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RISE-RATE TESTING OF URETHANE FOAMS

PDO 6984693, Final Report

T. E. Neet, Project Leader

Internal Distribution July 1973

Prepared for the
United States Atomic Energy Commission
Under Contract Number AT(29-1)-613 USAEC

Bendix Kansas City Division

DISCLAIMER

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Project Leader:
T. E. Neet
Department 814

PDO 6984693
Final Report

Technical Communications Bendix Kansas City Division

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RISE-RATE TESTING OF URETHANE FOAMS

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Prepared by T. E. Neet, D/814, under PDO 6984693

The effects of variations in processing parameters and material formulations on the rise-rate characteristics and physical properties of rigid urethane foams were evaluated. Of the eight variables studied, only the component temperature, mix ratio, water concentration, and material reactivity significantly altered the rise-rate results. The physical properties of the foams were not appreciably changed.

THE BENDIX CORPORATION

KANSAS CITY DIVISION

KANSAS CITY, MISSOURI

A prime contractor for the
Atomic Energy Commission
Contract Number AT(29-1)-613 USAEC

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CONTENTS

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ILLUSTRATIONS

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TABLES

SUMMARY

This study was undertaken to determine the effect of various material and processing variables on the rise-rate characteristics, gel time, and physical properties of rigid urethane foams. Of the eight variables studied--component temperature, mix time, mix speed, water concentration, mix ratio, mix quantity, pour weight, and material reactivity--only the component temperature, water concentration, mix ratio, and material reactivity had any significant effect on the evaluated properties of the materials. While the exact effect of these variables on all foam systems is not predictable from this work, the trends have been established and all similar materials should conform to these patterns.

The material reactivity, or catalyst concentration, was the most important variable affecting the rise-rate characteristics and gel times of the foams. However, not all of the foam systems are equally affected by equivalent changes in catalyst concentration. In any case, rise-rate testing may provide an efficient tool for checking the undefined catalyst level in urethane foams. The catalyst concentration and three other variables caused the gel time to change as the material reactivity changed. However, the gel time shift caused by catalyst concentration, did not change at the same rate as the rise-rate characteristics (the time-to-maximum velocity and the rise time). This indicates that the rise-to-gel ratio of foams can be significantly altered by proper catalyst and catalyst level selection.

The component temperature was the second most important variable evaluated. To obtain reproducible rise-rate results, the component temperatures must be maintained within ±2°F (1°C) of their specified values. Also, while changes in component temperature altered the gel times of the foams, these gel time changes were paralleled by changes in the rise-rate characteristics. This variable, then, could not be used for adjusting the rise-to-gel ratio.

The mix ratio, while a more significant variable than the water concentration, is not as influential as the component temperature or material reactivity in its effect on the rise-rate and gel-time properties of the foams. Present material specifications usually instruct that the mix ratios be maintained within ±1 percent of the indicated values. This percentage normally refers to the mix weights. The actual mix ratio variations possible are less than the changes represented by the levels in the study. Thus, present material specifications should provide adequate control over mix ratios for rise-rate testing purposes. As was the case with component temperature, the gel time changes were matched by changes in the rise-rate characteristics. This should eliminate mix ratio adjustments as a control for the rise-to-gel ratio.

The water concentration of the resin was the least effective of the four variables in altering the rise-rate characteristics and gel times of the foams. Present materials specifications provide sufficient control over the water content to give reproducible rise-rate results. Rise-rate testing can only detect resin-water concentrations considerably outside the established limits. The effect on the gel time is also minimal.

The compressive strengths of the foams were not significantly affected by six of the eight variables. Only changes in the mix ratio and component temperatures caused the compressive strength to change. As the mix ratio decreased, the compressive strength decreased because of the less-rigid polymer created by the lower isocyanate index. A low component temperature of 60°F (15°C) drastically lowered the compressive strength of the foam at the 325°F (162°C) test temperature. Further study of this temperature effect may be warranted.

DISCUSSION

SCOPE AND PURPOSE

Gross limits for rise-rate characteristics have been incorporated in Material Specification 2170402[1] and 3170567[2] defining, respectively, the Bendix Thermalthane 4002-4 and Bendix Thermalthane 4003-8 foam systems. Sandia Albuquerque had considered placing rise-rate requirements on additional foam materials. However, before meaningful limits could be established, the effect of changes in material and processing variables on the rise-rate characteristics had to be determined.

PRIOR WORK

An earlier short term process development order (PDO 6989016) proved that the rise-rate machine provides reproducible data for measuring the rise-rate characteristics of rigid urethane foams.[3] However, to obtain this reproducibility, potential material and process variables, such as catalyst concentrations, mix ratios, and component temperatures, must be closely controlled. In accordance with the findings of the process development order, the mechanized mixing apparatus, Gen K 2423-0-003, which provided the most reproducible results, was installed in the Materials Engineering Laboratory.

ACTIVITY

Experiments were conducted to determine how the rise-rate characteristics, gel times, and physical properties of three rigid urethane foams were affected by changes in processing parameters and material formulations.

Experiments

The experiments were planned to study the effect of changes in eight material and processing variables--component temperature, mix time, mix speed, water concentration, mix ratio, mix quantity, pour weight, and material reactivity--on the rise-rate and gel time characteristics and physical properties of the foam. After the first set of experiments, four of the eight variables proved to be insignificant. Therefore, the original designed experiments were modified by reducing the number of independent variables to two in each of the remaining experiments.

Three foam systems were included in the study. The variables investigated were divided to form two experiments for each foam system. Experiments 1, 3, and 5 were used to study the effects of the component temperature-percent water variables with the polymeric isocyanate-polyether polyol, the polymeric isocyanate-polyester polyol, and the TDI-polyester polyol materials, respectively. Experiments 2, 4, and 6 were used to study the effects of the mix ratio-material reactivity variables on the same isocyanate-polyol combinations.

Rise-Rate Procedure

The rise-rate characteristics of the foams were determined using a rise-rate measuring device similar to that shown in Figure 1. The lightweight foam float (A) is placed on the surface of the liquid material (B). A line attached to the float is connected to the transducer (C). As the liquid material in the cup begins to foam and rise, the float and line are lifted causing the transducer to turn. The movement of the transducer sends an electrical signal to the computer amplifier (D). This signal, when graphically recorded on chart (E), shown in Figure 2, plots the instantaneous rise-height of the foam versus time while chart (F), shown in Figure 3, records the instantaneous velocity of the foam versus time. Definitions for the symbols used on Figures 2 and 3 are given in Table 1. The following rise-rate characteristics are obtained from the graphs.

Initiation Time (T_I)	Time interval from start of mixing to the time when 2 percent of maximum foam height occurs.
Time to Maximum Velocity (T_{VM})	Time interval from start of mixing to the time the maximum velocity occurs.
Rise Time (R_R)	Time interval from start of mixing to the time when 95 percent of the maximum foam height occurs.
Maximum Velocity (V_M)	Maximum instantaneous velocity of the foam.
Maximum Height (X_M)	The final height of the foam.
Rise-Rate Constant (C)	V_M/X_M
Foaming Time (T_F)	$T_R - T_I$

