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TECHNICAL SERVICE REPORT

by

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Characterization of Vinyl Record
Preblends by Torque Rheometer
Techniques -- Part 2

for

THE RADIO CORPORATION
OF AMERICA
Indianapolis, Indiana

Union Carbide Corporation - Chemicals and Plastics
Research and Development Department
Tarrytown, New York

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CHARACTERIZATION OF VINYL RECORD PREBLEND
BY TORQUE RHEOMETER TECHNIQUES
PART 2

INTRODUCTION

This Technical Service Report is based on an extension of an earlier investigation(1) designed to assess critically the hot melt characteristics of two vinyl record preblends as provided by RCA. Both RCA and Union Carbide torque rheometer test methods are to be utilized. The results of the original investigation did not demonstrate sufficient differences as to warrant consideration as quality control test methods. A UCC method, WC-327-A1, Determination of the Apparent Melt Viscosity of Record Molding Resins, was indicated as the most effective method of the four that were examined. This report is based on test results obtained by slight modifications of WC-327-A1 in an attempt to obtain significant differences between the two RCA preblends.

EXPERIMENTAL

Prior to the receipt of additional quantities of the two RCA preblends for this study, the shear rate and test temperature of WC-327-A1 was varied with a single sample system (Figures 1 and 2). The results obtained from this investigation indicate that the 150° C. jacket temperature, over the various shear rates (rotor speeds), exhibits the least differences. The various shear rates at 130° and 140° C. jacket temperatures were selected to examine the performance of the two preblends. The 50 rpm rotor speed was also eliminated. The data are shown in Table I and illustrated in Figures 3 through 6. The initial fluxing data are, as expected by these procedures, quite erratic and insignificant and are included in Table I for academic interest only.

Apparent melt viscosity is normally defined by UCC as viscosity 15 minutes after peak torque and initial fluxing defined as the time to reach peak torque. Another parameter, final fluxing, is used in this investigation and is defined as the time, in seconds, for the torque curve to descend from peak torque to the point on the torque recording chart indicated by 200 chart units. This is the point at which the torque recorder may be switched consistently from the 5X to the 1X scale without adjusting the lever weight system. It is construed in this investigation as the point of final fluxing or an empirical definition of the point at which the compound is approaching complete homogeneity.

The data developed at the 30 rpm rotor speed and jacket temperature of 140° C. suggest more definite and consistent differences than at the other conditions. The two preblends were accordingly tested as follows:

- (1) Conkling, A. W., McCoy, P. T., Characterization of Vinyl Record Preblends by Torque Rheometer Techniques, Research and Development Department, Technical Service Report, November 14, 1967.

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	<u>Apparent Melt or Equilibrium Viscosity, m-g/sec.</u>	
	<u>A</u>	<u>B</u>
Simulated RCA Test (except 1:5 lever ratio)	2700	2760
Modified UCC WC-327-A1 50 gm (2)	2500	2610
Modified UCC WC-327-A1	2840 (57.6 g) (3)	2940 (57.9 g) (3)

(2) Specified by RCA

(3) Specified by UCC

The times to final fluxing for the latter two procedures follow:

	<u>Final Fluxing, sec.</u>	
	<u>A</u>	<u>B</u>
Modified WC-327-A1 50 gm.	48	60
Modified WC-327-A1	42	48

The test parameters relating to the simulated RCA method follow:

<u>Torque-Meter Grams Per Second</u>					
<u>Compound</u>	<u>Peak</u>	<u>2 Min.</u>	<u>At Suppression</u>	<u>After Suppression</u>	<u>7 Min.</u>
A	5800	4150	3050	3000	2700
B	5300	4150	3150	3070	2760

The band characteristics of the recorded torque at the 20 rpm rotor speed may be of significant interest to investigations of this type as illustrated in Figure 7. Note that each stroke of the pen is clearly distinguishable. This provides a more efficient means of accurately measuring band width which is a processing parameter that is considered by some investigators as quite significant and related to molecular weight distribution. The 20 rpm rotor speed is not considered applicable to studies on the Model PLV 33AA Plasti-Corder and earlier models, only on the Direct Current models such as the PLV-150 and later models.

RECOMMENDATIONS AND CONCLUSIONS

The examination of the influence of shear rate and test temperature on the processing characteristics of two vinyl record preblends in a torque rheometer suggests that a lower rotor speed is more sensitive to sample differences than those currently used by RCA and UCC. The reduction of the test temperature also provides more sensitivity but to a lesser degree than rotor speed. A test method based on these results has been prepared for consideration by the laboratories of UCC and by RCA, if desired. Comments as to the utility of the suggested method are solicited.

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Notebook Reference:

Book 693

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Attachments:

1 Table
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1 Suggested Test Method

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APPENDIX I

SUGGESTED TEST METHOD

DETERMINATION OF THE APPARENT MELT VISCOSITY OF RECORD MOLDING RESINS

1. SCOPE

A formulation and procedure for determining the apparent melt viscosity of record molding resins is described. The method is specifically designed to define the vinyl resin requirements for the Radio Corporation of America, only.

