

Resins

STATUS REPORT

THE UTILITY OF PROCESS 11 VINYL CHLORIDE RESINS IN FLOORING APPLICATIONS

PHASE 1 - UNFILLED COMPOUNDS

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ABSTRACT

The performance of three ethylene modified vinyl chloride resins in an unfilled vinyl tile formulation are compared to conventional poly(vinyl chloride) resin. The resins exhibit greater flexibility, more resiliency, improved brittle temperature, greater extensibility, and easier processing characteristics than conventional Union Carbide Corporation suspension PVC counterparts. The level of ethylene contained in the sample of QXOM-7 did not exhibit the degree of influence on the tile properties or processing as that shown for QXOH-7 and QXOS-7.

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Research and Development Department
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THE UTILITY OF PROCESS 11 VINYL CHLORIDE RESINS IN FLOORING APPLICATIONS

PHASE 1 - UNFILLED COMPOUNDS

INTRODUCTION

The first of a three phase program designed to compare the performance of Process 11 vinyl chloride resins in vinyl flooring applications with conventional poly(vinyl chloride) homopolymers has been completed.

SUMMARY AND CONCLUSIONS

The performances of QXOH-7, QXOM-7, and QXOS-7 as compared to QYSJ, QYTQ-7, and QYSL-7, respectively, in a typical unfilled vinyl tile formulation may be summarized, in general, as follows for the examined physical properties.

- 1) The influence of ethylene at three levels of external plasticization with FLEXOL® plasticizer DOP (20, 40, and 60 PHR) promotes softer, more flexible, less brittle, easier processing, and more extensible compounds. Larger differences are shown at the higher levels of plasticization.
- 2) The influence of ethylene is more pronounced on QXOH-7, and QXOS-7 than on QXOM-7; the respective levels are 1.05, 0.89, and 2.06 per cent.
- 3) The Process 11 resins require lesser amounts of external plasticization to achieve a desired physical property as indicated:

QXOH-7	11 per cent of total plasticizer in QYSJ
QXOM-7	7 per cent of total plasticizer in QYTQ-7
QXOS-7	21 per cent of total plasticizer in QYSL
- 4) Neither plasticizer incompatibility nor resin contribution to staining appear to be a problem.
- 5) QXOH-7 and QXOS-7 exhibit wider differences in processability than QXOM-7 when compared to their respective homopolymer controls. Higher torques are encountered during the fusion of the Process 11 resins when tested on the Torque-Rheometer.
- 6) QXOH-7 and QXOM-7 are suitable for conventional all vinyl tile; QXOS-7 may be used but higher processing temperatures are required.

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WAS:
YES. 585
TABLE #I
1.05
.89
2.06

PROPORTIONAL TO C₂H₄ CONTENT

Incorrect!

DISCUSSION

This study is designed to show the effects of comonomer level, molecular weight, and plasticizer level on several important unfilled vinyl tile properties. The data are shown in Tables I thru III and in Figures 1 through 17. The observations reported herein are based more on trends rather than single points. Comments relative to the specific tests conducted in this investigation follow:

Shore A and D Hardness - Both tests, a measure of indentation of the approximate hardness of rubberlike materials, were run to insure comparative ratings on the soft to hard compounds. The Process 11 resins are indicated as exhibiting consistently lower readings (softer) than the controls as follows in a table (and others that follow) to indicate relative differences. Please refer to Table II for specific comparisons.

<u>Resin</u>	<u>Average Test Units Lower Than Control</u>			
	<u>Shore A</u>		<u>Shore D</u>	
QXOH-7 1.05	2.0	(2%)	4.0	(9%)
QXOM-7 .50	2.0	(2.5%)	4.4	(7.2%)
QXOS-7 2.06	5.3	(6.0%)	8.7	(18.0%)

This indicates the effect of comonomer level and external plasticization on the subject resins; QXOS-7 is indicated as the most sensitive or most efficient in terms of internal plasticization.

McBurney Indentation, a measure of indentation resistance, indicates that the Process 11 resin is more susceptible to external plasticization than their homopolymer counterparts as shown below:

<u>Resin</u>	<u>Average Test Units Lower Than Control</u>			
	<u>McBurney</u>			
	<u>1 Minute, mils</u>		<u>10 Minute, mils</u>	
QXOH-7 1.05	5.9	(12.5%)	3.9	(11.0%)
QXOM-7 .50	0		3.4	(11.1%)
QXOS-7 2.06	8.8	(14.0%)	10.0	(13.6%)

Again, QXOS-7 is indicated as the most efficient of the Process 11 resins tested.

Residual Indentation is a measure of indentation and of indentation recovery. The differences between the Process 11 resins and the controls are very slight as indicated below:

<u>Resin</u>	<u>Average Test Units Higher Than Control</u>			
	<u>Residual Indentation, mils</u>		<u>Per Cent Recovery</u>	
QXOH-7	0.96	(24.5%)	-4	(5%)
QXOM-7	0.06	(1.9%)	-3	(3.7%)
QXOS-7	0.68	(25.0%)	+17	(21%)

All resins except QYSL exhibited a recovery of greater than 75 per cent, the minimum acceptable limit (Federal Specification L-T-751).

Tensile and Elongation are measures of extensibility. The Process 11 resins show a slight loss in tensile strength and a slight gain in elongation as expected. A comparison with the control resins follows:

<u>Resin</u>	<u>Average Test Units Compared With Control</u>			
	<u>Tensile, psi</u>		<u>Elongation, %</u>	
	<u>Lower</u>		<u>Higher</u>	
QXOH-7	162	(6.3%)	32	(13.8%)
QXOM-7	89	(3.0%)	18	(6.3%)
QXOS-7	286	(8.5%)	33	(11.1%)

The slight loss in tensile is not considered as serious for most flooring applications.

100 Per Cent Modulus is a measure of room temperature stiffness. The Process 11 resins exhibit a consistently lower value than the respective homopolymer control i.e. less stiff. A comparison with the controls follows:

<u>Resin</u>	<u>Average Test Units Lower Than Control</u>	
	<u>Modulus, psi</u>	
QXOH-7	222	(10.5%)
QXOM-7	165	(7.6%)
QXOS-7	433	(22.0%)

QXOS-7, again, is indicated as the most efficient of the resins tested.

Brittle Temperature (ASTM-D-746) is a measure of low temperature performance in impact. The Process 11 resins exhibit consistently better performance as shown by the following data:

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<u>Resin</u>	<u>Average Test Units Lower Than Control</u>	
	<u>Brittleness, °C</u>	
QXOH-7	4	(67%)
QXOM-7	3.3	(36%)
QXOS-7	6.7	(59%)

Apparent Melt Viscosity is a measure of processability. These compounds were tested at the standard conditions for plasticized compounds. The comparison with the controls follow:

<u>Resin</u>	<u>Average Test Units Lower Than Control</u>	
	<u>Apparent Melt Viscosity</u>	<u>Equilibrium Temperature</u>
	<u>m-g/sec</u>	<u>°C</u>
QXOH-7	210 (15%)	2 (1%)
QXOM-7	13 (0.8%)	0
QXOS-7	200 (5%) *	1 (0.7%) *

* 40 phr only

The melt characteristics of QXOH-7 and QXOS-7 are in general agreement with previous findings that indicate easier processing; the performance of QXOM-7 did not follow these previous results and is not consistent with the QXOH-7 and QXOS-7 performances.

Fusion Characteristics are a measure of the energy requirements in the initial processing operation. The Process 11 resins are more easily fused as indicated below:

<u>Resin</u>	<u>Average Test Units Lower Than Control</u>	
	<u>Fluxing Time</u>	<u>Fusion</u>
	<u>min.</u>	<u>Temperature, °C</u>
QXOH-7	1.2 (20%)	7 (6%)
QXOM-7	4.2 (43%)	11 (10%)
QXOS-7	11.0 (81%) *	25 (22%) *

* - 60 phr

Higher maximum torques are obtained with the Process 11 resins which may be related to differences in particle geometrics. The faster fluxing of the Process 11 resins is thought to be related to the higher torques and thus higher frictional heat.

Stain Resistance - The Process 11 resins exhibit a stain level comparable to the controls. The stain levels of all resins were directly proportional to plasticizer concentration.

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Attachments:

3 Tables

17 Figures

TABLE I

PROCESS 11 - VINYL CHLORIDE RESINS

CHEMICAL AND PHYSICAL PROPERTIES

	<u>QXOH-7</u> <u>Blend 1</u>	<u>QXOM-7</u> <u>Blend 3</u>	<u>QXOS-7</u> <u>Blend 4</u>
Ethylene, %	1.05	0.89	2.06
Inherent viscosity, ASTM A	0.775	0.98	1.31
Heating Loss, %	0.3	0.2	0.6
Contamination, slurry	2	1	2
Apparent density, lbs/ft ³	32.5	29	27
Particle size, median	160	181	215
Thru 200 - mesh, %	7	2	2
Hard resin particles,			
17.5 mil, ft. ⁻²	40	0	0
10 mil, ft. ⁻²	1152	25	83
Clear fisheye test	8	2	2
Initial color mill test	102	113	114
Resin flow time, sec/400 cc	3.4	-	-
Dry blend time, min.	4.8	3.3	2.0
°C	81	79	77