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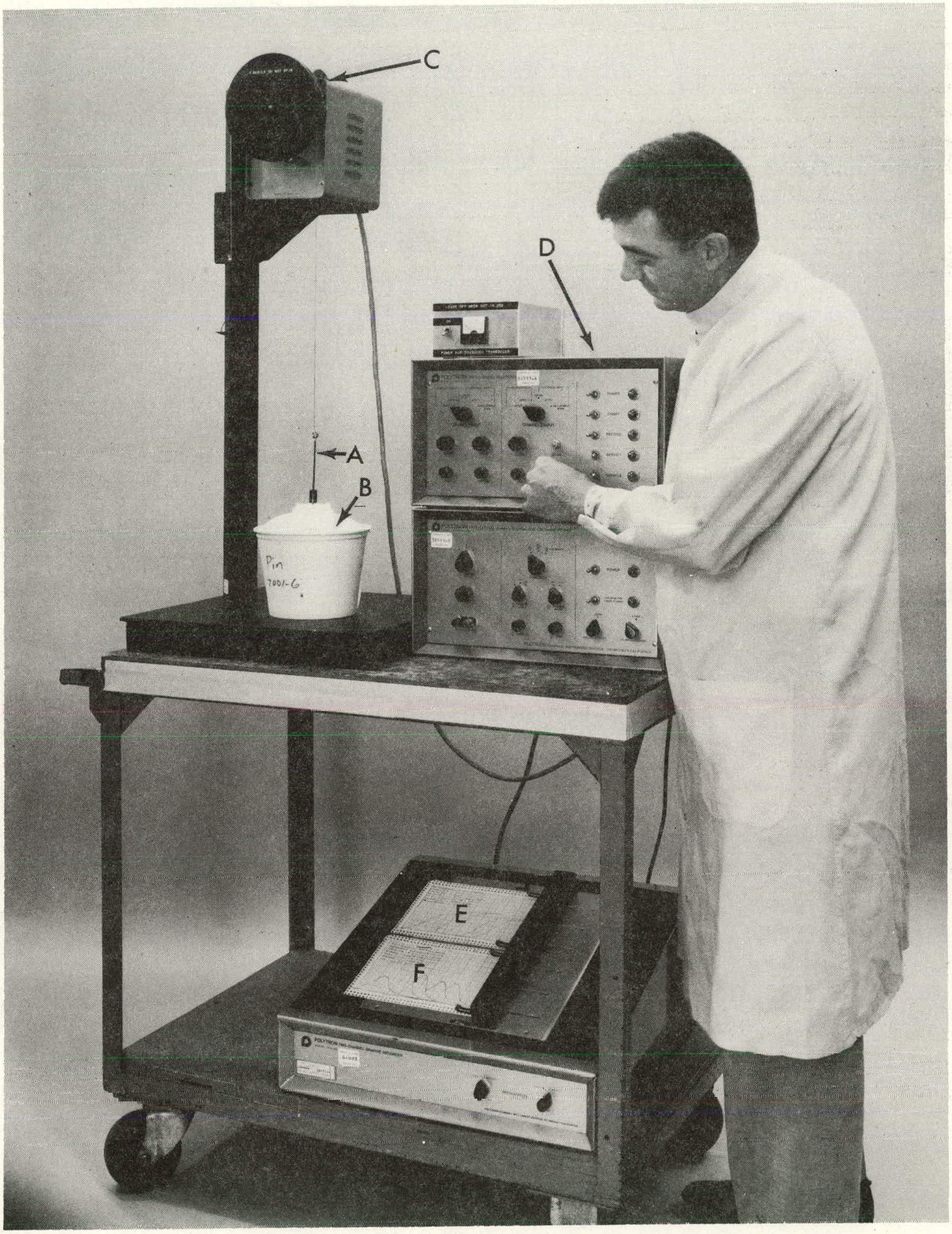
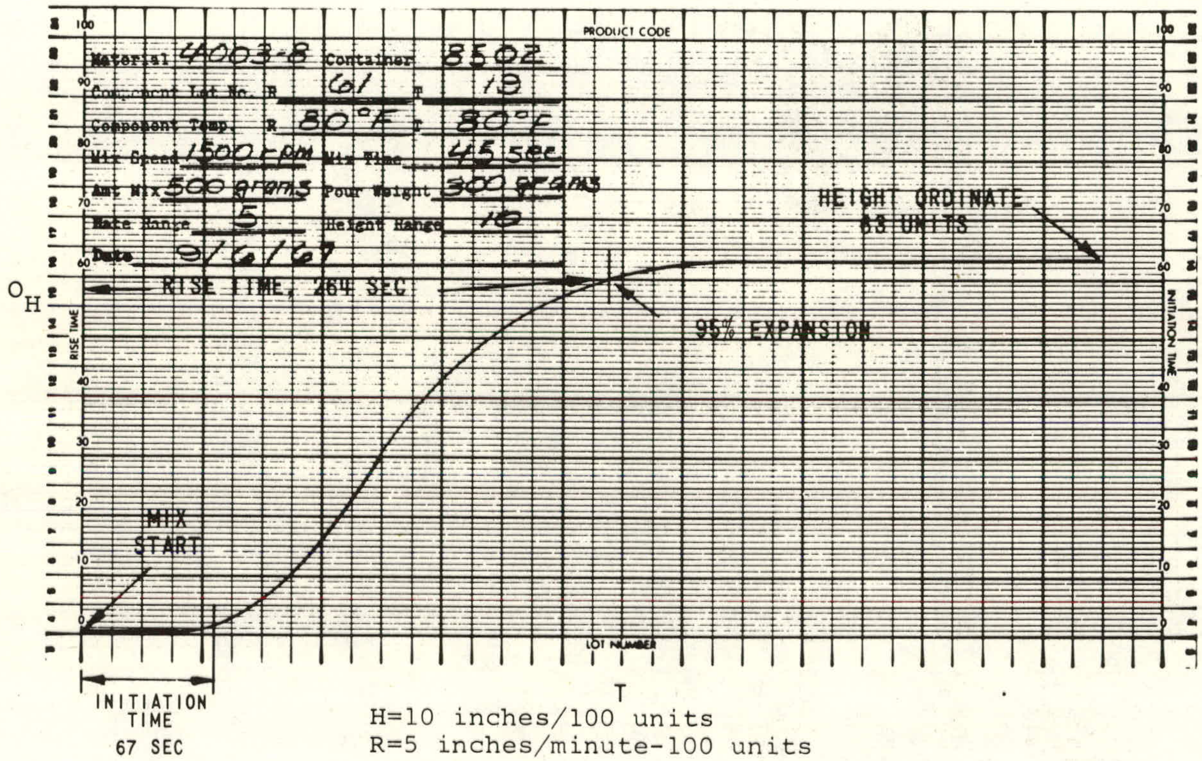


Figure 1. Rise-Rate Machine



Material 4003-8 Container 8502
Component Lot No. R 61 T 19
Component Temp. R 80°F T 80°F
Mix Speed 1500 rpm Mix Time 45 sec
Amt Mix 500 grams Pour Weight 300 grams
Rate Range 5 Height Range 10
Date 9/6/67

H=10 inches/100 units
R=5 inches/minute-100 units

RISE RECORDER
CLASS B MATERIAL

MIX START:

$X_o = 0$ INCHES

$T_o = 0$ SECONDS

MAXIMUM HEIGHT:

$$X_m = (H/100)\,(O_{H_m})$$
$$= (0.1)\,(63)$$
$$= 6.3 \text{ INCHES } (160 \text{ mm})$$

2 PERCENT EXPANSION:

$X_i = (2 \text{ PERCENT})\,(X_m) = (0.02)\,(6.3) = 0.13$ INCHES (3.3 mm)

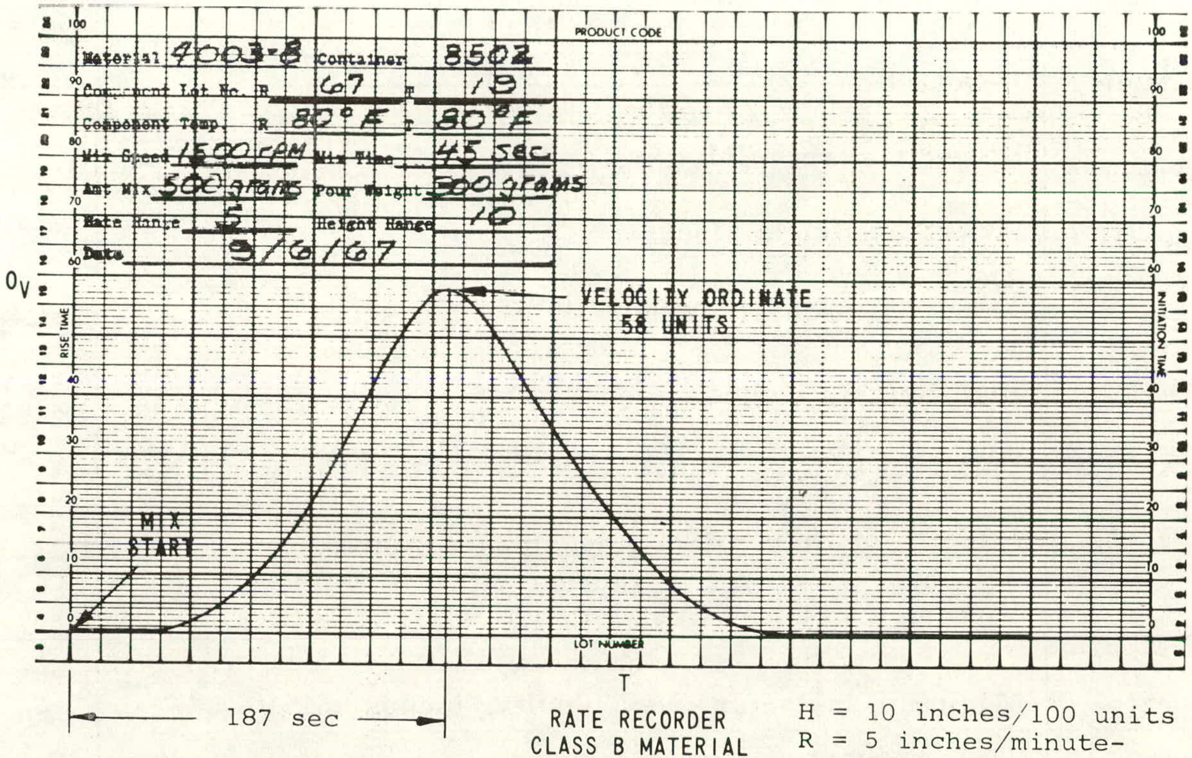
$T_i = 67$ SECONDS

95 PERCENT EXPANSION

$X_r = (95 \text{ PERCENT})\,(X_m) = (0.95)\,(6.3) = 6.0$ INCHES (152 mm)