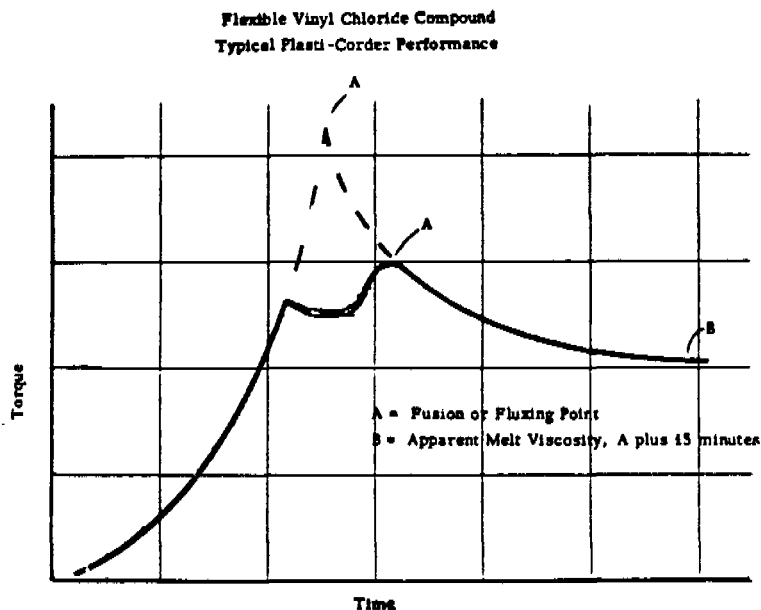
2. DEFINITIONS

2a Fusion or Fluxing Point

The point at which the sample becomes molten and torque begins to decline. In Fig. 1, A₁ identifies the fusion or fluxing point of compounds which flux quickly while A₂ identifies the fusion or fluxing point of compounds which flux more gradually.

2b Apparent Melt Viscosity

The term used to describe the dynamic equilibrium melt consistency of vinyl compounds under specified shear stress, temperature, and time as measured with a torque rheometer. The test results, in terms of equilibrium temperature, time, and torque, are recorded in meter-grams per second (a horse-power parameter). The apparent melt viscosity, shown as point B in Fig. 1 is taken 15 minutes after the fluxing point, i. e., B = A plus 15 minutes.



CAUTION - a torque level of 8000 meter grams per second should not be exceeded, otherwise equipment damage could result.

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3. APPARATUS

- 3a C. W. Brabender Plasti-Corder
- 3b Oil circulating bath and suitable silicone-type oil.
- 3c Roller mixer head, number 5, with provisions for measuring and recording jacket and stock temperatures.
- 3d Quick load chute and a 5 kg. weight available from C. W. Brabender Instruments Incorporated.
- 3e Balance capable of weighing 500 ± 0.1 gram.
- 3f Suitable analytical balance.
- 3g Weighing dish 0.5 x 3 x 5 inches constructed of heavy gauge aluminum foil, open at one end.
- 3h Spatula, brush of brass or copper composition.
- 3i One-inch paint brush.

4. TEST MATERIALS

- 4a A sample of the resin to be tested.
- 4b Ferro 13V57 (magnesium-modified lead stearate in powder form) from the Ferro Chemical Company.

5. PROCEDURE

5a Preliminary Instrument Preparations

- 5aa Clean the chamber and rotors thoroughly; place several drops of FLEXOL DOP between the rotors and fixed mixer walls and remove the excess with an air jet and cloth. Brass shim stock is useful in removing particles of solid materials that are difficult to dislodge.
- 5ab Adjust the jacket temperature to 160°C . for calibration.
- 5ac Calibrate the roller mixer head in accordance with the C. W. Brabender Standard Calibration Procedure, Roller Style Measuring Head, Procedure Number 150/Union Carbide Corporation, Plastics Division, Standard Testing Method WC-99-F.
- 5ad Compare the resultant calibration curve with the calibration curve provided by the manufacturer at the time of purchase of the instrument. A deviation greater than ± 35 units from the standard calibration curve will necessitate a thorough check of the instrument as to zero point, jacket temperature, rotor binding, bearing wear, etc. The manufacturer should be contacted if corrective measures fail.
- 5ae Calibrate the instrument once a week with continuous use or twice a month with intermittent use.

5b Sample Preparation

- 5ba Prepare the test compound as follows:

Formulation

Resin	100 parts (57.03 grams)
Ferro 13V57	1.6 phr (0.91 grams)

The required amount of sample in grams is determined according to the following equation which takes into account the average specific gravity of the compound and the volume of the roller mixer head:

$$\text{Average specific gravity of the compound} \times 42.7 = \text{quantity of sample in grams}$$

Thus, the melt characteristics are defined using a constant volume of the compounds being tested.

Example:

<u>Ingredient</u>	<u>Parts</u>	<u>Grams</u>	<u>% of Compound</u>	<u>Specific Gravity of Ingredient</u>	<u>phr/ Specific Gravity</u>
Resin	100	57.03	98.43	1.36	73.53
Ferro	1.6	.91	1.57	1.21	<u>1.32</u>
13V57					74.85

$$\text{Average specific gravity of compound} = \frac{101.6}{74.85} = 1.357$$

$$1.357 \times 42.7 = 57.94 = \text{required quantity of sample in grams.}$$

5bb Resin and stabilizer in the amounts determined according to 5ba are placed in an aluminum weighing dish and are mixed thoroughly with a spatula for five minutes.