TABLE II

PROCESS 7 AND 11 POLY(VINYL CHLORIDE) RESIN

UNFILLED VINYL TILE PROPERTIES

Resin	Plasticizer Concentra- tion, phr	Durometer		McBurney Indentation,		Residual Indentation		Tensile Strength, psi	Ultimate Elonga- tion, %	100% Modulus, psi	Brittle- ness, %	Stain - Relative ⁽¹⁾		Apparent Melt Viscosity		Fusion Characteristics		
				Mils		Mils	Recov- ery %					Asphalt	Sandura	m-g/sec	°C	Time, min	Temp. °C	Max. Torque
		"A"	"D"	1 min	10 min													
QYSJ	20	97	73	4.8	6.4	0.16	84.5	3290	140	3280	+20	1	1	2000	154	4.0	111	4600
	40	84	38	30.9	37.4	2.3	93.8	2700	240	2050	-14	3	4	1270	146	6.8	109	1270
	60	71	22	36.5	51.0	6.4	73.8	1820	310	1070	-24	5	5	860	141	7.0	107	860
QXOH-7 Blend 1	20	96	68	7.3	9.8	0.99	78.3	3090	210	3040	+10	1	1	1720	151	3.3	110	4650
	40	82	33	34.4	36.0	2.5	94.4	2590	260	1810	-16	3	4	1090	144	3.8	97	3350
	60	68	20	48.4	60.8	8.3	68.2	1640	330	880	-24	5	5	690	140	7.0	98	1800
QYTQ-7	20	98	74	4.6	6.4	0.16	79.2	3550	200	3420	+16	1	1	2400	158	4.7	114	4700
	40	85	40	33.2	40.0	2.8	93.2	3040	280	2080	-16	3	4	1540	149	11.4	120	2550
	60	70	23	45.0	49.6	6.1	83.3	2330	380	1050	-28	5	5	1050	144	13.2	114	1050
QXOM-7 Blend 3	20	96	70	6.3	9.6	0.45	79.3	3340	200	3230	+12	1	1	2440	157	2.5	101	2440
	40	83	35	28.3	39.6	2.3	94.2	2960	300	1840	-18	3	4	1490	148	7.2	109	1490
	60	68	22	47.8	57.5	6.5	72.3	2350	400	990	-32	5	5	1020	144	7.2	106	1020
QYSL-7	20	97	76	4.0	5.0	0.0	16.7	3800	220	3650	+18	1	1	DNF			DNF	
	40	89	43	25.8	37.2	1.8	94.5	3590	300	2410	-20	3	4	2100	152		DNF	
	60	75	25	35.0	39.6	4.3	79.1	2700	370	1250	-32	5	5	1500	147	19.9	138	1350
QXOS-7 Blend 4	20	97	67	9.2	13.6	0.82	84.6	3700	260	3010	+10	1	1	3050	162		DNF	
	40	81	31	36.2	43.2	2.2	94.1	3060	320	1740	-26	3	4	1900	151	7.7	117	3000
	60	67	20	45.9	55.2	5.1	71.2	2470	410	970	-38	5	5	1170	145	8.9	113	2150

Test Formulation

Resin 100 parts
FLEXOL® DOP as shown
Mark KCB 3
Stearic Acid 0.25

Specimen Preparation

Two roll 8" x 16" mill
Roll speed - 70 fpm
Roll Temperature - 170°C
Mill time - 4 minutes after fluxing
Hydraulic press - platen temperature - 170°C

1) Relative rating System 1 = Good 5 = Poor

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TABLE III

PROCESS 7 AND 11 POLY (VINYL CHLORIDE) RESIN

UNFILLED VINYL TILE PROPERTIES

PERFORMANCE SUMMARY

<u>Test</u>	<u>Efficiency Parameter</u>	<u>Per Cent Less Plasticizer Required By Process 11 Resins Than Control</u>		
		<u>QXOH-7</u>	<u>QXOM-7</u>	<u>QXOS-7</u>
Shore A	80	8.6	6.4	24.1
Shore D	50	10.3	14.3	27.3
McBurney One-Minute, mils	22	5.1	10.6	24.4
Residual Indentation, mils	4.0	3.2	0	6.9
Tensile Strength, psi	3000	22.0	7.7	25.7
Ultimate Elongation, %	300	8.5	9.1	22.7
100% Modulus, psi	2000	9.8	9.6	24.1
Brittleness, °C	0	6.9	4.3	12.4
Apparent Melt Viscosity, m-g/sec	1500	21.5	0	20.7

Figure 1

All Vinyl Tile - Shore Hardness "A"

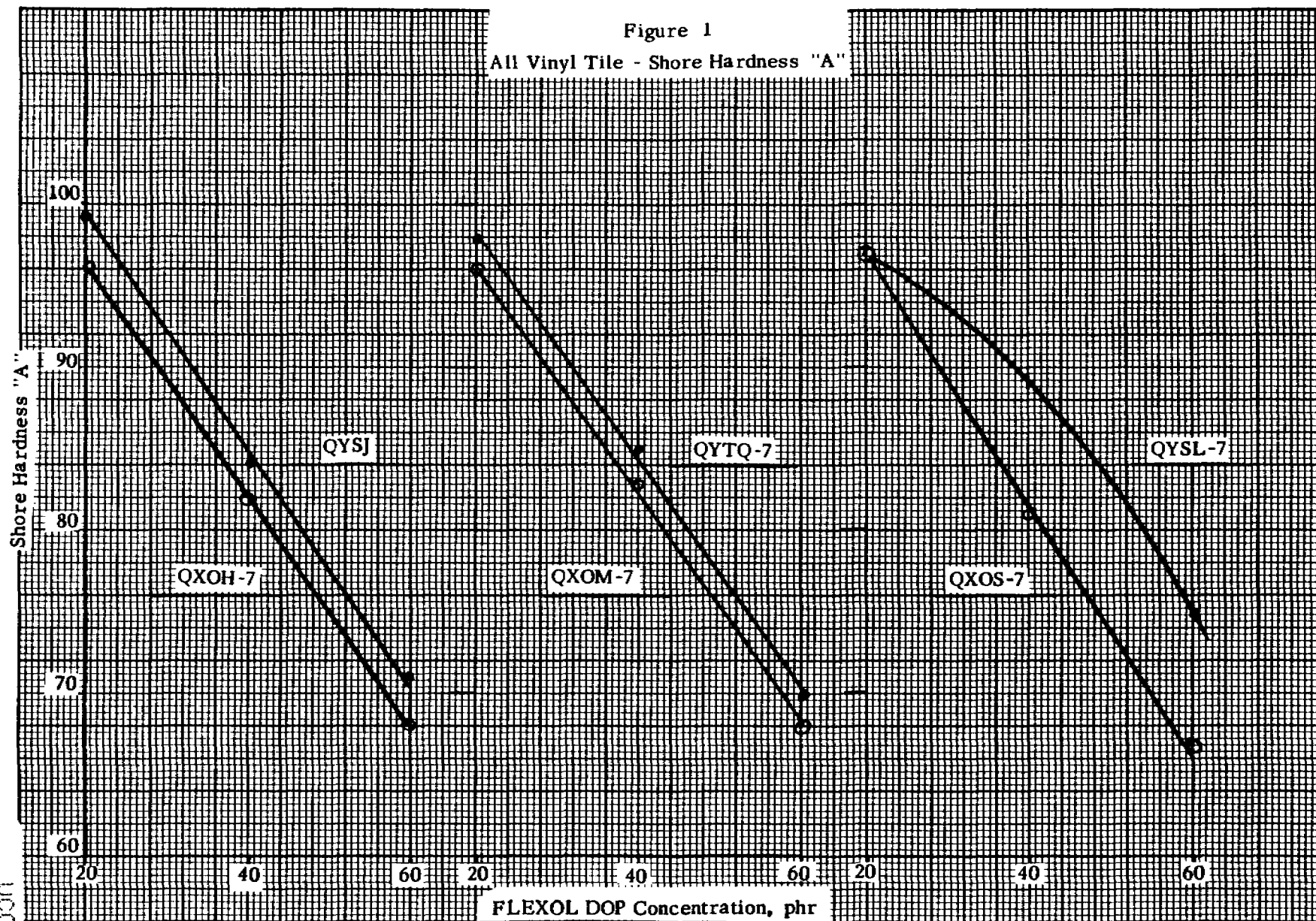


Figure 2

All Vinyl Tile - Shore Hardness "D"