$T_r = 264$ SECONDS

Figure 2. Rise Recorder Curve



Material 4003-8 Container 8502
Component Lot No. R 67 T 19
Component Temp. R 80°F T 80°F
Mix Speed 1500 rpm Mix Time 45 sec
Amt Mix 500 grams Pour Weight 300 grams
Rate Range 5 Height Range 10
Date 9/6/67

RATE RECORDER
CLASS B MATERIAL

H = 10 inches/100 units
R = 5 inches/minute-
100 units

MIX START:

V_0 = 0 INCHES/MINUTE
T_0 = 0 SECONDS

MAXIMUM VELOCITY:

$V_m = (R/100)\ (O_{V_m}) = (0.05)\ (58) = 2.9$ INCHES/MINUTE (123 mm/s)

$T_{V_m} = 187$ SECONDS

$C = V_m/X_m$
$= (2.9)/(6.3)$
$= 0.460$ INCHES/MINUTE/INCH (0.008 mm/s/mm)

Figure 3. Rate Recorder Curve

Table 1. Definitions of Symbols

Symbol	Description
X_0	Height at Start of Mixing, inches
X_m	Maximum Height, inches
O_{H_m}	Maximum Height Ordinate, units
X_r	Rise Height, inches
X_i	Initiation Height, inches
V_o	Velocity at Start of Mixing, inches/minute
O_{V_M}	Maximum Velocity Ordinate, units
V_m	Maximum Instantaneous Velocity, inches/minute
T_o	Time to Start of Mixing, seconds
T_i	Initiation Time, seconds
T_r	Rise Time, seconds
T_{V_m}	Time to Maximum Velocity, seconds
T_f	Foaming Time, Tr-Ti
C	Rise-Rate Constant, inches/minute/inch
R	Rate Range Selector Setting, inches/100 units
H	Height Range Selector Setting, inches/minute-100 units
O_v	Velocity Ordinate
O_H	Height Ordinate

Gel Time Machine

The gel times of the foam systems were determined using the Fluidyne Model 121 Pressure Measuring System. This equipment consists of a hollow cylinder with a pressure sensing element at its base. As the foam rises in the column, a curve of pressure versus time is recorded, as shown in Figure 4. Since, at the gel point, the viscosity and thus, from Poiseuille's law, the pressure drop in the column increases rapidly, the gel point of the foam can be determined by the intersection of the tangents of the relatively linear portion of the curve surrounding the inflection point.[4]

Foam Systems

Originally, it was planned to test four materials representing each of the basic types of foams:

Polymeric Isocyanate-Polyether Polyol

Polymeric Isocyanate-Polyester Polyol,

Toluene Diisocyanate (TDI)-Polyether Polyol, and

Toluene Diisocyanate (TDI)-Polyester Polyol.

One system, the TDI-Polyether polyol foam, was abandoned because of its insufficient rise-rate response.

Polymeric Isocyanate-Polyether Polyol Material

This foam system, CPR X57-14B, was purchased from the CPR Division of the Upjohn Company. The standard formulation for this material was established as follows:

R-Component	PBW
CPR X57-14B Resin (OH No. ≈ 435)	100.0
Distilled Water	3.1
Catalyst (N-methylmorpholine)	1.0
Cell Stabilizer (DC 193)	1.0
Polymeric Isocyanate (A.E. ≈ 135)	176.7

A mix ratio of 62.7 parts polymeric isocyanate to 37.3 parts of the R-Component, yielding a material with an isocyanate index of 1.17, was used.

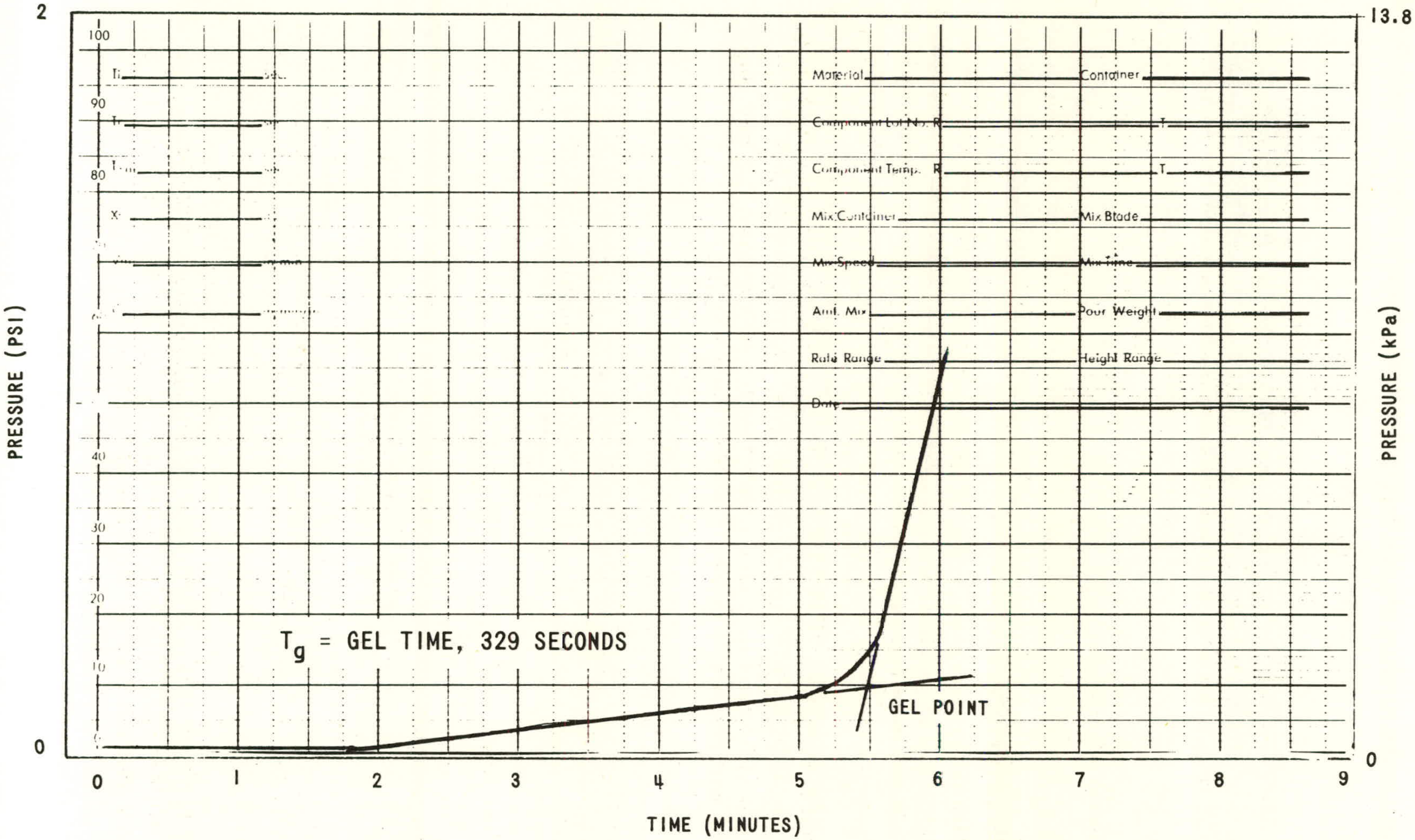


Figure 4. Typical Gel Pressure Curve

Polymeric Isocyanate-Polyester Polyol Material

The original foam system, CPR X57-29D, planned for this project was not used because the material was not stable after the addition of water and N-methylmorpholine catalyst. That is, there was a drastic change in the rise-rate response of the material over a period of five days. The standard formulation for this material was established as follows.