5c Testing Procedure

- 5ca Adjust the jacket temperature of the roller mixer head to 140° C. one-half hour prior to sample evaluation. Start the mixer rotors at approximately 30 rpm at the same time.
- 5cb Set the Plasti-Corder scale setting on X1 and the sensitivity lever ratio at 1:5 during the one-half hour conditioning period. Adjust the sampler mechanism setting to zero. Insure proper operation of the torque recording chart and pen at 50 mm./min. Adjust the torque indicating scale and chart recording pen to zero scale reading.
- 5cc Set the scale reading on X5 and introduce about 60 grams of a suitable purge compound (BAKELITE compound VFD 9400 is satisfactory) into the chamber of the roller-mixer head. Permit the compound to mix for about ten minutes and adjust rotor speed to 30 rpm by visual count during this time. Remove the purge compound, then clean the chamber and rotors with a brass spatula/brush only. Place several drops of FLEXOL DOP behind the rotors; remove any excess and reassemble the mixer head. Insure that residual compound does not remain between the rotors and stationary mixer wall.
- 5cd Allow the mixer head and rotors to equilibrate with rotors running for ten minutes. During this period, set the scale setting on X1 and, if necessary, readjust the scale setting to zero; then reset to X5.

- 5ce Start the torque recording chart at the end of the nine minute reading of the ten minute preheat period. Place the quick load chute, minus the ram assembly, into the throat of the roller mixer head at a reading of 7.5 minutes on the torque chart.
- 5cf Turn on the temperature recording instrument and introduce the test sample into the quick load chute at zero time on the torque recording chart with the rotors running. Place the quick load chute ram loaded with 5 kg. weight into the cavity of the quick load chute and firmly but slowly force the test sample into the chamber of the roller mixer head. This operation normally requires less than one minute.
- 5cg Remove the quick load chute assembly from the throat of the roller mixer head about one-half minute after peak torque or fluxing point has been reached, then lower the roller mixer head ram assembly. Cool the quick load chute assembly to room temperature before using again.

CAUTION: Peak torque will not always occur at the end of one minute, or the end of the sample loading operation, for stiffer compounds; immediate fluxing is often encountered for softer compounds at this point. Testing experience greatly aids the analyst in this judgment.

- 5ch Adjust the scale setting from 5X at a chart consistency reading of 200 to X1. An adjustment of the sliding lever weight is not necessary. Remove suppression of 1.0 when required, while on X1 setting. Continue the test to a period of at least 15 minutes past the fluxing point.
- 5ci Stop the rotors and recording charts; disassemble the roller mixer head and remove as much of the test compound as possible. Start the rotors and remove any remaining compound from the holes in the ends of the rotors.
- 5cj Reassemble the roller mixer head, turn the rotors on, set the scale on X5, and repeat Sections 5cc through 5cj for subsequent sample evaluation.
- 5ck Remove the torque and temperature charts.

6. REPORT

- 6a Resin identification.
- 6b Time and temperature at 5X to 1X scale change.
- 6c Apparent Melt Viscosity - torque and temperature 15 minutes after fluxing point.
- 6d Any unusual observations.
- 6e Room temperature (thermometer on Plasti-Corder Scale Head).
- 6f Analyst
- 6g Date.

TABLE I

RCA RECORD PREBLEND TORQUE-RHEOMETER CHARACTERISTICS

<u>Jacket Temperature, °C.</u>	<u>Rotor Speed, rpm.</u>	<u>Compound</u>	<u>Initial Fluxing</u>			<u>Final Fluxing, sec.</u>	<u>Apparent Melt Viscosity</u>	
			<u>Seconds</u>	<u>°C.</u>	<u>Torque</u>		<u>m-g/sec.</u>	<u>°C.</u>
130	20	A	54	118	6400	72	3500	142
		B	48	121	6200	60	3600	143
	30	A	36	116	6850	48	3070	148
		B	30	120	6700	54	3220	149
	40	A	36	114	7500	48	2780	151
		B	18	118	7250	42	2780	152
140	20	A	36	122	5500	96	2970	148
		B	24	117	5450	108	3130	147
	30	A	24	119	6300	78	2650	152
		B	36	124	6000	102	2780	153
	40	A	18	126	6700	66	2440	158
		B	36	124	6400	84	2490	159