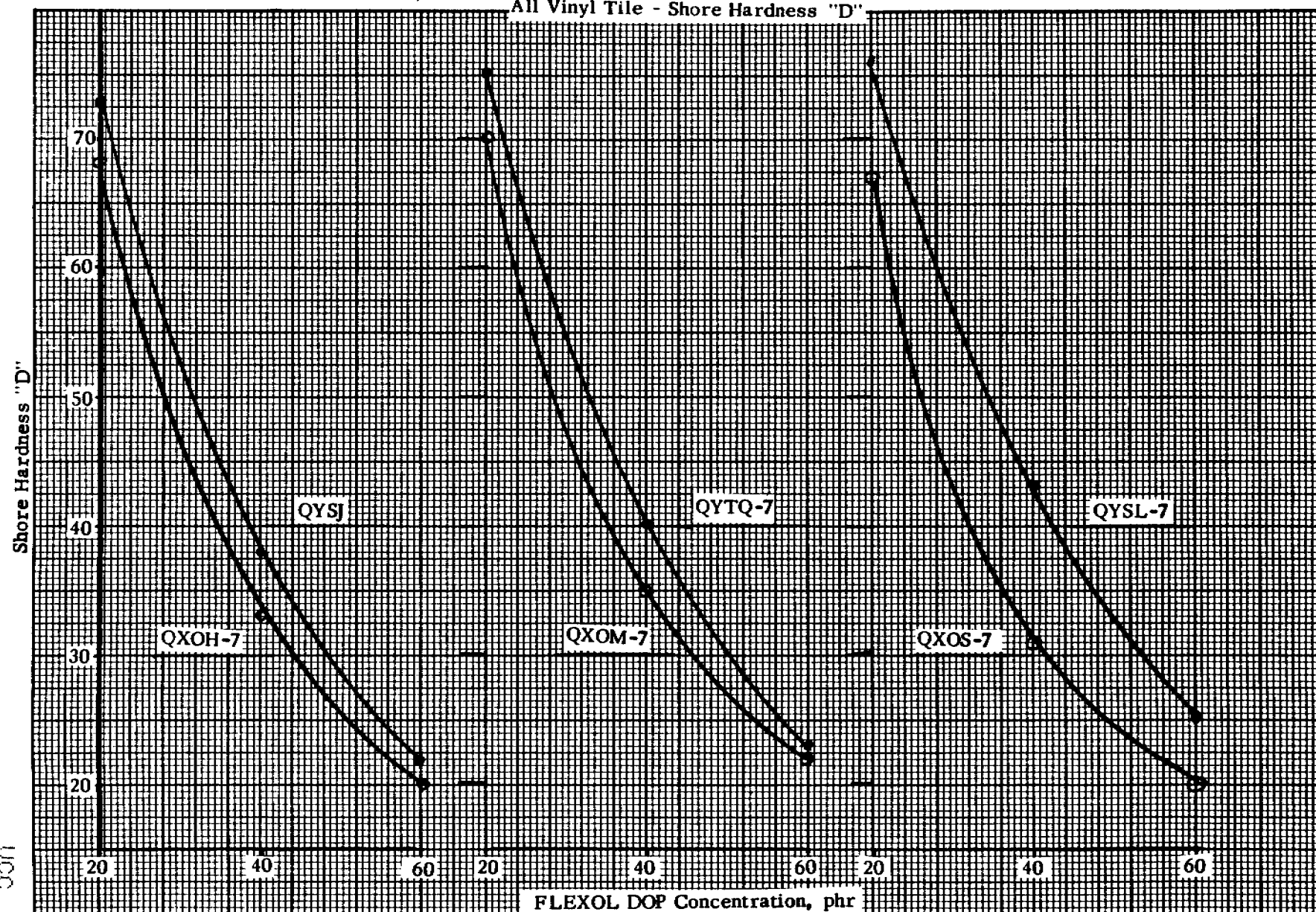


Figure 3

All Vinyl Tile - One Minute McBurney

McBurney Indentation, One Minute, Mills

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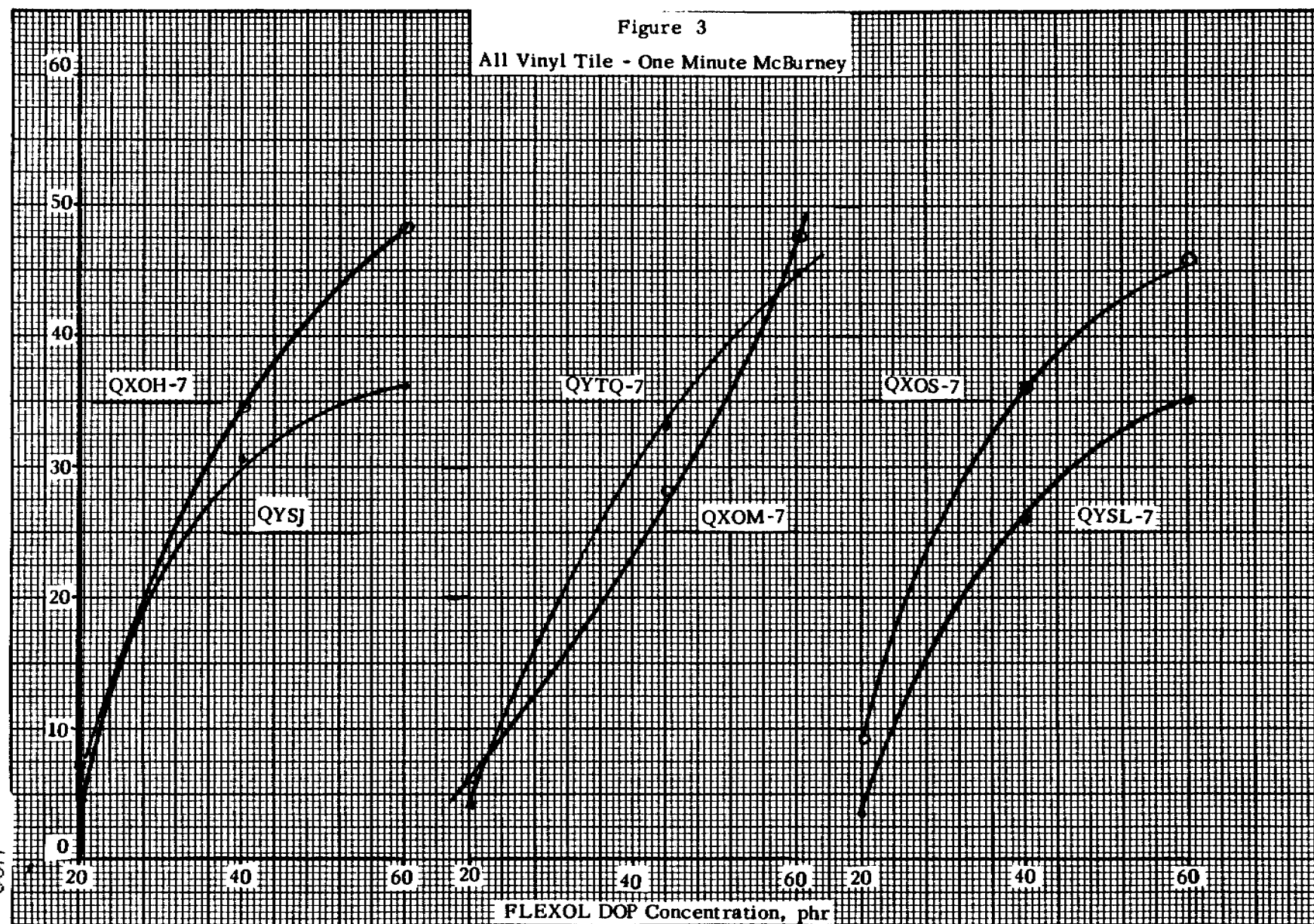


Figure 4

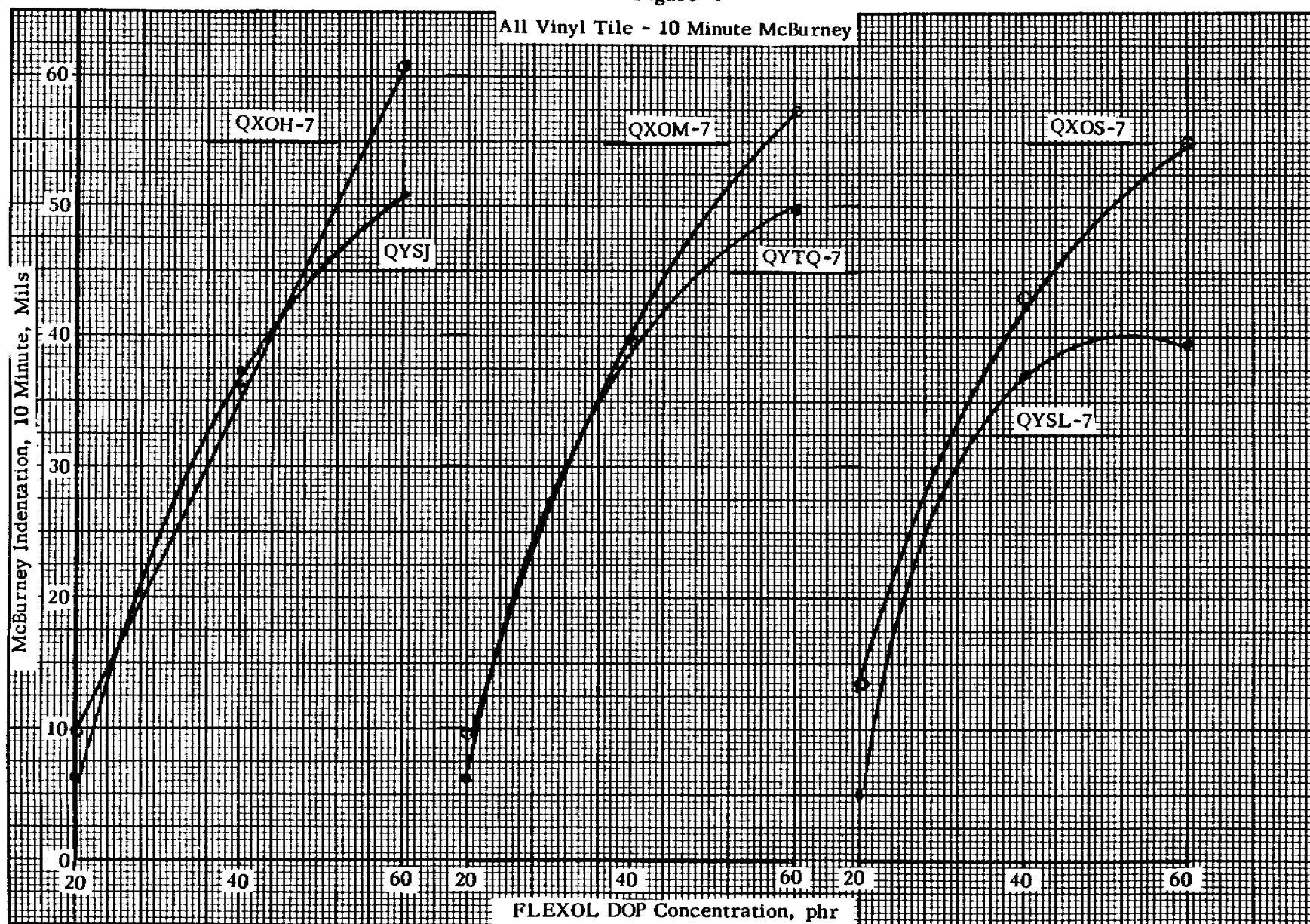
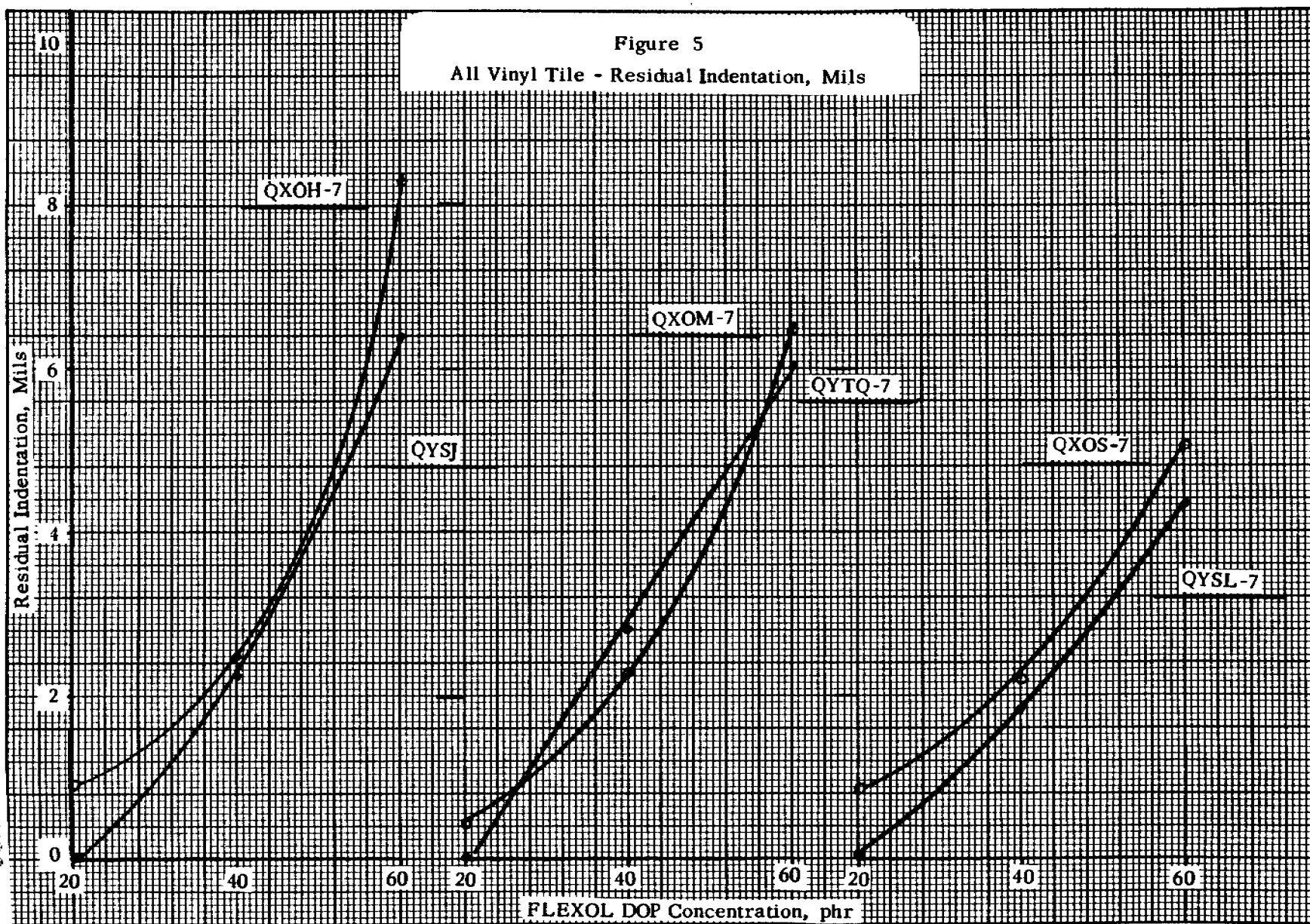


