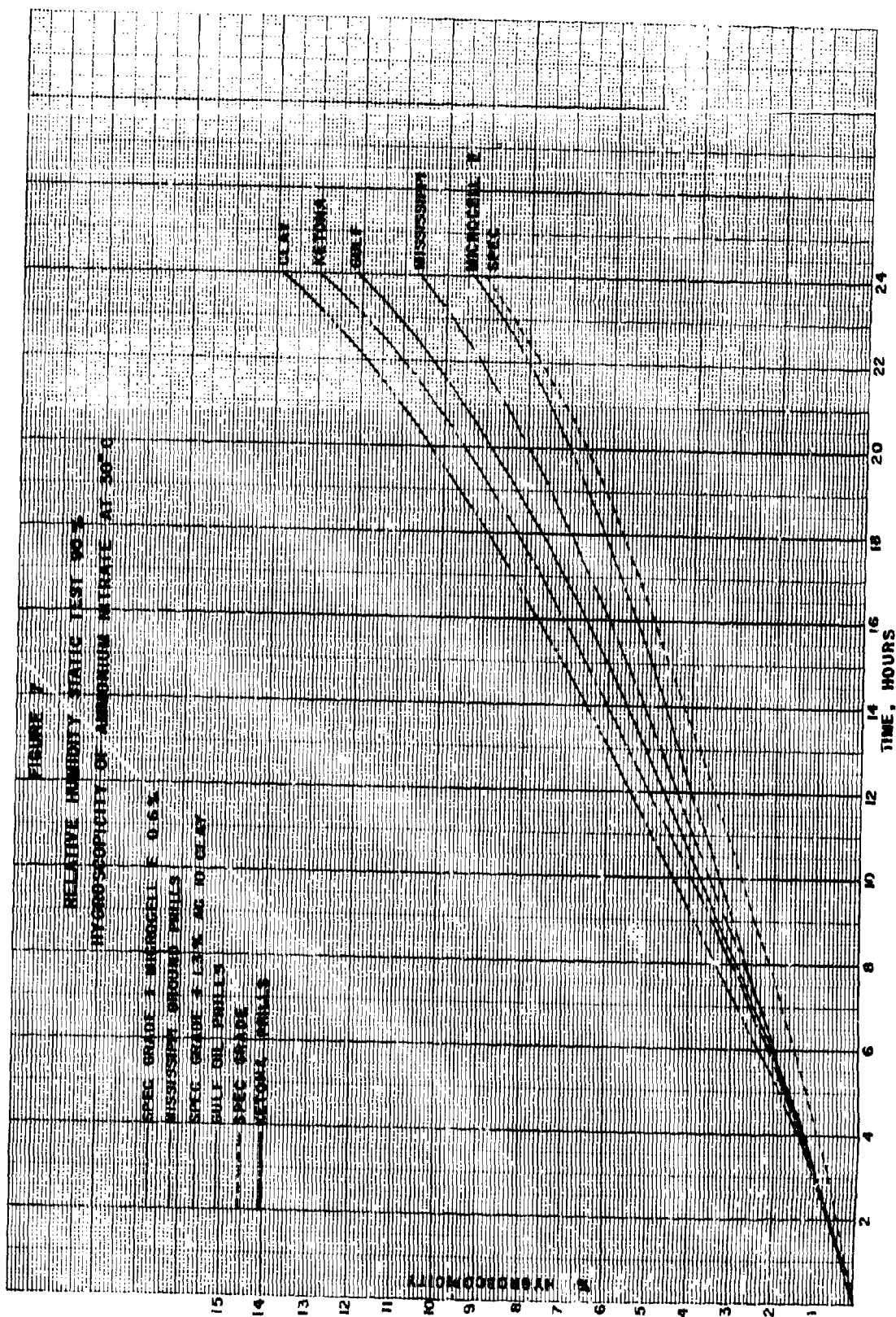
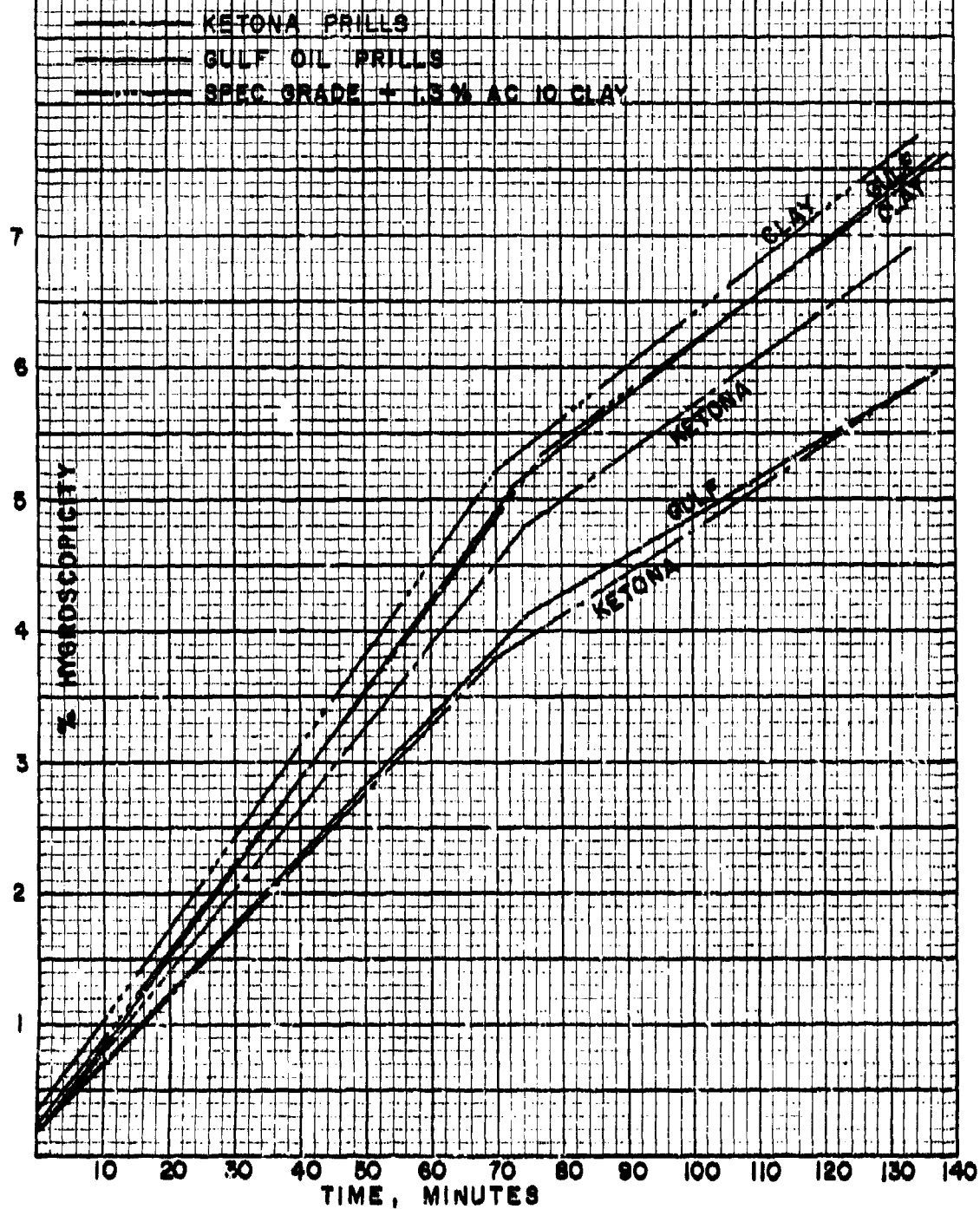
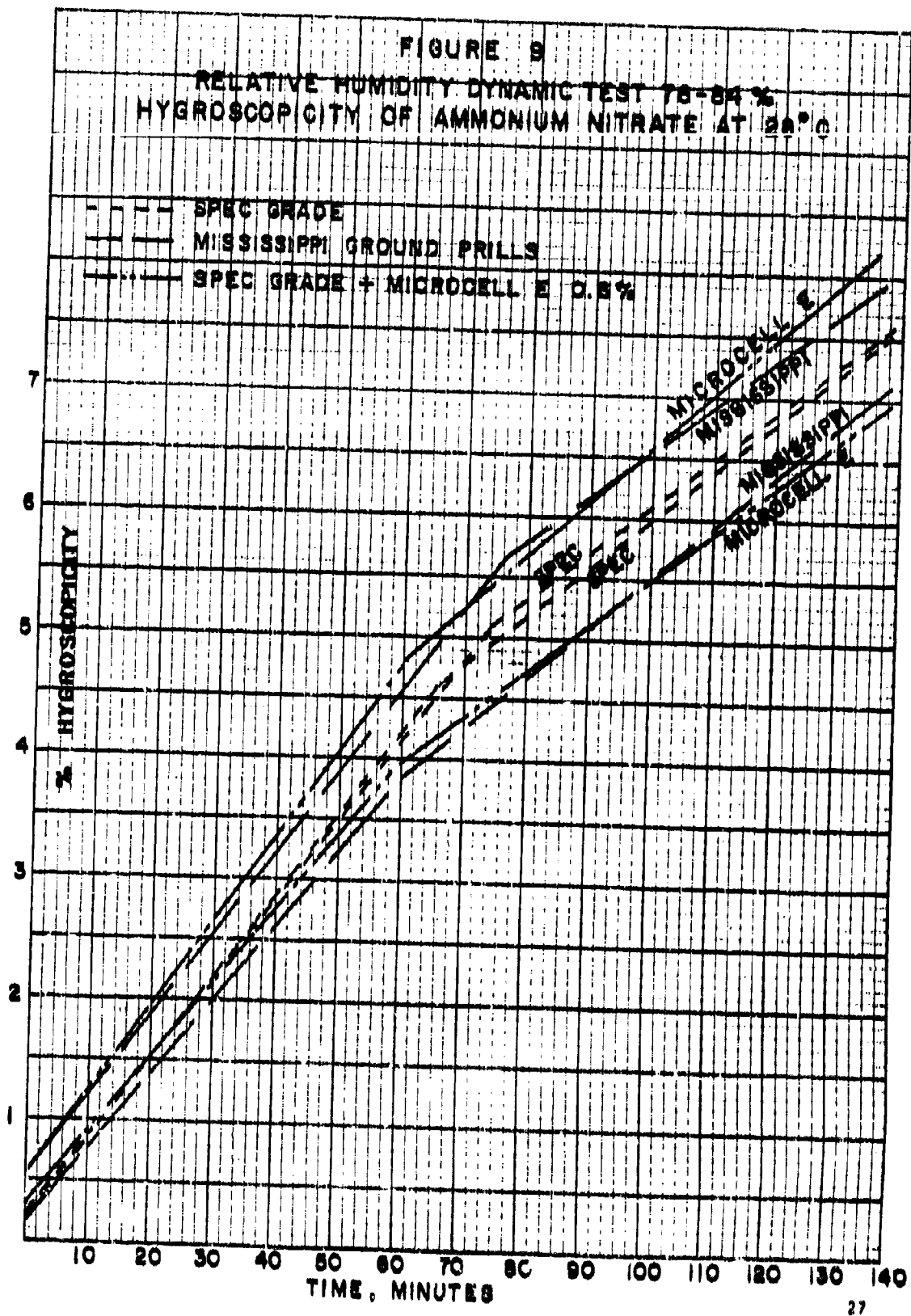
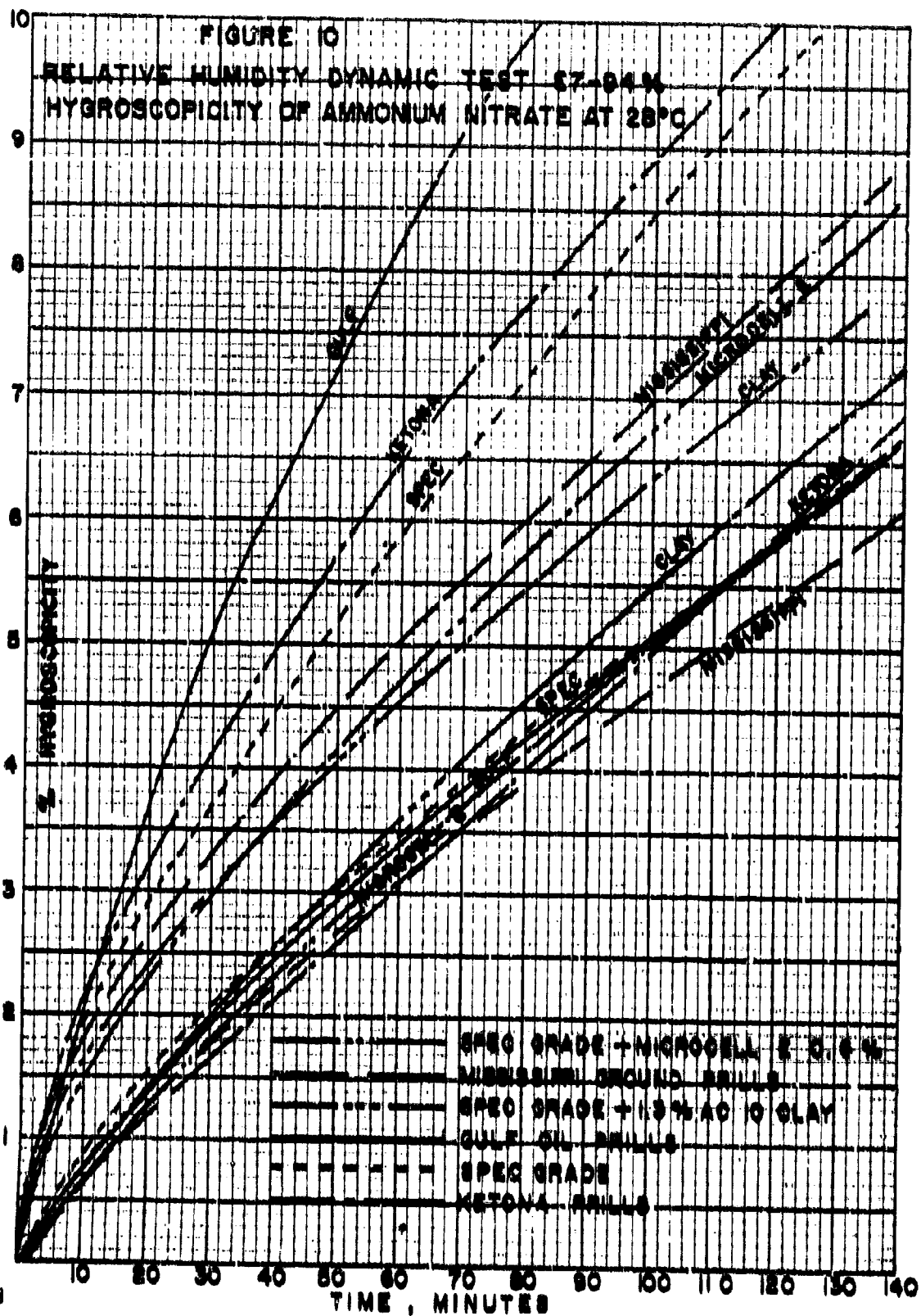


FIGURE 8
RELATIVE HUMIDITY DYNAMIC TEST 76-84%