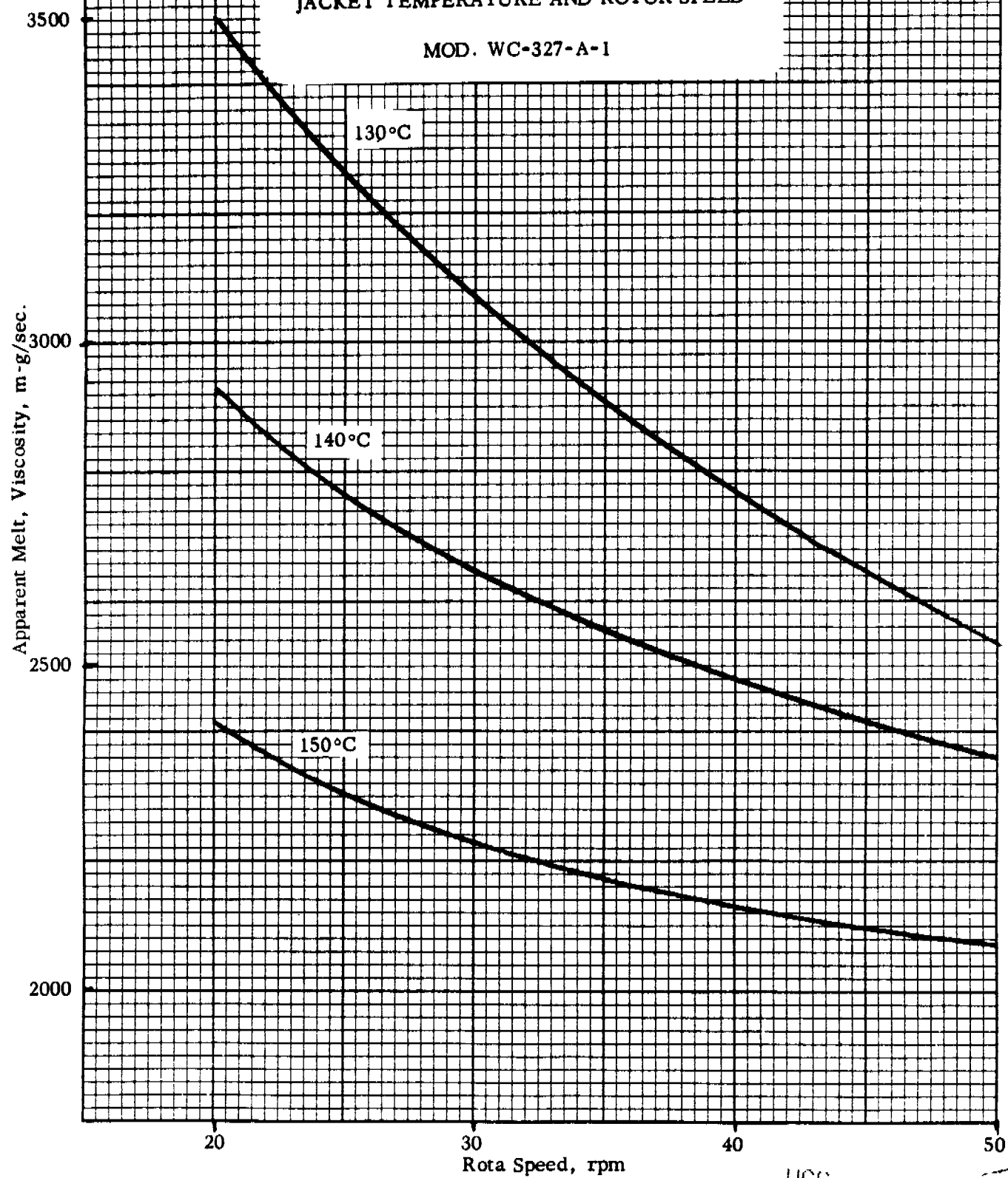
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FIGURE 1