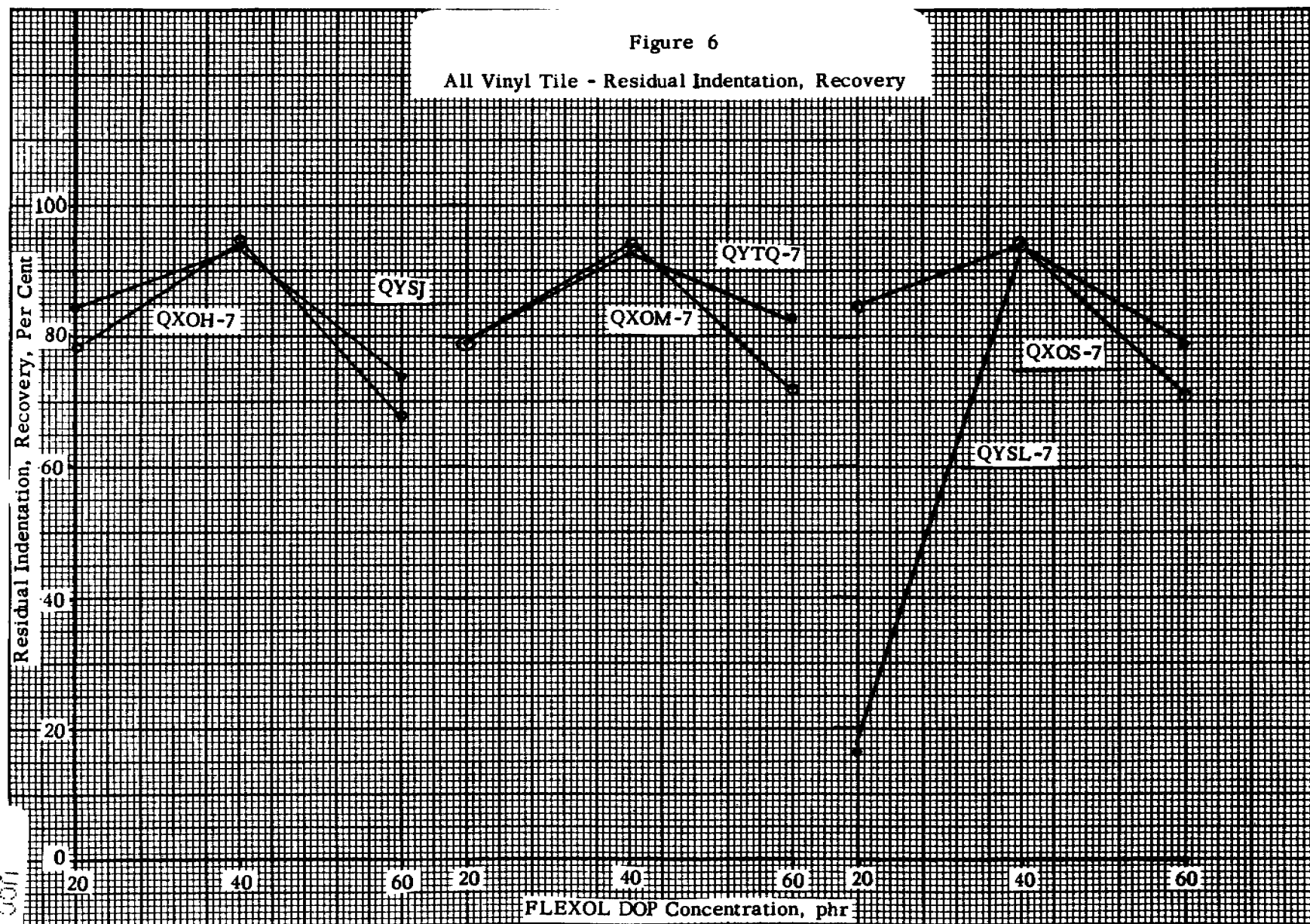
Figure 5
All Vinyl Tile - Residual Indentation, Mils



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Figure 6

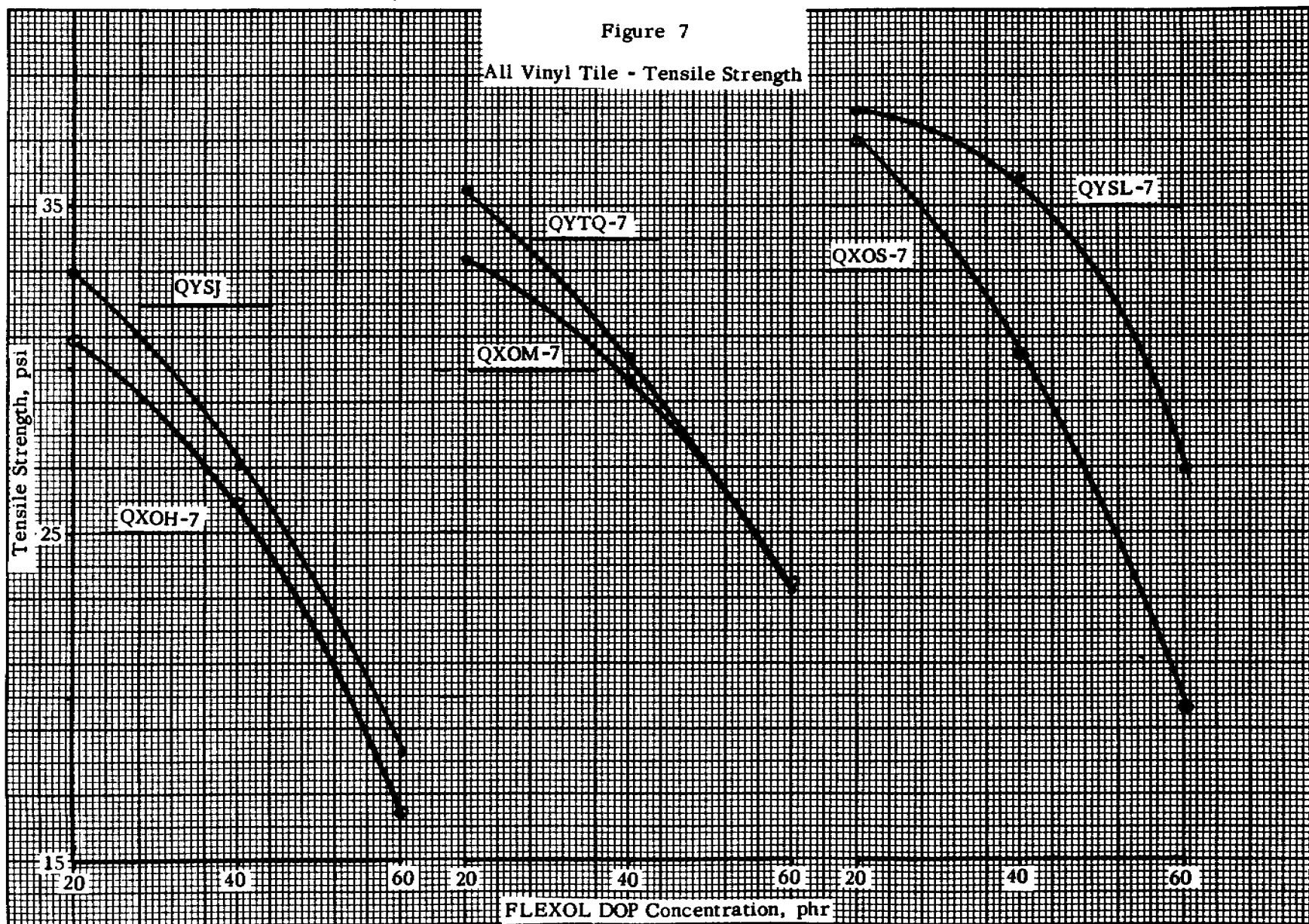
All Vinyl Tile - Residual Indentation, Recovery