R-Component	PBW
CPR X 57-29D Resin (OH No. ≈ 498)	100
Distilled Water	3.1
Catalyst (N-methylmorpholine)	1.1
Cell Stabilizer (DC 193)	1.0

A mix ratio of 65.3 parts polymeric isocyanate to 34.7 parts of the R-Component was used.

The rise-rate characteristics of the material were determined according to Process Specification 9952034[5], using a mix time of 60 seconds and a mix speed of 1,250 rpm (130 rad/s). The experiments, performed the day the resin was blended and five days later, yielded these results:

	Day 1	Day 5
Initiation Time, Sec.	159	195
Time to Max. Vel., Sec.	294	368
Rise Time, Sec.	413	505
Max. Vel., in/min. (mm/s)	2.78(1.18)	2.21
Max. Height, in. (mm)	5.30(135)	5.30
Rise-Rate Constant	0.525	0.417

No differences between the base resin and aged R-Component could be detected by gel permeation chromatography. The acid number of both materials was 3.61. It appeared that some of the amine catalyst was being deactivated by conversion to its salt.

As a result, the resin for this system was replaced by BKC 1170 Resin, an alpha methylglucoside/ϵ-caprolactone polyester polyol. The standard formulation for this foam system was established as follows:

R-Component	PBW
BKC 1170 Resin (OH No. ≈ 420)	100.0
Distilled Water	3.0
Catalyst (N-methylmorpholine)	0.9
Cell Stabilizer (DC 193)	0.5
Polymeric Isocyanate (AE ≈ 135)	171.3

A mix ratio of 62.2 parts polymeric isocyanate to 37.8 parts of the R-Component was used, yielding a material with an isocyanate index of 1.17.

Toluene Diisocyanate-Polyether Polyol Material

This foam system, CPR X57-14A, was abandoned because the material would not give a satisfactory rise-rate response. To achieve a velocity profile that could be read accurately, the material had to be over-catalyzed to such an extent that it could not be adequately mixed. Another material of similar type was not substituted for the X57-14A because, to do so, would have destroyed any correlation between it and the other polyether polyol material.

Toluene Diisocyanate-Polyester Polyol Material

This material, CPR X57-29C, since it contained the same polyol as did the CPR X57-29D, experienced the same reactivity changes on aging. As a result, the polyol was replaced with BKC 1170 Resin. The toluene diisocyanate prepolymer was also replaced with a BKC produced toluene diisocyanate prepolymer which utilized Bendix 1170 Resin in its manufacture. The standard formulation for this replacement system was as follows:

R-Component	PBW
BKC 1170 Resin (OH No. ≈ 420)	100.0
Distilled Water	3.0
Catalyst (N-methylmorpholine)	0.35
Cell Stabilizer (DC 193)	0.5
Toluene Diisocyanate Prepolymer (AE ≈ 130)	157.1

A mix ratio of 60.2 parts prepolymer to 39.8 parts of the R-Component was used, yielding a material with an isocyanate index of 1.12.

Rise-Rate and Gel Time Results

The rise-rate and gel time experiments were conducted using the variable coded levels shown in Tables 2 through 7 for each formulation. The actual experimental results obtained from the various combinations of levels are shown in Tables 8 through 13. From these tables specific experimental values for the variables at any level can be obtained. The eight variables used for the polymeric isocyanate-polyether polyol material, Experiments 1 and 2, were reduced to four for Experiments 3, 4, 5, and 6 by the deletion of mix time, mix quantity, mix speed, and pour weight. Since these variables did not alter the rise-rate and gel time responses in the first two experiments, it was assumed that their effect would also be negligible in the remaining experiments. This was confirmed by evaluating these variables at their extreme limits. When no effect on the properties of the materials was noticed under these conditions, the previous conclusion was deemed correct and no further experiments using these variables were conducted.

Rise-Rate and Gel Time Versus Component Temperature

The rise-rate and gel time variables for all three foam systems were affected similarly by changes in component temperatures. Graphical representations of these effects are shown in Figure 5.

Initiation Time. The initiation times of all three types of foam systems were affected similarly by changes in component temperatures. This response reaches a minimum, limited by mix time, as the temperature is increased. As the component temperature is decreased, the initiation time will approach a maximum of infinity when the material becomes so cold that it will not foam. The initiation times of the materials decreased approximately 25 percent for each 10 degrees F (5°C) increase in component temperature..

Time to Maximum Velocity. The effect of component temperature on this response is the same as it was for initiation time. Again the times to maximum velocity for the materials decreased approximately 25 percent for each 10 degrees F increase in component temperature.

Rise Time. The effect of component temperature on the rise time--increases in temperature giving decreases in time--is nearly identical to the temperature effect on initiation time. The rise time of the material decreases about 20 percent for each 10 degree F increase in component temperature.

Maximum Velocity. The maximum velocity of each of the three materials increased approximately 20 percent with each 10 degree F rise in component temperature.

Text continued on page 38.

Table 2. Levels of Variables in Experiment 1 for Polymeric Isocyanate-Polyether Polyol Foam System.

Coded Level	X1 Comp. Temp (°F) (°C)	X2 Mix Time (sec)	X3 Mix Speed (rpm) (Rad/s)	X4 Water (Percent)
-2	60 (15.6)	40	1000 (104.7)	2.48
-1	70 (21.1)	50	1125 (117.8)	2.79
0	80 (26.7)	60	1250 (130.9)	3.10
+1	90 (32.2)	70	1375 (144.0)	3.41
+2	100 (37.8)	80	1500 (157.1)	3.72

Mix ratio = 62.7 percent T-component

Mix quantity = 400 g

Pour weight = 200 g

Grams catalyst per 100.0 parts resin = 1.0

Table 3. Levels of Variables in Experiment 2 for Polymeric Isocyanate-Polyether Polyol Foam System

Coded Level	X1 Mix Ratio (Percent T)	X2 Mix Quantity (g)	X3 Pour Weight (g)	X4 Material Reactivity
-2	56.7	320	150	- - -
-1	59.7	360	175	Slow*
0	62.7	400	200	Medium**
+1	65.7	440	225	Fast***
+2	68.7	480	250	- - -

*0.6 parts catalyst (N-methylmorpholine)/100.0 parts resin

**1.0 parts catalyst (N-methylmorpholine)/100.0 parts resin

***1.4 parts catalyst (N-methylmorpholine)/100.0 parts resin

Mix time = 60 sec

Component temperature = 80°F (26.7°C)

Mix blade speed = 1250 rpm (130 rad/s)

Parts water per 100.0 parts resin = 3.1

Table 4. Levels of Variables in Experiment 3 for Polymeric Isocyanate-Polyester Polyol Foam System

Coded Level	X1 Component Temperature (°F) (°C)	X2 Water (Percent)
-2	60 (15.6)	2.4
-1	70 (21.1)	2.7
0	80 (26.7)	3.0
+1	90 (32.2)	3.3
+2	100 (37.8)	3.6

Mix time = 60 sec

Mix quantity = 400 g

Pour weight = 200 g

Mix speed = 1250 rpm (130 rad/s)

Parts catalyst per 100.0 parts Resin = 0.9

Mix ratio = 62.2 percent T-Component

Table 5. Levels of Variables in Experiment 4 for Polymeric Isocyanate-Polyester Polyol Foam System

Coded Level	X1 Mix Ratio (Percent T)	X2 Material Reactivity
-2	56.2	- - -
-1	59.2	slow*
0	62.2	medium**
+1	65.2	fast***
+2	68.2	- - -

*0.8 parts catalyst (N-methylmorpholine)/ 100.0 parts resin

**0.9 parts catalyst (N-methylmorpholine)/ 100.0 parts resin

***1.0 parts catalyst (N-methylmorpholine)/ 100.0 parts resin

Mix time = 60 sec
Mix quantity = 400 g
Pour weight = 200 g
Mix speed = 1250 rpm (130 rad/s)
Parts water per 100.0 parts resin = 3.0
Component temperature = 80°F (26.7°C)

Table 6. Levels of Variables in Experiment 5 for TDI-Polyester Polyol Foam System

Coded Level	X1 Component Temperature (°F)	X2 Water (Percent)
-2	60 (15.6)	2.4
-1	70 (21.1)	2.7
0	80 (26.7)	3.0
+1	90 (32.2)	3.3
+2	100 (37.8)	3.6

Mix time = 25 sec

Mix quantity = 400 g

Pour weight = 200 g

Mix speed = 1250 rpm (130 rad/s)

Parts catalyst/
100.0 parts resin = 0.9

Mix ratio = 60.2 percent T-Component

Table 7. Levels of Variables in Experiment 6 for TDI-Polyester Polyol Foam System

Coded Level	X1 Mix Ratio (Percent T)	X2 Material Reactivity
-2	54.2	- - -
-1	57.2	slow[1]
0	60.2	medium[2]
+1	63.2	fast[3]
+2	66.2	- - -

1. 0.20 parts catalyst (N-methylmorpholine)/ 100.0 parts resin.
2. 0.35 parts catalyst (N-methylmorpholine)/ 100.0 parts resin.
3. 0.50 parts catalyst (N-methylmorpholine)/ 100.0 parts resin.