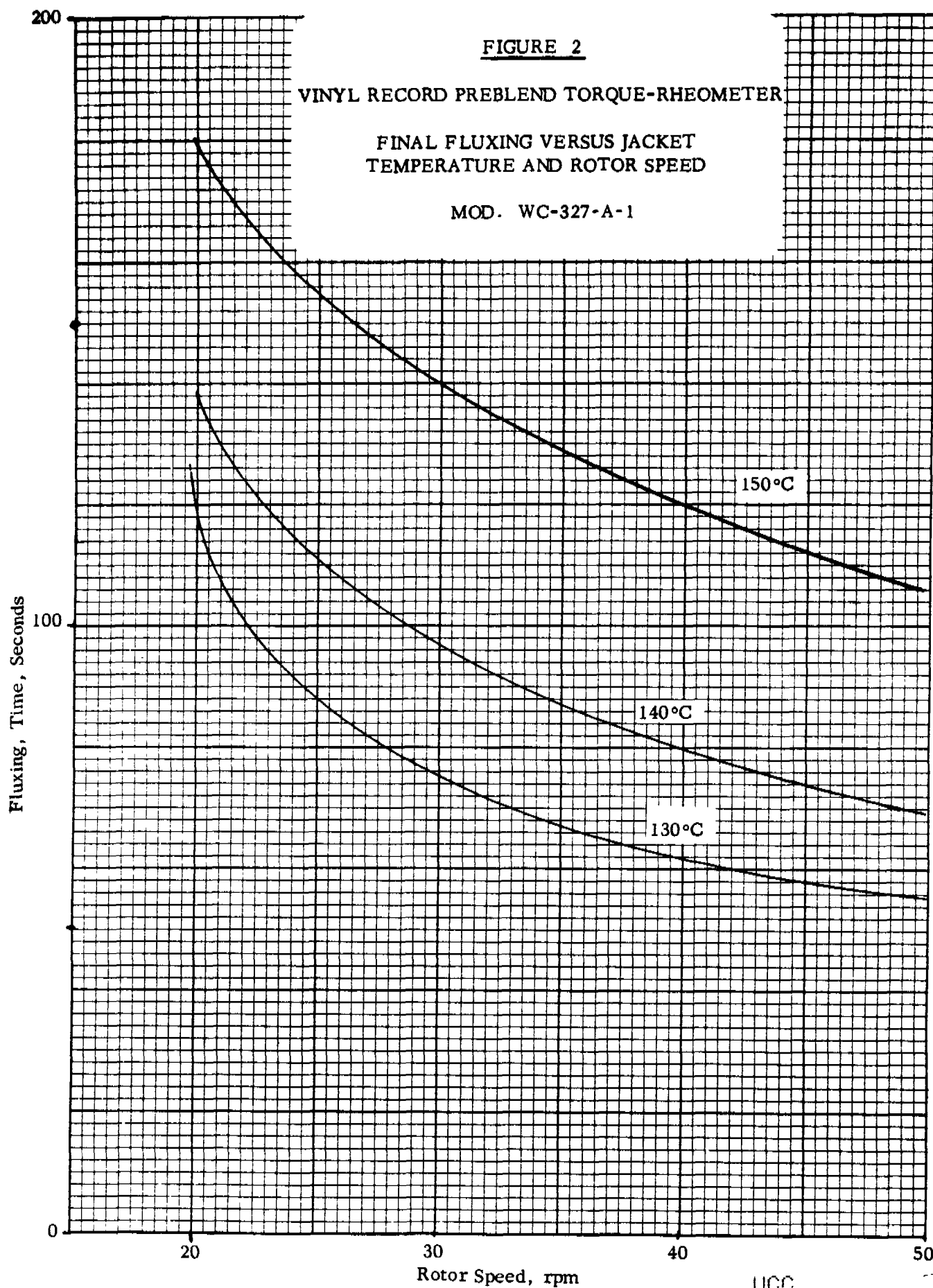
VINYL RECORD PREBLEND TORQUE-RHEOMETER

APPARENT MELT VISCOSITY VERSUS
JACKET TEMPERATURE AND ROTOR SPEED

MOD. WC-327-A-1



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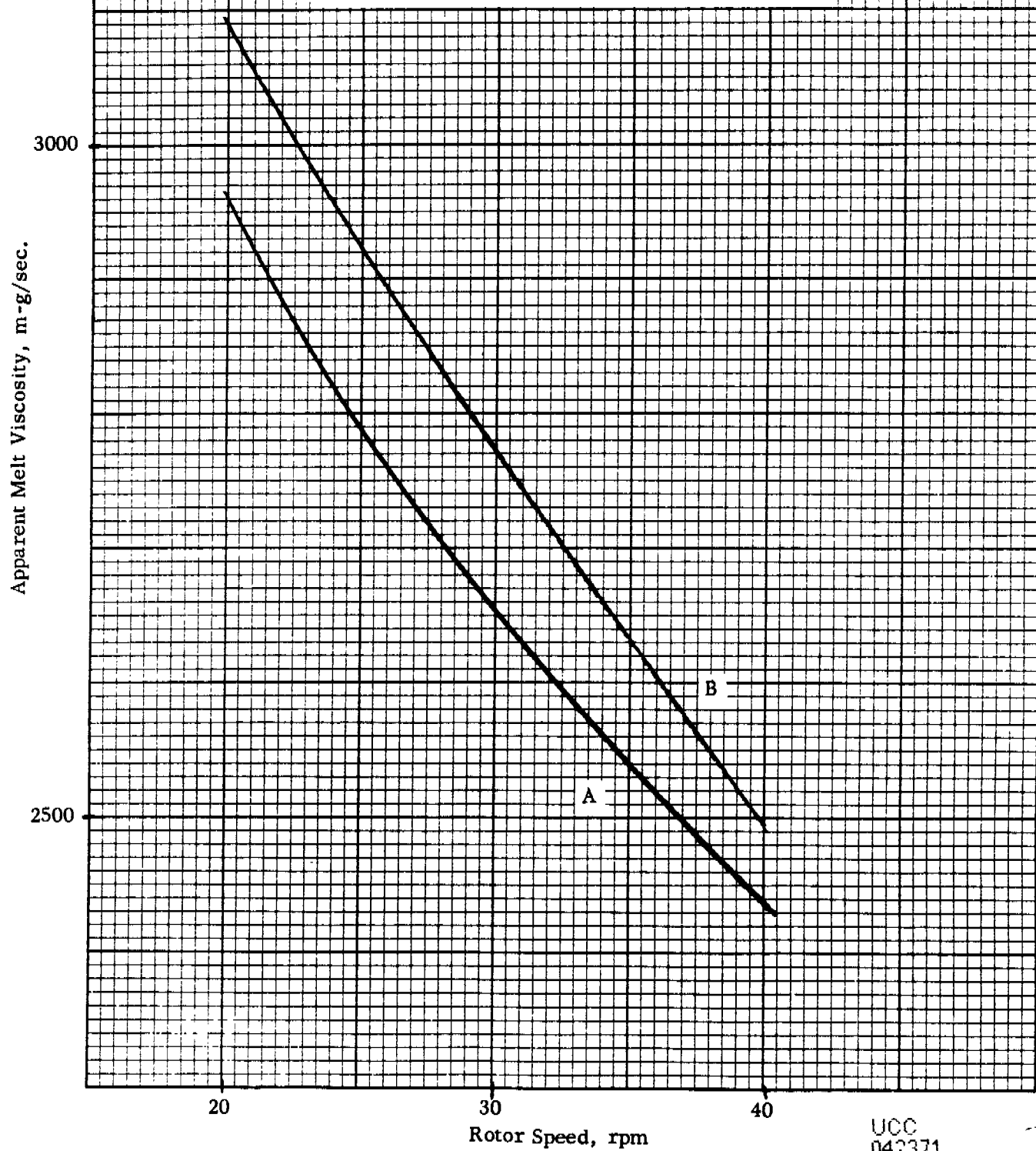
FIGURE 3

RCA RECORD PREBLEND
TORQUE-RHEOMETER

APPARENT MELT VISCOSITY VERSUS
ROTOR SPEED

JACKET TEMPERATURE 140° C.