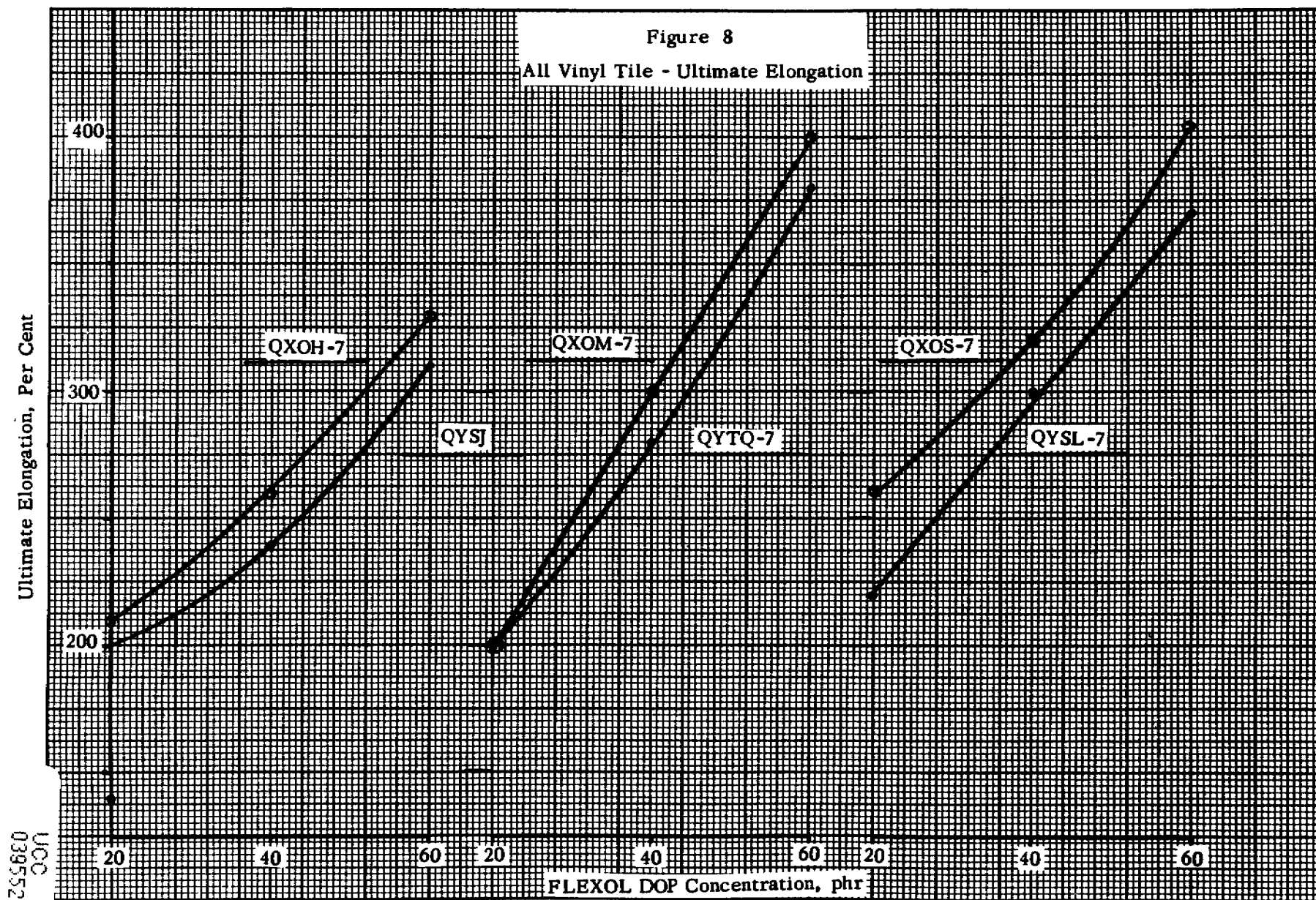
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Figure 7

All Vinyl Tile - Tensile Strength



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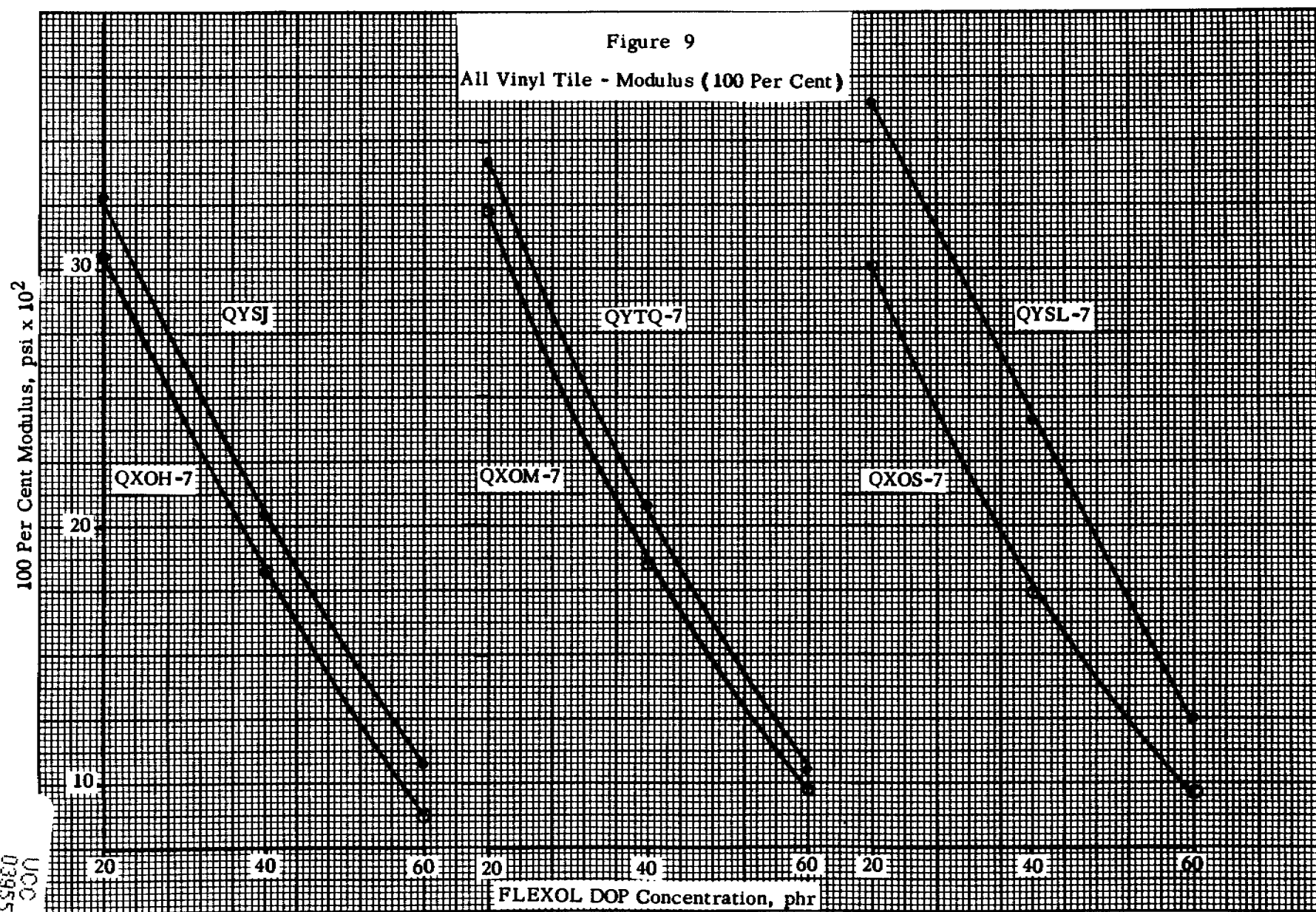
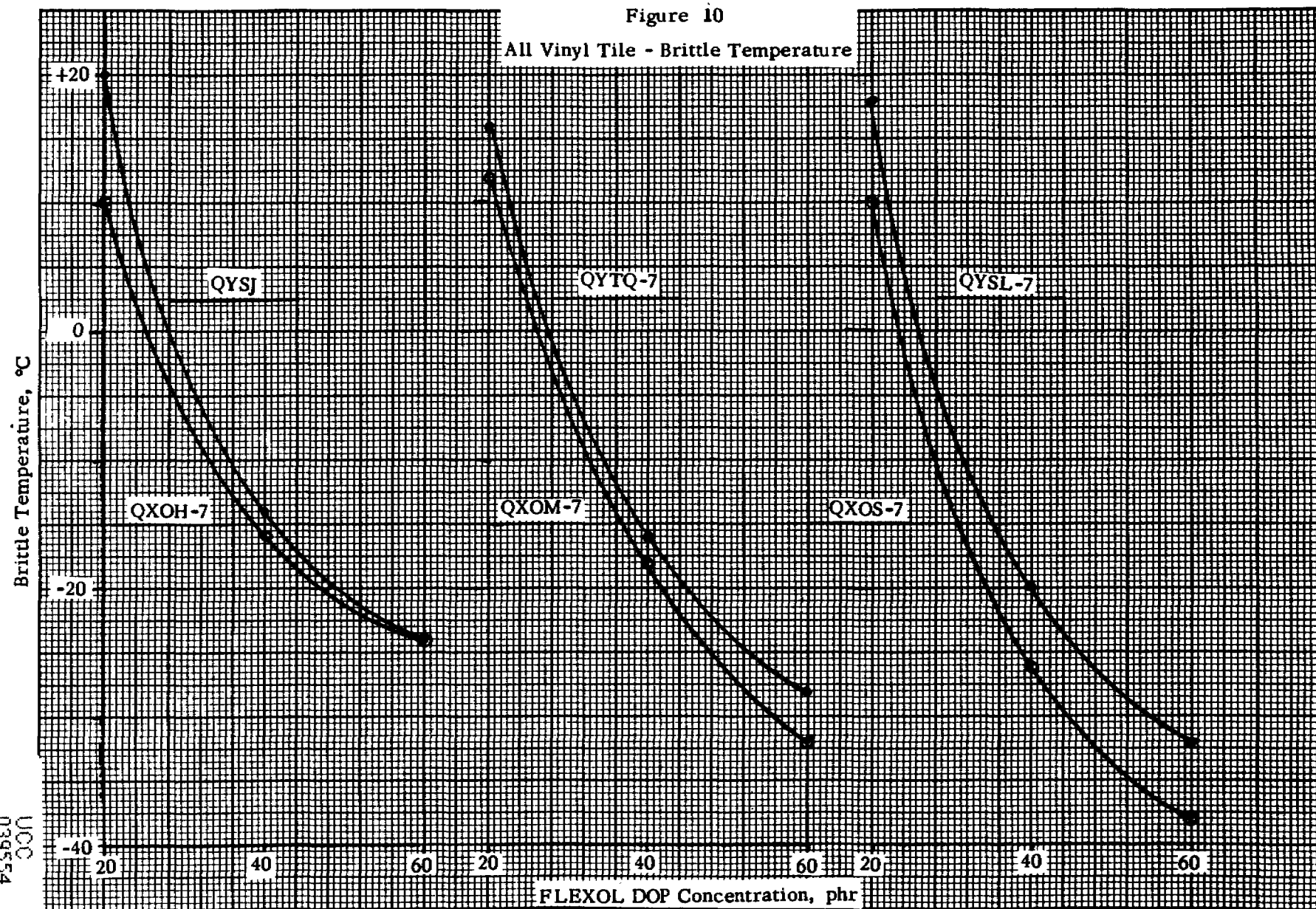