Mix speed = 1250 rpm (130 rad/s)
Mix time = 25 sec
Mix quantity = 400 g
Pour weight = 200 g
Parts water/ 100.0 parts resin = 3.0
Component temperature = 80°F (26.7°C)

Table 8. Averages and Standard Deviations for the Responses in Experiment 1.

Design Matrix					Response**							
Line No.	X1*	X2	X3	X4	T_i	T_{v_m}	T_r	V_m	X_m	C	T_f	T_g
1	-1	-1	-1	-1	166.3	379.3	642.0	1.28	6.75	0.190	475.0	474
					2.9	7.8	4.4	0.04	0.13	0.004	5.0	
2	+1	+1	-1	-1	105.0	232.4	454.4	1.69	6.56	0.258	349.4	
					3.5	10.0	9.3	0.09	0.23	0.006	3.2	
3	+1	-1	+1	-1	90.0	232.0	441.0	1.72	6.56	0.262	351.0	348
					6.1	5.3	17.2	0.17	0.23	0.016	12.6	
4	+1	-1	-1	+1	91.5	214.8	427.5	2.16	7.63	0.283	336.0	
					3.4	2.5	11.1	0.04	0.18	0.003	9.6	
5	-1	+1	+1	-1	159.2	376.6	620.2	1.30	6.74	0.192	461.0	
					16.1	15.4	17.3	0.03	0.10	0.005	8.5	
6	-1	+1	-1	+1	159.0	378.0	617.0	1.55	7.81	0.199	458.0	487
					1.7	3.6	4.6	0.03	0.09	0.001	3.0	
7	-1	-1	+1	+1	153.7	371.7	574.1	1.55	7.13	0.218	422.4	
					10.1	14.2	27.1	0.21	0.82	0.023	22.6	
8	+1	+1	+1	+1	102.0	205.8	408.0	2.23	7.33	0.302	306.0	355
					1.9	8.7	7.3	0.12	0.15	0.013	8.8	
9	0	0	0	0	122.6	294.8	517.8	1.65	6.94	0.238	395.2	412
					1.52	2.7	7.2	0.02	0.06	0.001	6.3	
10	0	0	0	0	117.7	276.0	475.7	1.80	7.33	0.245	358.0	420
					3.5	10.1	16.6	0.04	0.06	0.004	14.5	
11	+2	0	0	0	86.7	157.0	343.0	2.83	6.70	0.422	256.3	341
					3.1	12.2	20.1	0.02	0.10	0.009	18.2	
12	-2	0	0	0	203.0	482.0	743.6	1.22	7.13	0.170	540.6	615
					7.7	1.6	16.9	0.06	0.20	0.006	10.1	
13	0	+2	0	0	127.2	288.4	505.8	1.72	7.07	0.243	378.6	
					2.3	1.3	11.0	0.05	0.17	0.002	10.0	
14	0	-2	0	0	127.8	310.5	529.5	1.49	6.99	0.213	401.8	
					2.6	9.3	3.7	0.04	0.08	0.004	1.5	
15	0	0	+2	0	115.6	287.4	510.4	1.52	6.80	0.224	395.8	
					4.1	3.9	5.1	0.02	0.07	0.002	8.9	
16	0	0	-2	0	132.0	306.3	547.7	1.62	6.79	0.239	415.7	
					2.6	3.8	6.8	0.03	0.09	0.001	9.3	
17	0	0	0	+2	110.7	284.0	489.3	2.00	7.99	0.253	378.7	371
					2.1	1.0	5.1	0.0	0.16	0.0	6.5	
18	0	0	0	-2	140.3	329.0	568.0	1.25	6.08	0.205	427.7	479
					0.6	1.7	1.0	0.01	0.07	0.002	1.5	

* Variable levels from Table 2

** Definitions from Table 1 and Figure 4

Table 9. Averages and Standard Deviations for the Responses in Experiment 2.

Design Matrix					Response ***							
Line No.	X1	X2	X3 *	X4 **	T_i	T_{v_m}	T_r	V_m	X_m	C	T_f	T_g
1	-1	-1	-1	-1	162.7	400.2	716.5	1.04	6.42	0.162	554.7	540
					6.0	4.8	2.9	0.02	0.14	0.005	5.2	
2	+1	+1	-1	-1	214.0	539.0	900.7	0.70	5.55	0.126	688.0	661
					3.5	1.7	9.7	0.09	0.20	0.001	10.5	
3	+1	-1	+1	-1	178.5	482.8	873.5	0.94	7.25	0.129	695.0	
					7.2	3.8	12.5	0.03	0.17	0.003	12.7	
4	+1	-1	-1	+1	113.3	269.3	463.3	1.65	6.11	0.273	350.0	
					1.5	1.2	2.9	0.05	0.19	0.002	2.0	
5	-1	+1	+1	-1	148.7	385.3	689.0	1.41	8.50	0.166	540.3	
					1.2	1.5	1.7	0.06	0.32	0.001	1.5	
6	-1	+1	-1	+1	103.0	243.0	375.0	2.55	7.52	0.339	272.0	273
					2.6	3.5	4.0	0.04	0.14	0.001	6.6	
7	-1	-1	+1	+1	104.0	248.0	384.0	3.15	8.99	0.351	280.0	
					1.4	0.0	1.4	0.0	0.01	0.001	0.0	
7*	-1	-1	-1	+1	102.0	240.0	379.0	2.57	7.41	0.343	277.0	
					0.0	0.0	0.0	0.04	0.0	0.0	0.0	
8	+1	+1	+1	+1	111.5	268.0	456.5	2.05	7.99	0.256	345.0	345
					0.7	2.8	0.7	0.0	0.09	0.001	0.0	
9	0	0	0	0	118.5	292.5	524.7	1.66	7.20	0.231	406.2	429
					3.1	3.3	7.2	0.04	0.21	0.004	7.6	
10	0	0	0	0	122.5	296.3	503.8	1.62	6.98	0.231	381.3	433
					3.0	4.0	0.5	0.06	0.22	0.002	3.4	
11	+2	0	0	0	159.0	370.7	751.0	0.84	5.81	0.143	592.0	662
					2.6	0.6	4.6	0.01	0.06	0.001	3.6	
12	-2	0	0	0	111.3	278.3	445.3	2.28	8.05	0.283	334.0	341
					1.0	3.8	2.5	0.02	0.08	0.001	2.4	
13	0	+2	0	0	126.7	309.3	552.0	1.54	7.14	0.216	425.3	
					1.5	0.6	3.0	0.02	0.08	0.0	2.3	
14	0	-2	0	0	114.0	303.3	542.0	1.55	7.16	0.217	428.0	
					2.0	1.5	2.0	0.02	0.08	0.001	0.0	
15	0	0	+2	0	114.3	294.3	517.3	1.89	8.47	0.223	403.0	
					2.5	4.5	5.5	0.05	0.10	0.001	5.2	
16	0	0	-2	0	136.0	319.3	539.0	1.24	5.52	0.224	403.3	
					2.7	1.0	2.7	0.01	0.07	0.002	1.3	
17	0	0	0	-1	171.6	444.6	785.8	1.00	6.92	0.144	614.2	
					3.0	4.8	12.5	0.05	0.18	0.005	10.3	