MOD. WC-327-A-1



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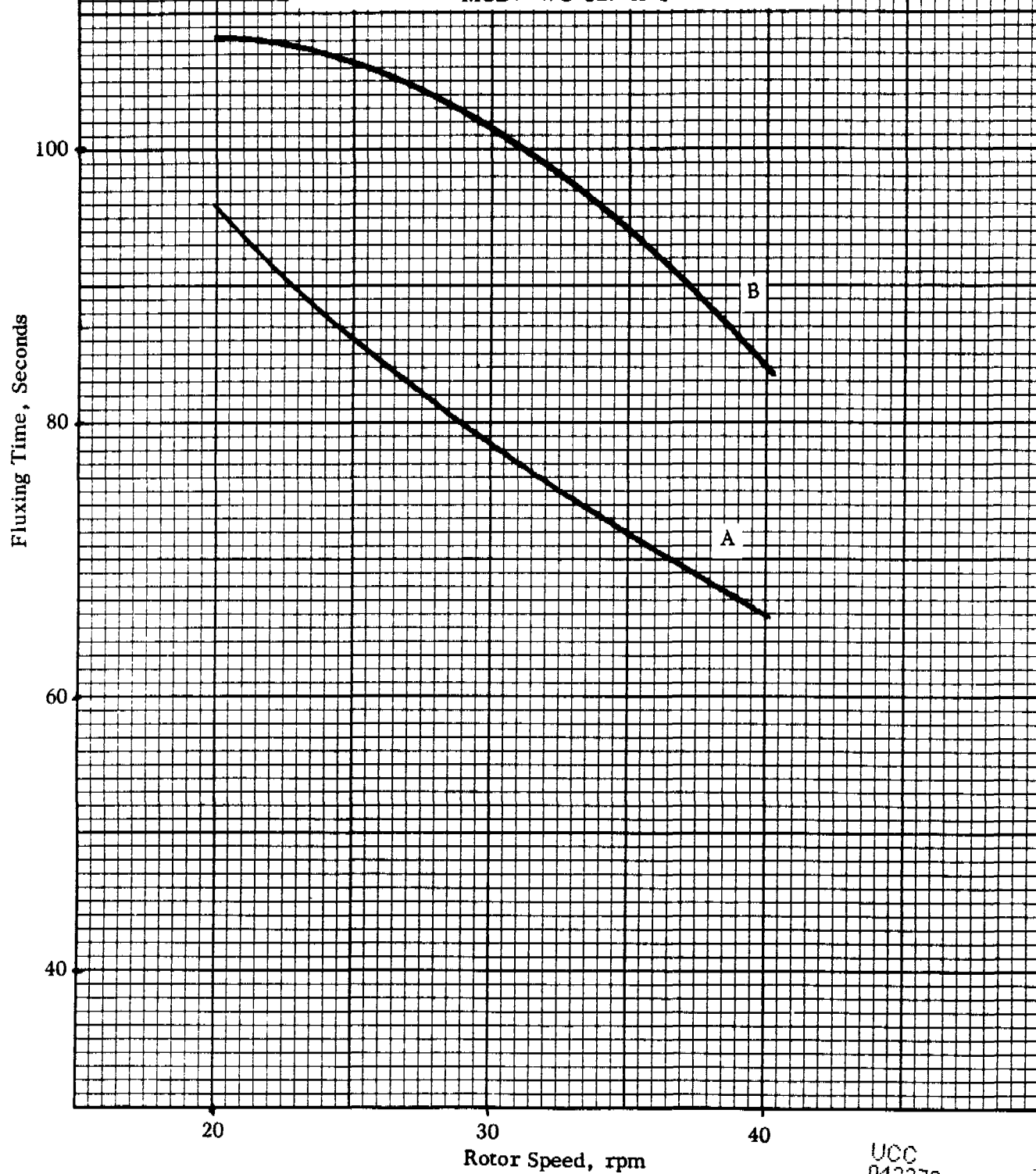
FIGURE 4

RCA RECORD PREBLEND
TORQUE-RHEOMETER

FINAL FLUXING TIME VERSUS
ROTOR SPEED

JACKET TEMPERATURE 140° C.

MOD. WC-327-A-1



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FIGURE 5

RCA RECORD PREBLEND
TORQUE-RHEOMETER

APPARENT MELT VISCOSITY VERSUS
ROTOR SPEED

JACKET TEMPERATURE 130° C.

MOD. WC-327-A-1

Apparent Melt Viscosity, m-g/sec.