Figure 10

All Vinyl Tile - Brittle Temperature



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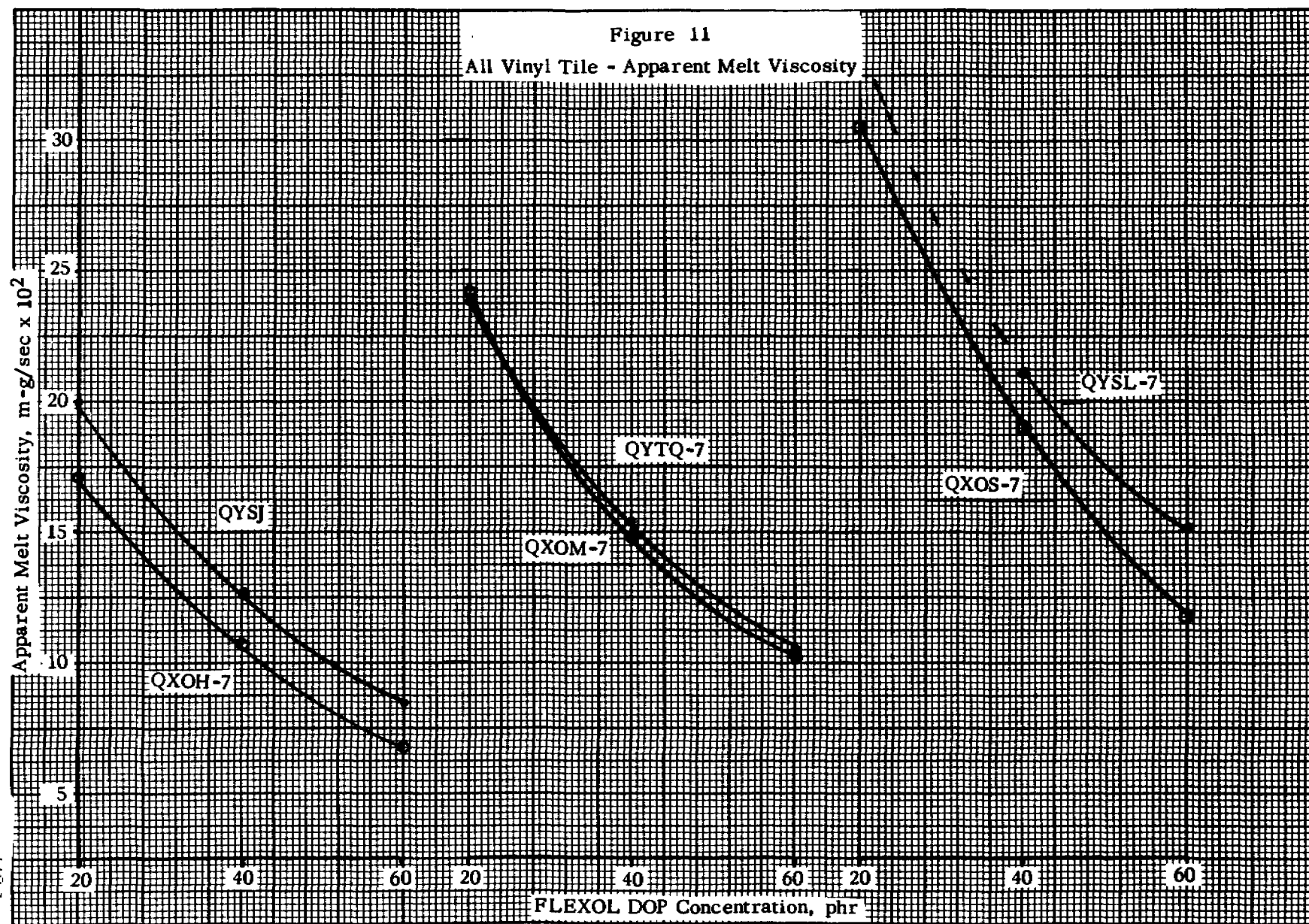