HYGROSCOPICITY OF AMMONIUM NITRATE AT 28°C







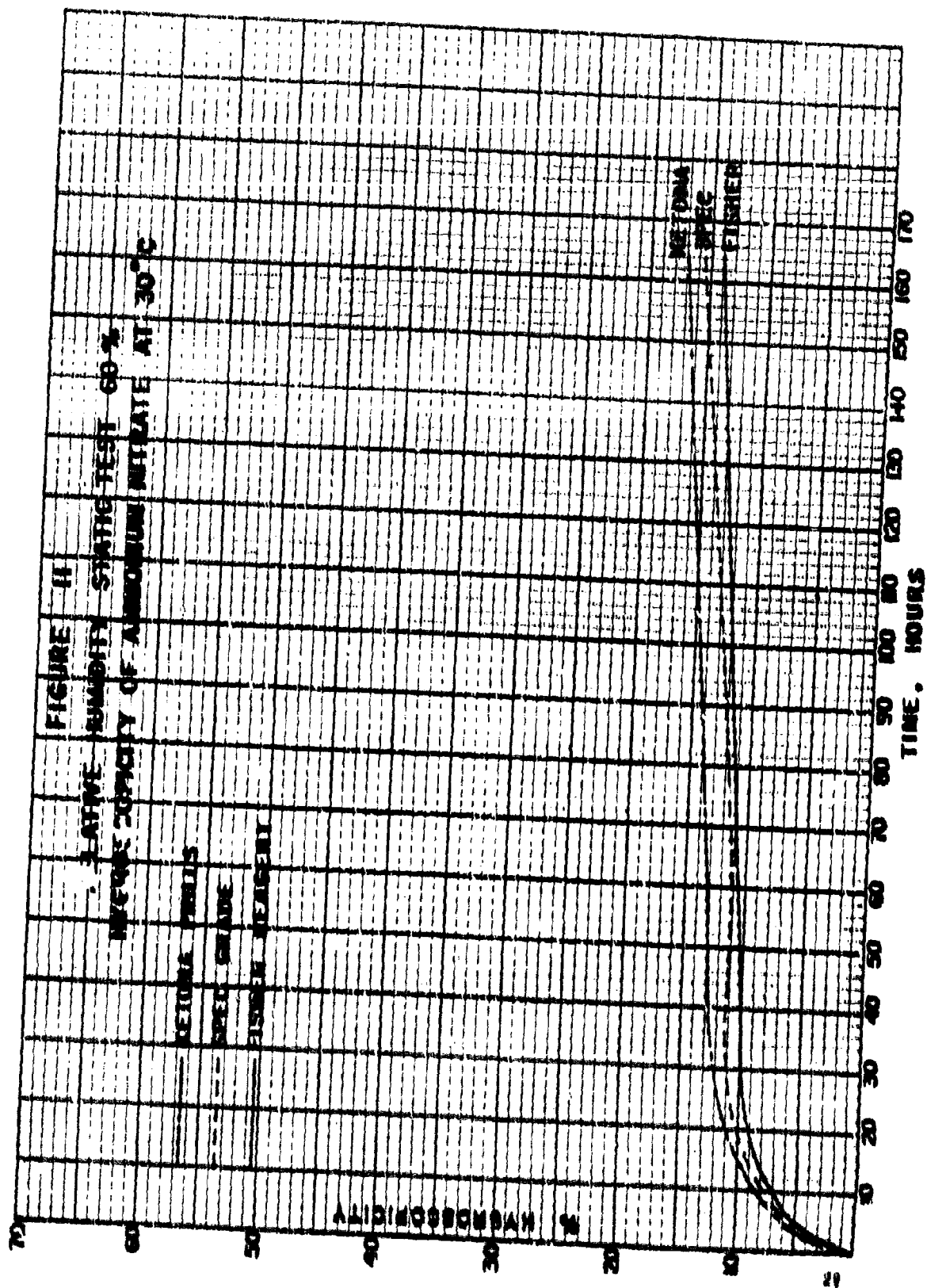
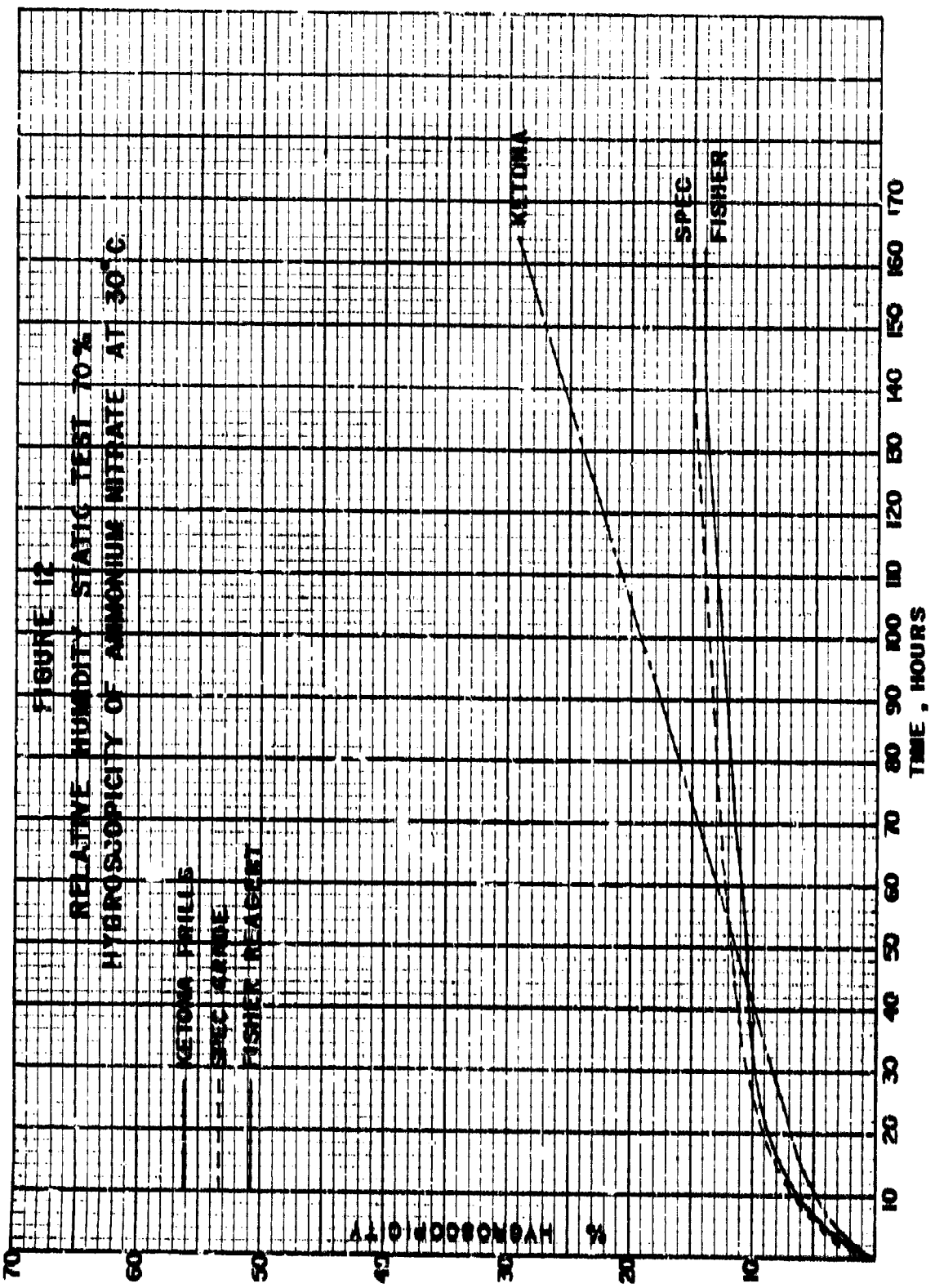
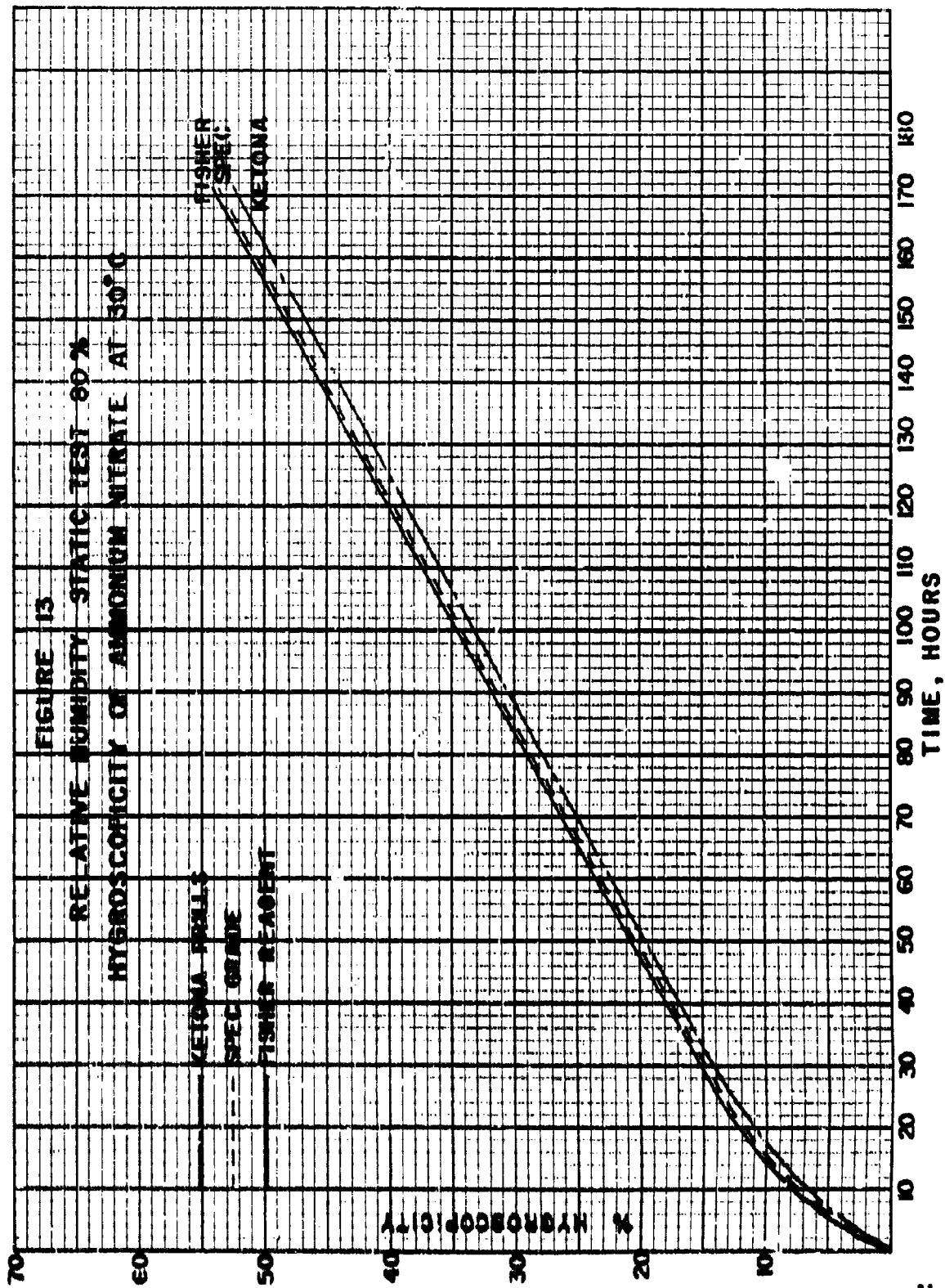