3500

3000

20

30
Rotor Speed, rpm

40

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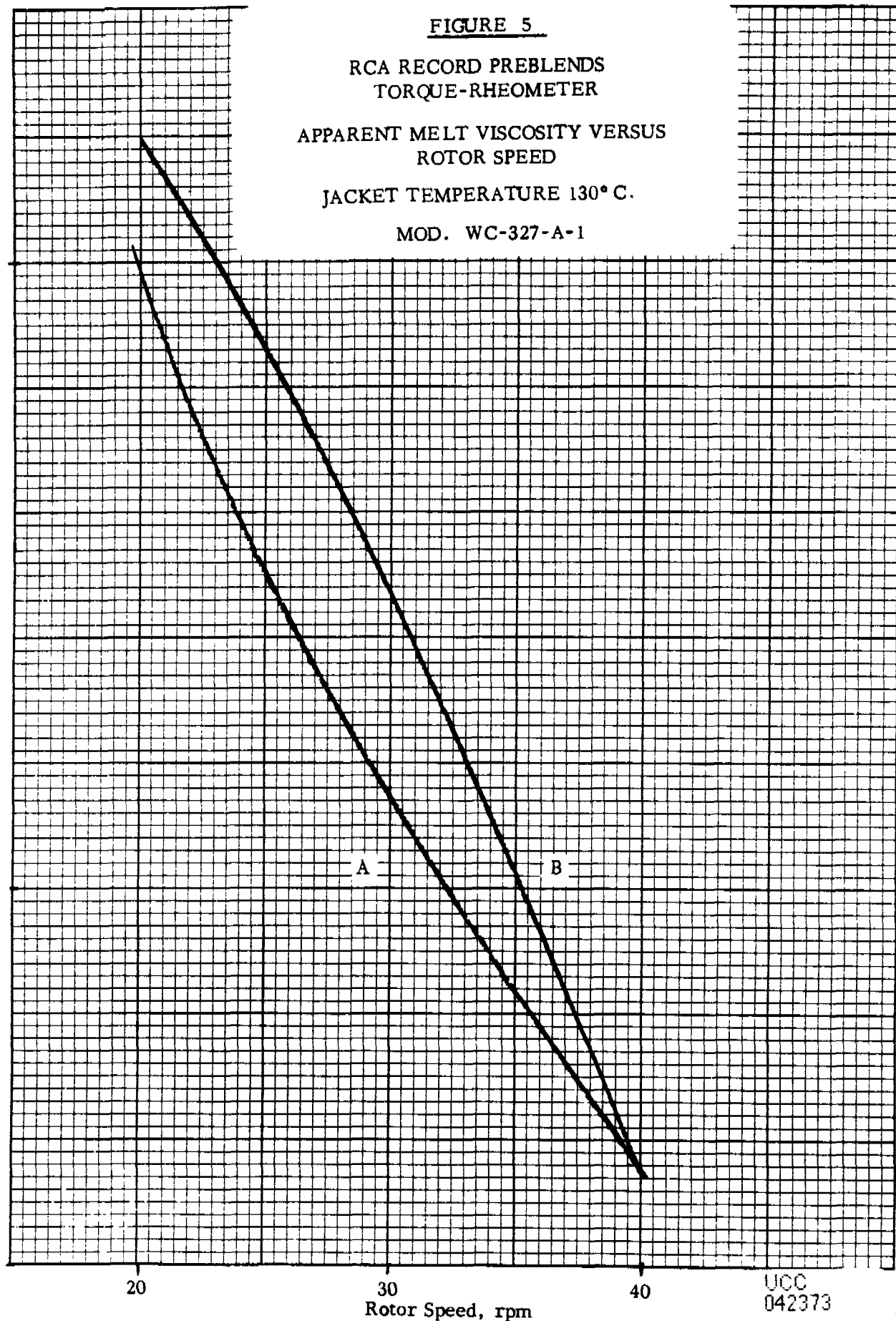