Figure 12
All Vinyl Tile - Fluxing Time

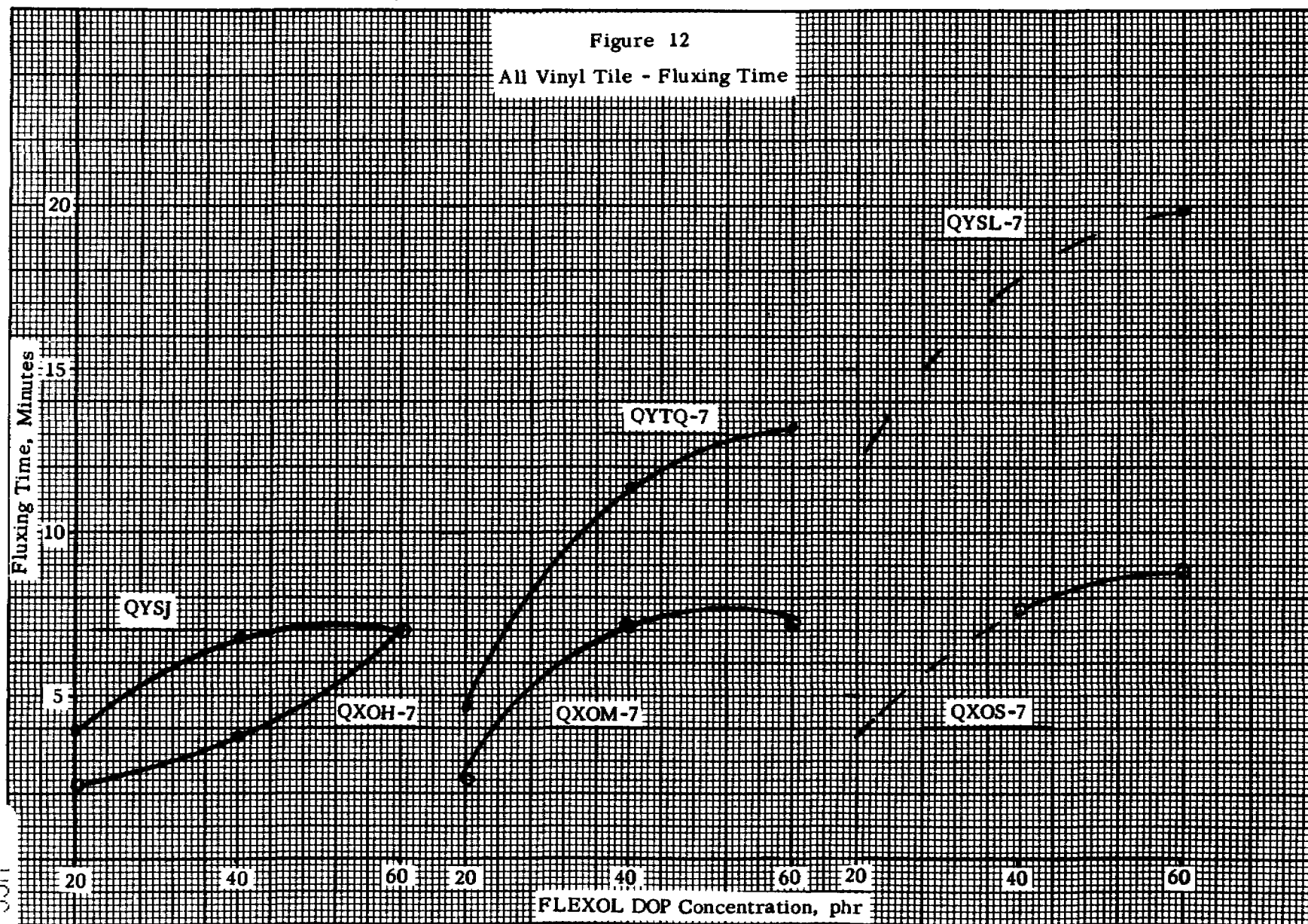
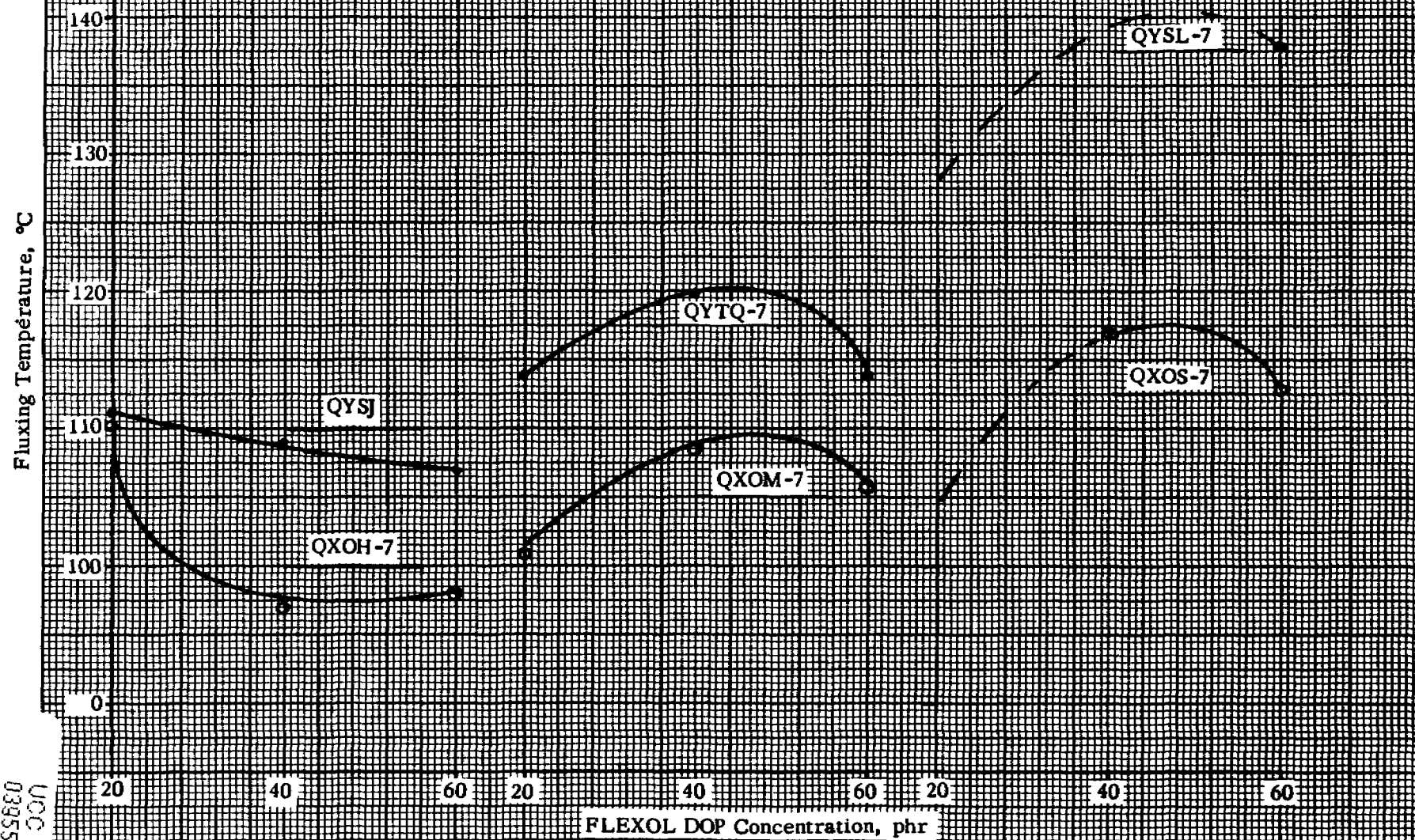


Figure 13

All Vinyl Tile - Fluxing Temperature



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Figure 14

All Vinyl Tile - Fluxing Torque

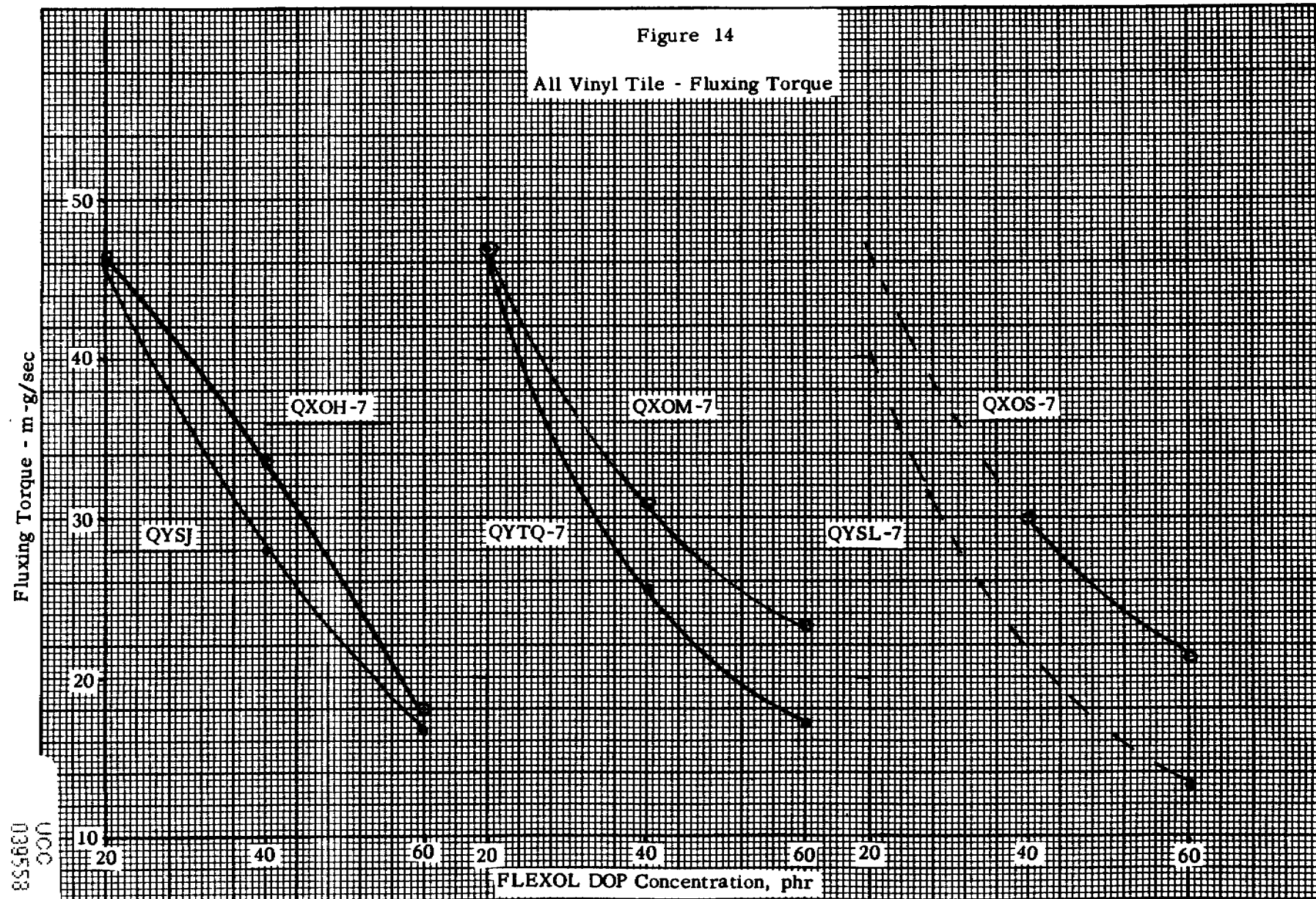


Figure 15
All Vinyl Tile - Physical Properties
at 50 Parts FLEXOL DOP Plasticizer