* X3 set at a -1 level instead of +1 level

** Variable levels from Table 3

*** Definitions from Table 1 and Figure 4

Table 10. Averages and Standard Deviations for the Responses in Experiment 3

Design Matrix			Response**							
Line No.	X1*	X2	T_i	T_{V_m}	T_r	V_m	X_m	C	T_f	T_g
1	+1	+1	105.7	243.8	428.0	2.573	6.900	0.3753	322.3	343
			1.7	3.7	2.2	0.071	0.071	0.0040	1.9	
2	-1	+1	176.0	365.0	571.8	1.920	6.593	0.2910	395.8	44
			0.8	2.2	2.4	0.014	0.015	0.0014	2.5	
3	+1	-1	101.8	219.8	391.0	2.550	6.040	0.4222	287.2	333
			0.8	5.0	6.1	0.044	0.055	0.0045	5.4	
4	-1	-1	199.3	368.5	565.0	1.725	5.738	0.3007	365.8	447
			4.2	1.3	3.3	0.006	0.015	0.0013	3.5	
5	0	0	140.0	308.8	522.5	1.850	6.300	0.2937	382.5	365
			0.8	2.1	2.1	0.014	0.041	0.0012	1.9	
6	0	0	139.0	306.5	520.0	1.730	6.085	0.2930	381.0	370
			1.6	1.3	2.2	0.062	0.189	0.0017	1.0	
7	+2	0	89.5	184.0	393.0	2.528	6.050	0.4180	303.5	252
			0.6	2.6	2.9	0.032	0.091	0.0014	2.9	
8	-2	0	233.3	474.0	740.0	1.463	6.115	0.2390	506.8	550
			1.0	1.8	0.8	0.013	0.037	0.0008	1.5	
9	0	+2	126.8	264.0	440.8	2.768	6.923	0.3998	314.0	352
			1.7	3.7	1.0	0.047	0.092	0.0025	2.2	
10	0	-2	137.0	280.8	461.0	1.863	5.120	0.3693	324.0	362
			1.4	2.1	3.2	0.015	0.024	0.0097	2.9	

*Variable levels from Table 4
**Definitions from Table 1 and Figure 4

Table 11. Averages and Standard Deviations for the Responses in Experiment 4

Design Matrix			Response**							
Line No.	X1*	X2	T_i	T_{V_m}	T_r	V_m	X_m	C	T_f	T_g
1	+1	+1	139.5	305.3	517.8	1.618	5.613	0.2880	378.3	461
			4.4	1.5	2.2	0.013	0.030	0.0008	2.4	
2	-1	+1	120.3	230.0	353.0	3.425	6.500	0.5270	232.8	333
			0.5	3.6	2.4	0.010	0.000	0.0014	2.1	
3	+1	-1	159.0	398.0	813.0	0.895	5.185	0.1728	654.0	
			2.2	0.8	2.9	0.013	0.064	0.0005	2.4	
4	-1	-1	134.3	288.8	462.5	2.233	5.075	0.4398	328.8	618
			1.0	1.0	3.8	0.024	0.050	0.0019	1.9	
5	0	0	137.8	304.3	512.8	1.753	6.068	0.2890	375.0	368
			2.5	2.1	5.0	0.010	0.013	0.0020	7.5	
6	0	0	139.6	303.0	521.2	1.784	6.042	0.2954	381.6	365
			3.4	6.1	4.7	0.050	0.168	0.0017	6.7	
7	+2	0	170.8	424.5	925.0	0.650	4.893	0.1328	754.3	
			1.4	1.3	0.0	0.008	0.060	0.0005	2.5	
8	-2	0	116.0	239.0	348.0	3.523	6.978	0.5048	232.3	
			1.4	2.4	2.9	0.032	0.045	0.0033	2.5	
9	0	+1								316
10	0	-1								450

*Variable levels from Table 5
**Definitions from Table 1 and Figure 4

Table 12. Averages and Standard Deviations for the Responses in Experiment 5

Design Matrix			Response**							
Line No.	X1*	X2	T_i	T_{V_m}	T_r	V_m	X_m	C	T_f	T_g
1	+1	+1	97.0	166.7	398.7	2.92	7.43	0.393	301.7	315
			1.0	0.6	2.3	0.03	0.05	0.001	1.5	
2	-1	+1	172.7	287.7	569.7	1.89	7.40	0.255	397.0	438
			0.6	0.6	2.9	0.01	0.0	0.017	3.5	
3	+1	-1	112.5	181.0	453.5	2.01	6.36	0.316	341.0	338
			2.5	1.2	1.7	0.03	0.03	0.004	3.6	
4	-1	-1	186.3	290.5	655.0	1.35	6.10	0.221	468.8	464
			3.9	3.9	4.1	0.01	0.0	0.001	3.0	
5	0	0	127.5	212.8	501.3	203	6.87	0.293	373.8	387
			2.9	2.2	2.6	0.02	0.02	0.006	1.9	
6	0	0	140.4	226.2	519.0	1.91	6.70	0.286	378.6	375
			1.8	2.2	2.9	0.02	0.03	0.003	2.4	
7	+2	0	70.0	151.3	389.3	2.49	6.71	0.370	319.3	330
			0.0	2.9	4.3	0.07	0.20	0.001	4.3	
8	-2	0	235.0	419.3	779.0	1.19	6.64	0.179	544.0	695
			5.7	5.1	2.9	0.03	0.05	0.004	4.1	
9	0	+2	111.8	190.6	425.2	2.94	7.97	0.369	313.4	368
			2.5	0.9	3.0	0.02	0.04	0.003	5.0	
10	0	-2	149.0	225.8	566.0	1.43	5.69	0.252	417.0	480
			1.6	2.3	2.2	0.04	0.03	0.006	2.7	

*Variable Levels from Table 6
**Definitions from Table 1 and Figure 4

Table 13. Averages and Standard Deviations for the Responses in Experiment 6

Design Matrix			Response**							
Line No.	X1*	X2	T_i	T_{V_m}	T_r	V_m	X_m	C	T_f	T_g
1	+1	+1	123.0	206.0	453.0	2.388	6.883	0.345	328.8	357
			0.8	1.4	0.0	0.051	0.146	0.004	3.2	
2	-1	+1	114.5	176.0	389.8	3.000	7.413	0.406	275.3	280
			2.6	1.4	3.2	0.070	0.085	0.007	4.1	
3	+1	-1	157.5	344.3	726.5	0.955	5.930	0.161	569.0	480
			3.8	5.9	5.2	0.042	0.220	0.002	5.6	
4	-1	-1	135.7	261.7	586.0	1.400	6.470	0.213	447.0	480
			2.1	0.6	6.1	0.070	0.060	0.004	1.7	
5	0	0	129.8	221.8	519.0	2.020	6.74	0.300	389.3	390
			0.5	0.5	2.7	0.030	0.05	0.003	3.0	
6	0	0	130.8	221.5	250.0	2.040	6.84	0.298	389.3	384
			1.0	2.6	0.0	0.05	0.03	0.006	1.0	
7	+2	0	153.4	264.4	630.0	1.370	5.75	0.238	476.6	600
			1.1	0.9	7.9	0.030	0.06	0.004	7.2	
8	-2	0	117.7	187.3	451.7	2.470	7.23	0.341	334.0	361
			2.5	2.5	1.2	0.060	0.04	0.008	3.6	