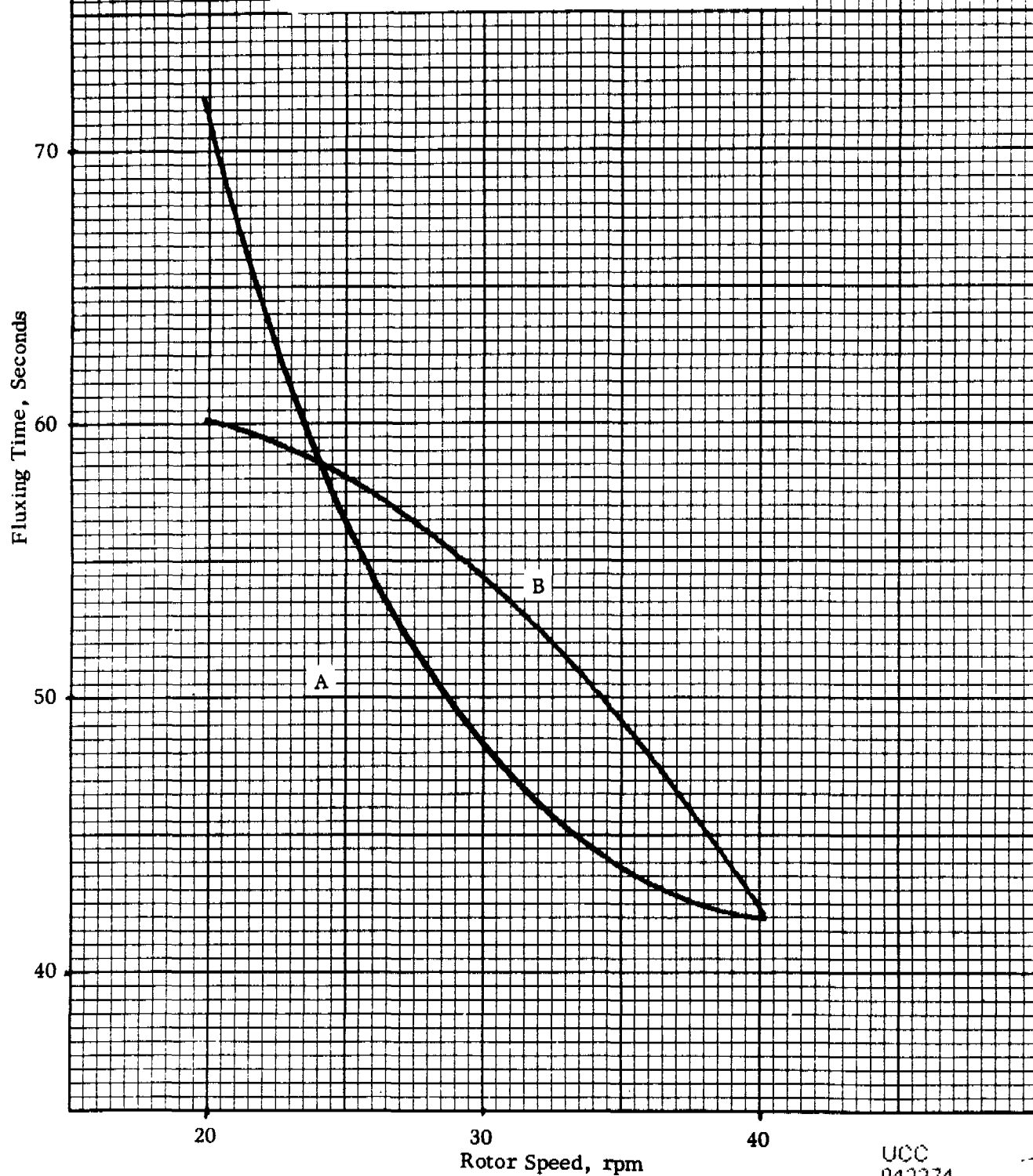
FIGURE 6

RCA RECORD PREBLEND
TORQUE-RHEOMETER

FINAL FLUXING TIME VERSUS
ROTOR SPEED

JACKET TEMPERATURE 130° C.

MOD. WC-327-A-1



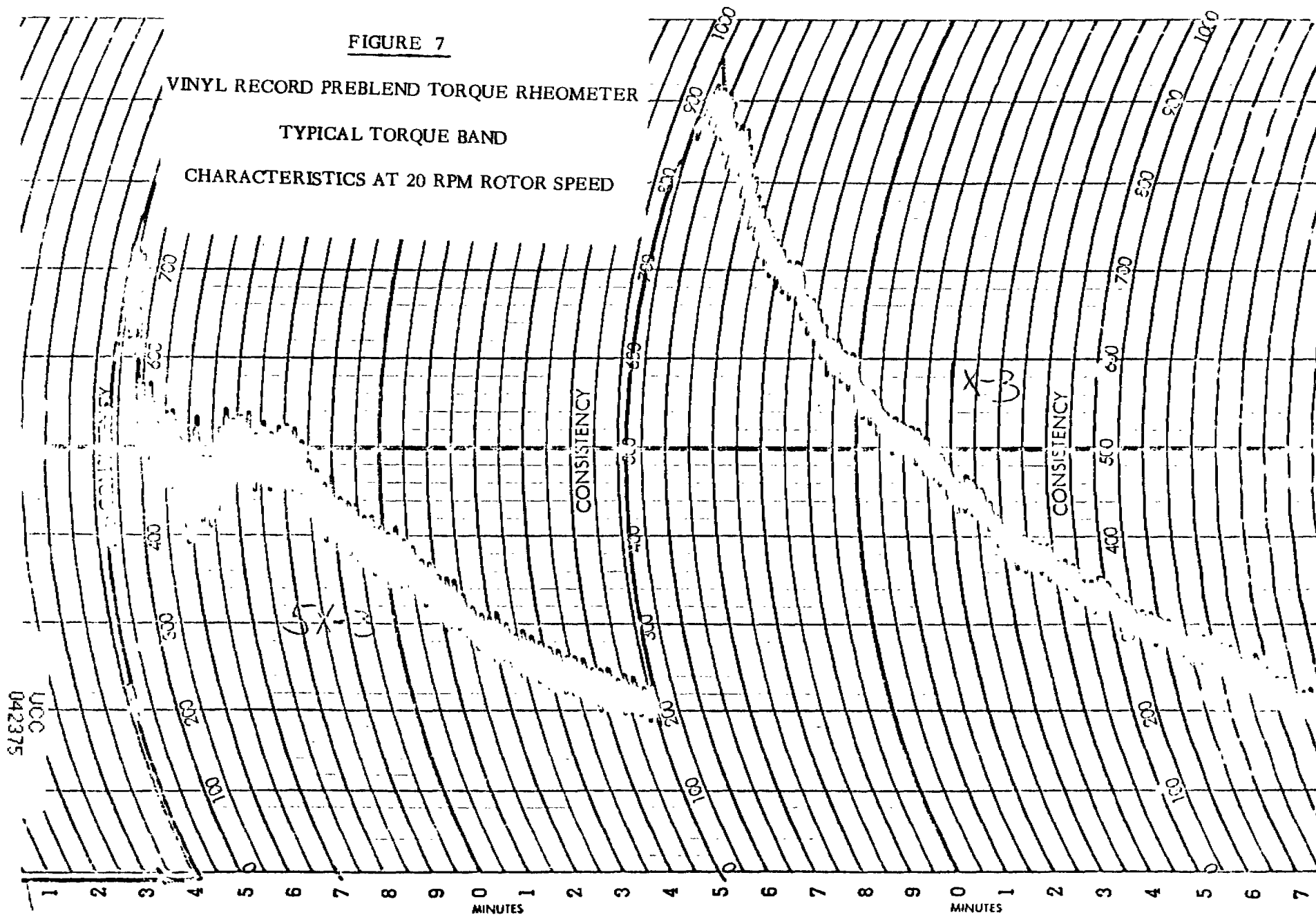
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FIGURE 7

VINYL RECORD PREBLEND TORQUE RHEOMETER

TYPICAL TORQUE BAND

CHARACTERISTICS AT 20 RPM ROTOR SPEED



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