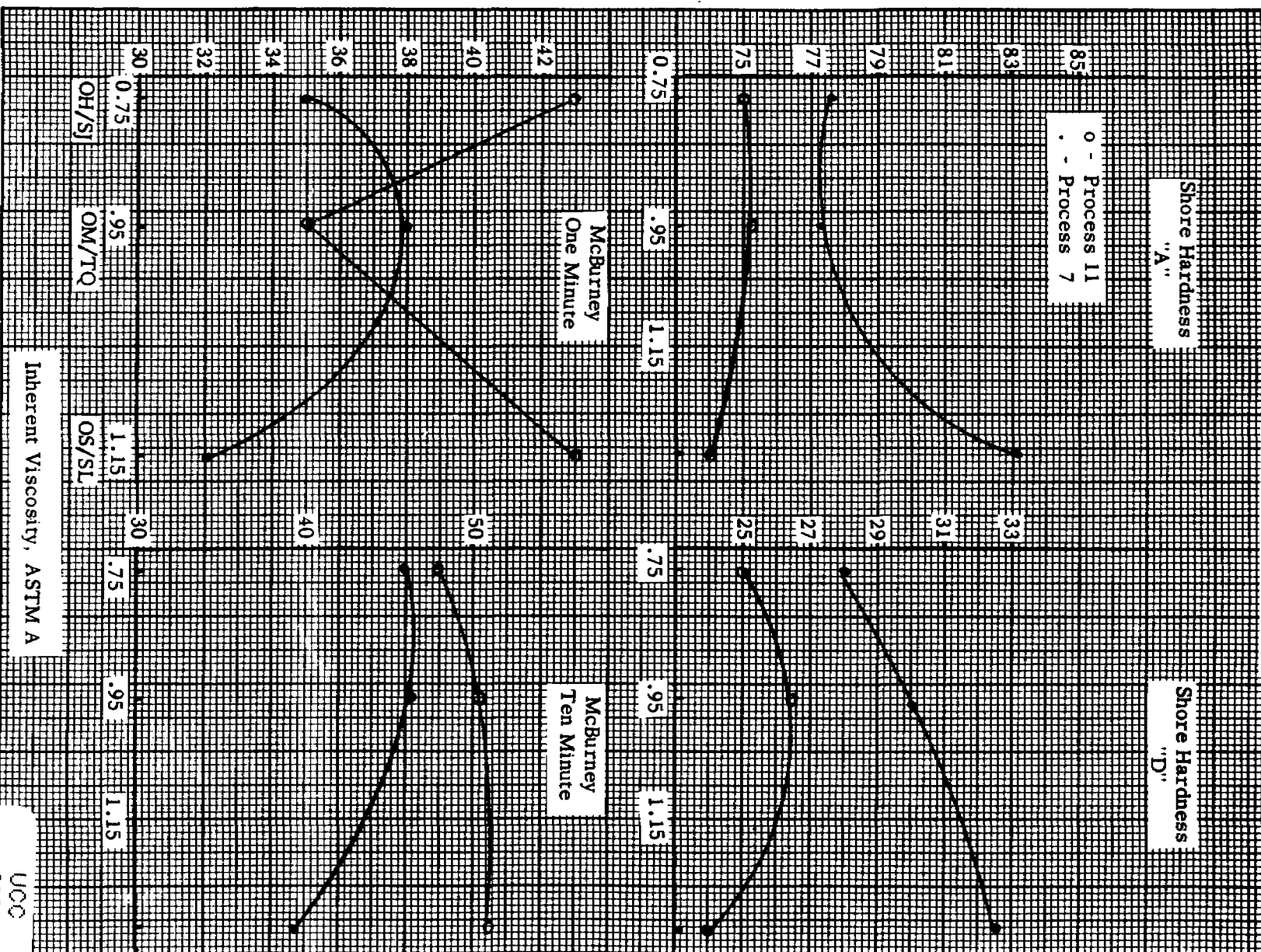


Figure 16

All Vinyl Tile - Physical Properties
at 50 Parts FLEXOL DOP Plasticizer

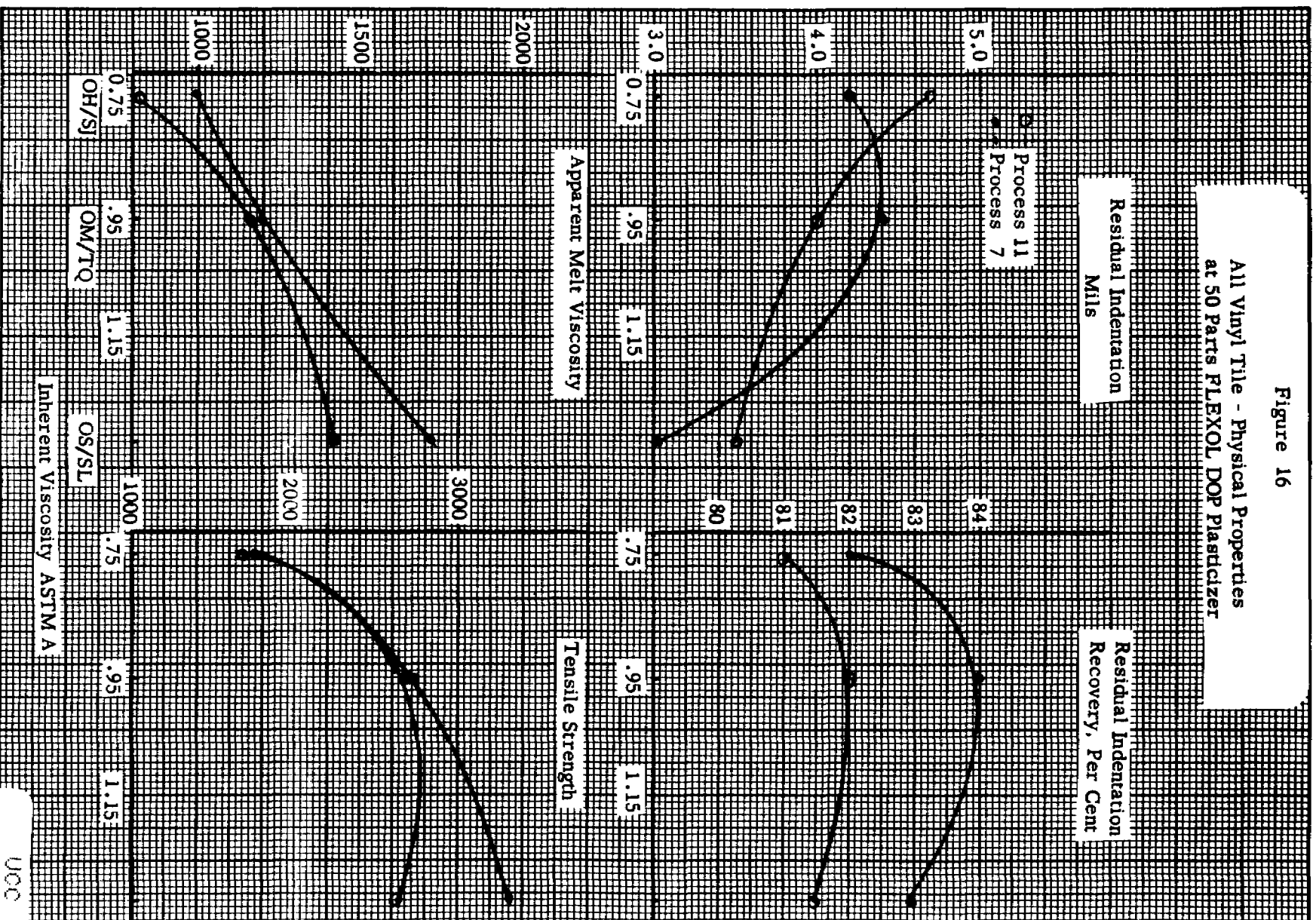
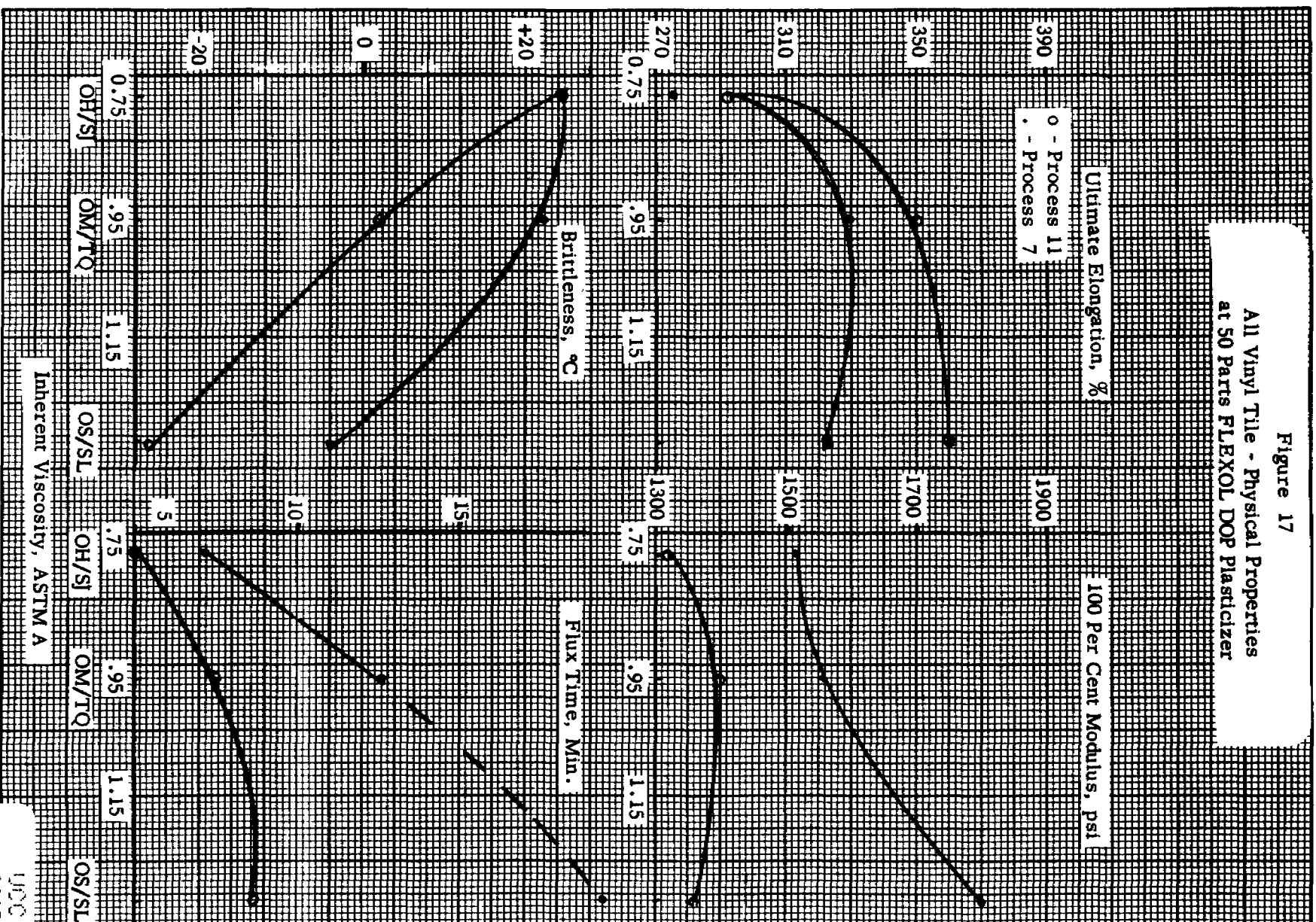


Figure 17
All Vinyl Tile - Physical Properties
at 50 Parts FLEXOL DOP Plasticizer



Notebook Reference: 443 - pages 23 - 34

Period Covered: March 21 to April 15, 1966

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