*Variable Levels from Table 7
**Definitions from Table 1 and Figure 4

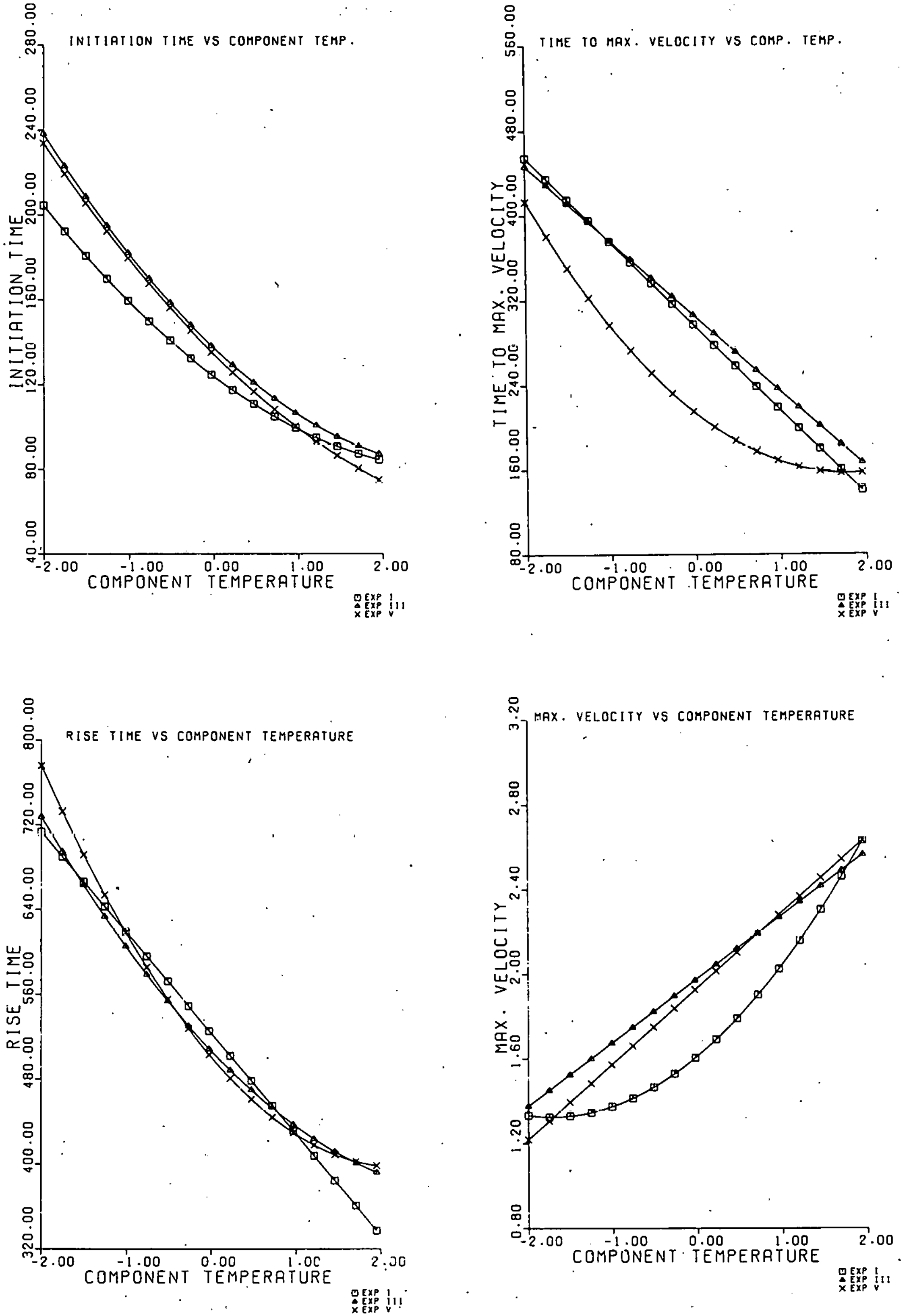


Figure 5. Rise-Rate and Gel Time Results Versus Component Temperature (Reduced Computer Printouts.)

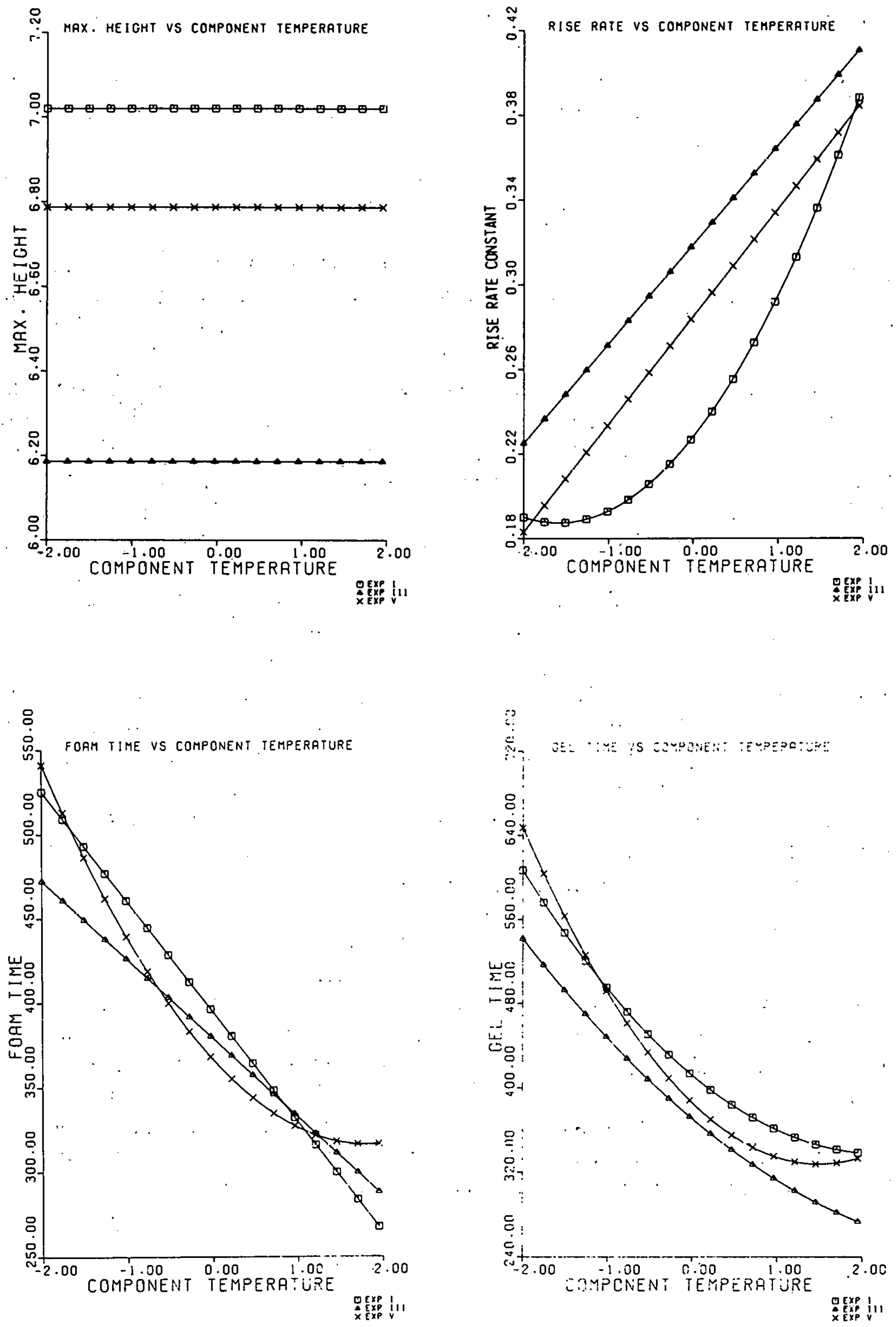


Figure 5 Continued. Rise-Rate and Gel Time Results Versus Component Temperature

Maximum Height. Changes in component temperature do not alter the final height of the foam.

Rise-Rate Constant. Since the rise-rate constant is defined as the maximum velocity divided by the maximum height, and since the component temperature has no effect on the final foam height, the effect of changes in component temperature on the rise-rate constant is identical to the effect that changes in component temperature have on the maximum velocity.

Foaming Time. As the component temperature is increased, the foaming time decreases. That is, while both the initiation time and rise time decrease with increases in component temperature, the rise time decreases faster.

Gel Time. Changes in the component temperature had the same effect, higher temperature and shorter gel time, on the gel time of the foams as it did on the rise-rate results, specifically the time to maximum velocity and the rise time. Also, while the rise time did decrease faster than the gel time for a corresponding increase in component temperature, the difference was not quite large enough to be of major significance. The component temperature, therefore, can be used for small adjustements in the rise-to-gel ratio but could probably not be used to cause a major alteration in the ratio of the rise time to gel time.

From this work, it is impossible to predict the quantitative change in rise-rate characteristics that will occur in all foam systems as the component temperature is altered by a specified amount. The general trend is evident and most similar foam materials should conform to these patterns. However, it should be emphasized, that to obtain reproducible results, the component temperatures of materials undergoing rise-rate tests must be maintained at least within ±2°F (±1°C) of the specified values.