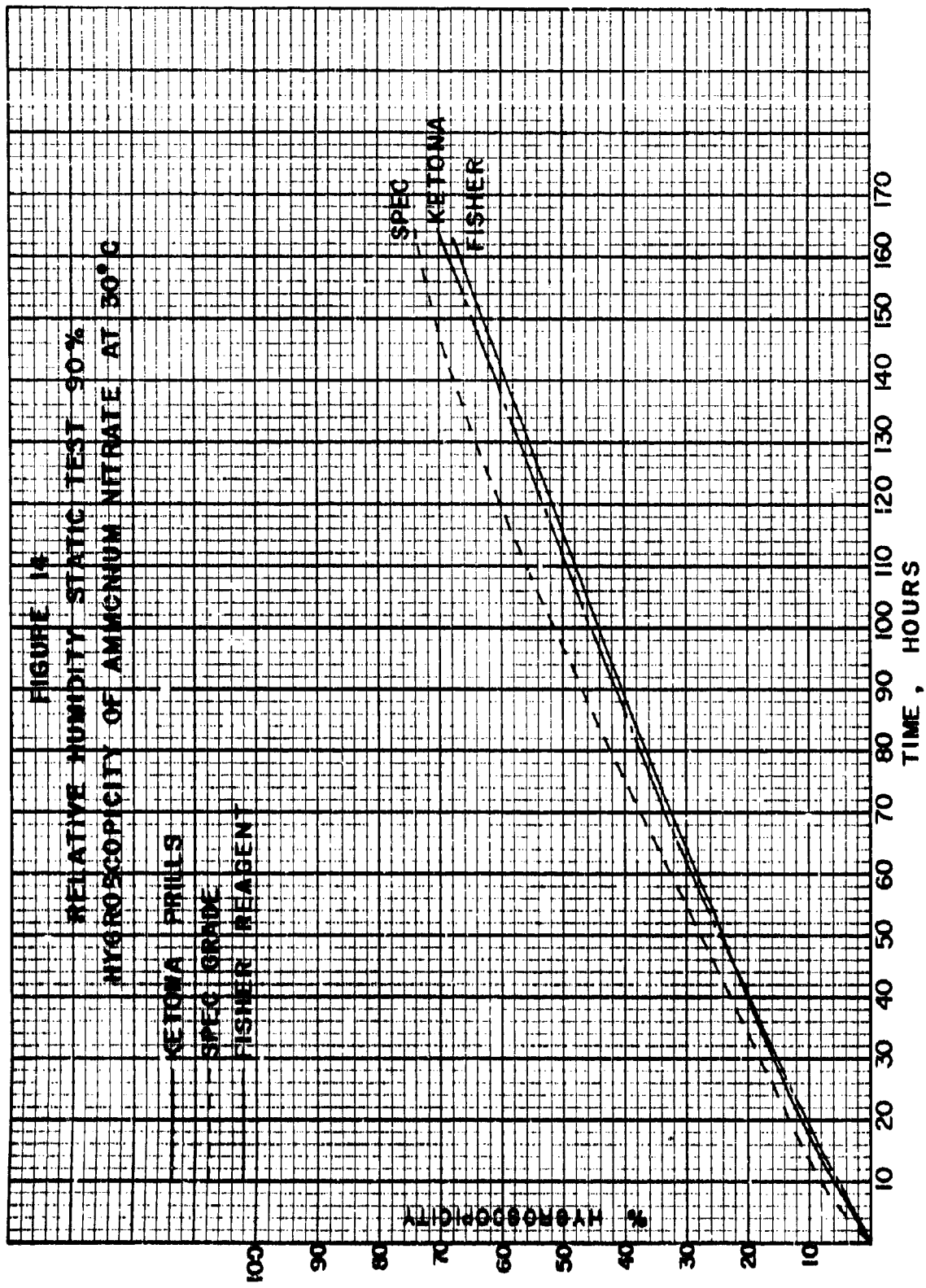


FIGURE 12

RELATIVE HUMIDITY - STATIC TEST 70%
HYGROSCOPICITY OF AMMONIUM NITRATE AT 30°C







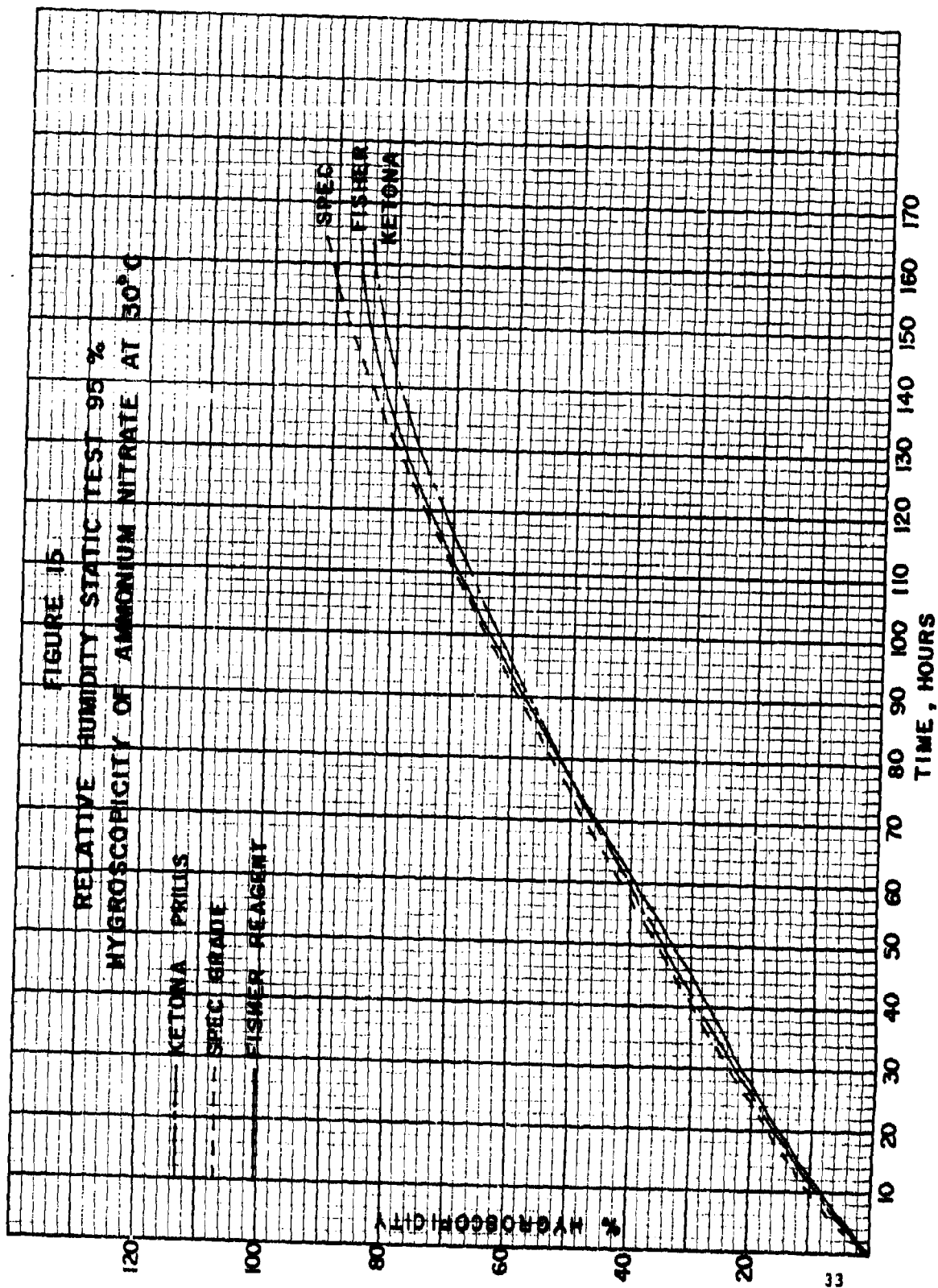
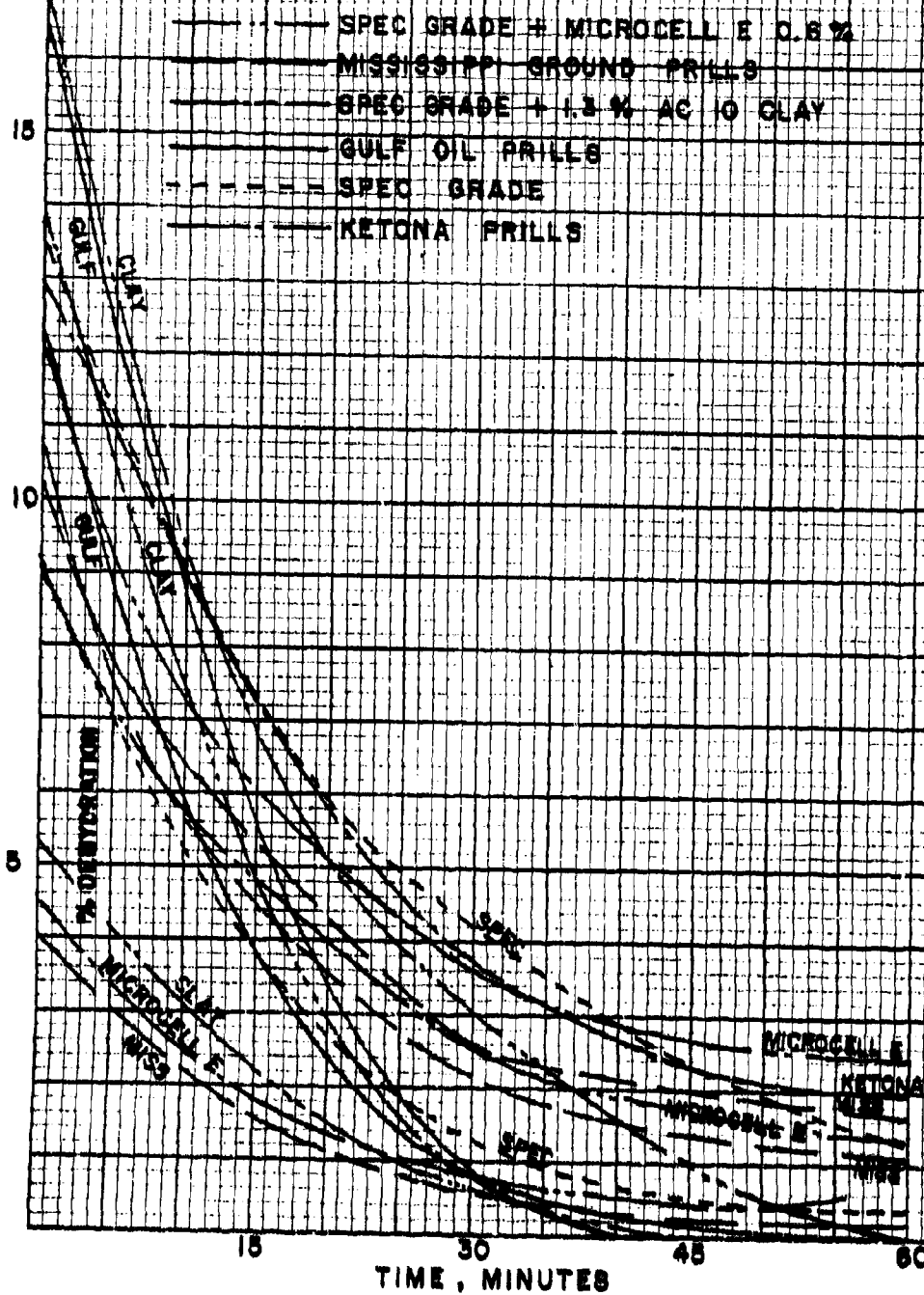
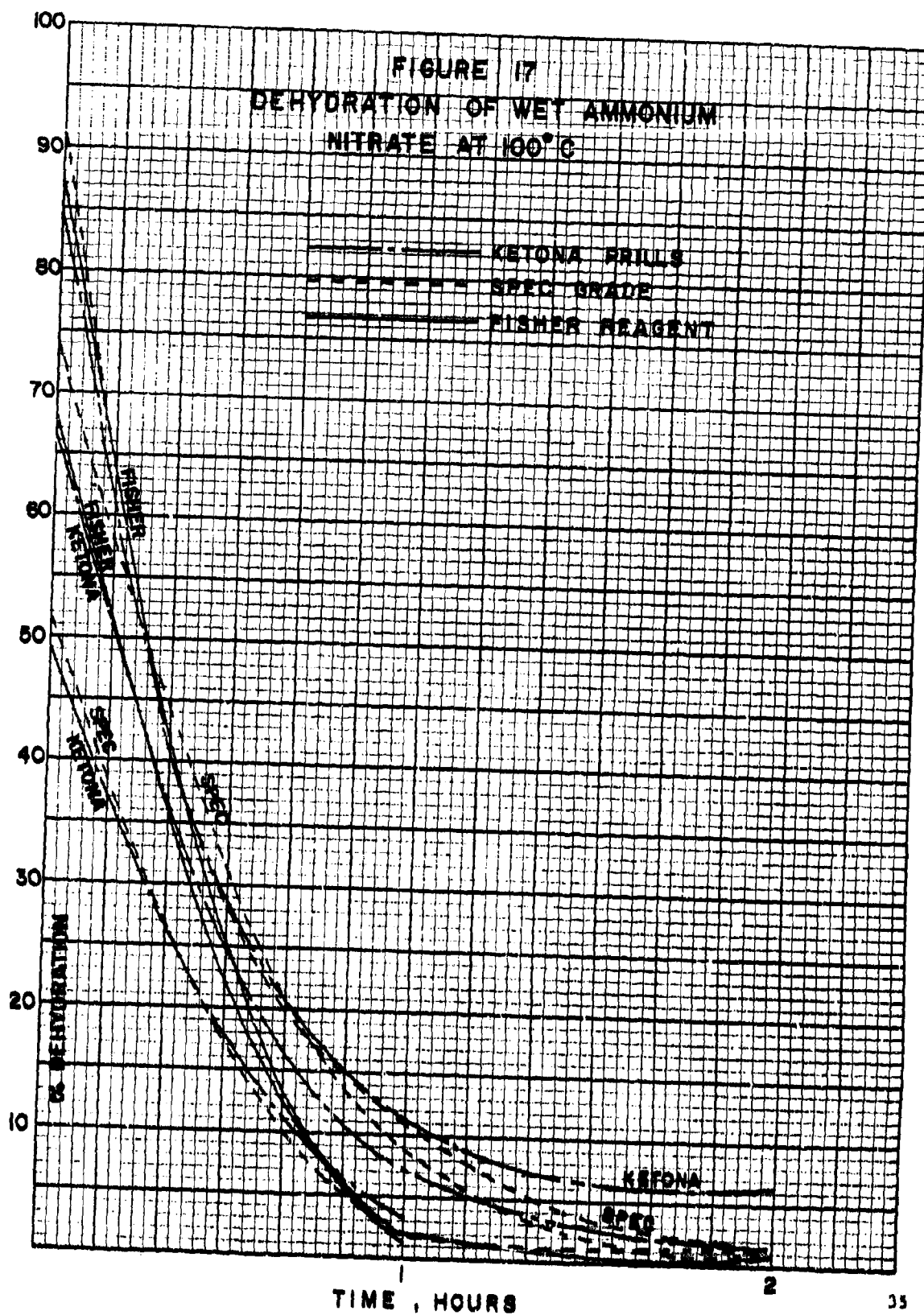


FIGURE 16
DEHYDRATION OF WET AMMONIUM
NITRATE AT 100°C





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13. ABSTRACT Ammonium nitrate is a component of many explosive systems, but a disadvantage in its use is its hygroscopicity. To extend the shelf life of explosives containing ammonium nitrate, it is important to minimize its hygroscopicity. Samples of ammonium nitrate were tested by the Ammunition Engineering Directorate's Artillery Ammunition Laboratory for hygroscopicity at relative humidity levels of 60%, 70%, 80% and 90% and between 28-30°C. Most of the samples of ammonium nitrate were coated; some were manufactured by a process producing prills; clay material was added in two cases. The results indicated that the ammonium nitrate which contained the least amount of impurities or additives also was the least hygroscopic.			

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