Rise-Rate and Gel Time Results Versus Percent Water

The polymeric isocyanate-polyether polyol and the toluene diisocyanate-polyester polyol materials were affected similarly by changes in resin water concentration. These changes had a greater effect on the toluene diisocyanate material than they did on the polymeric isocyanate-polyether polyol foam. Most of the rise-rate responses of the polymeric isocyanate-polyester polyol system were not altered by changes in resin water concentrations. Graphical representations of these effects are shown in Figure 6.

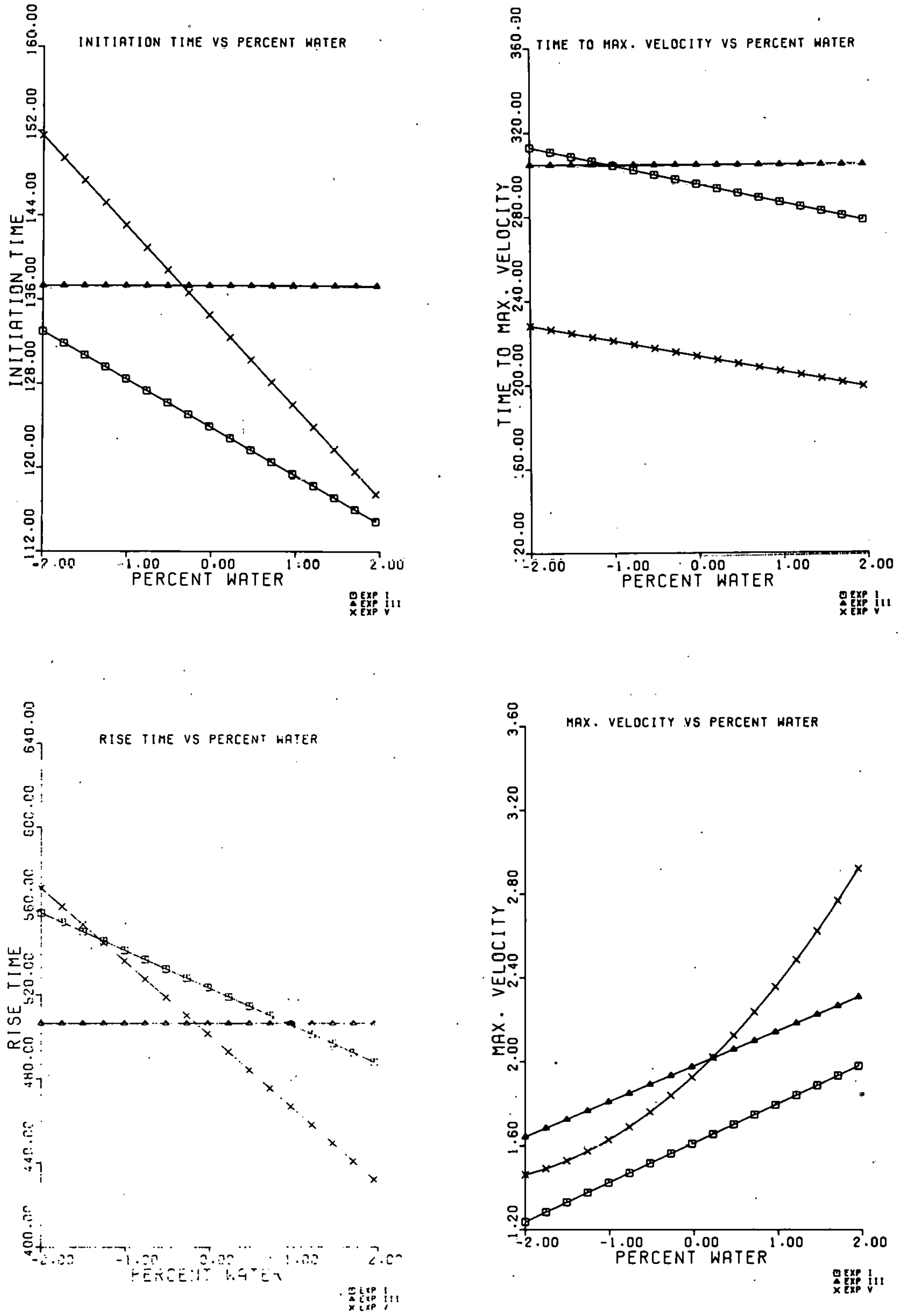


Figure 6. Rise-Rate and Gel Time Results Versus Percent Water (Reduced Computer Printouts.)

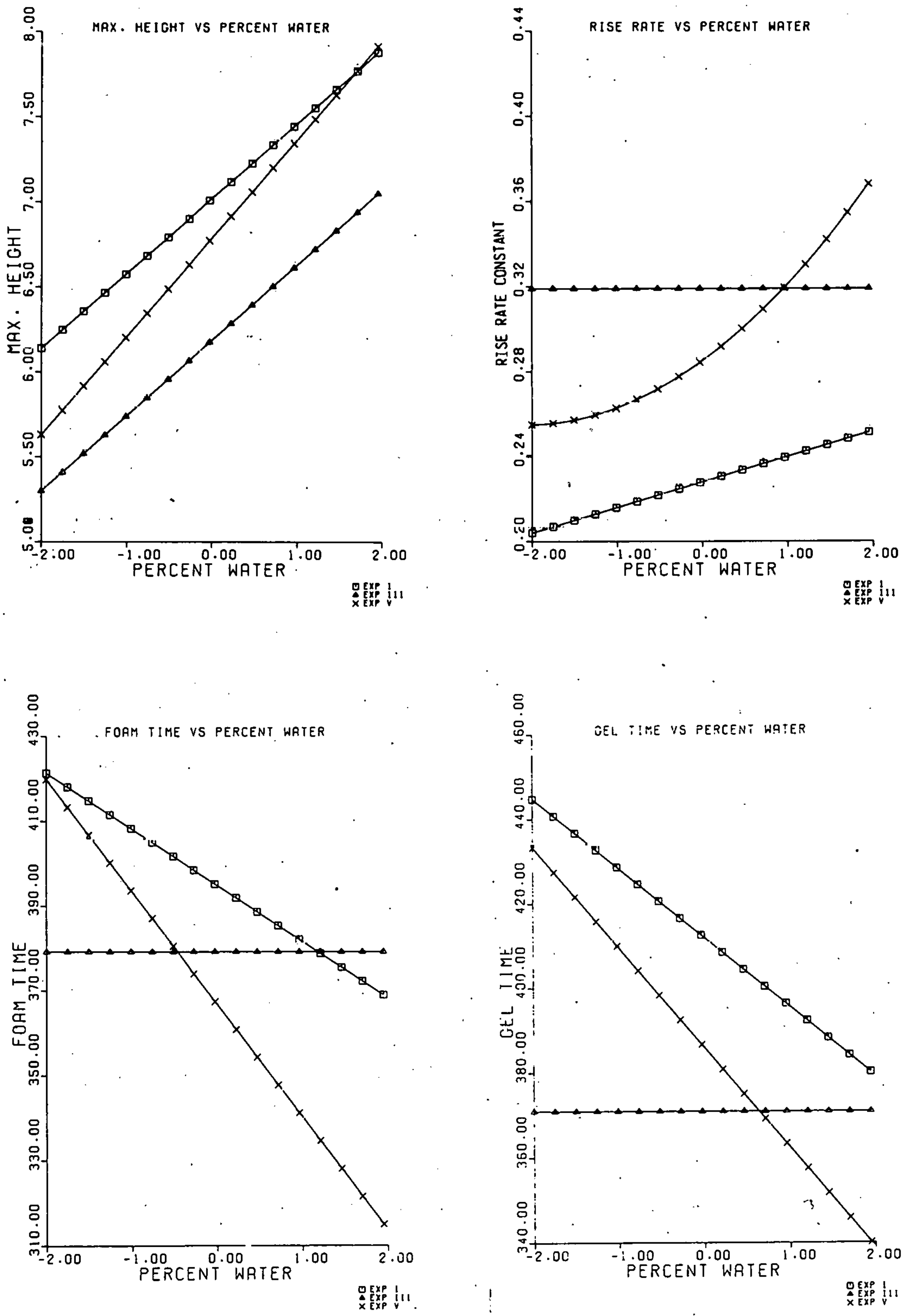


Figure 6 Continued. Rise-Rate and Gel Time Results Versus Percent Water

Initiation Time. The initiation times of the two affected materials were decreased slightly with increased water concentration. This might be expected since the reaction between the water and the isocyanate creates an amine which could catalyze further reactions.[6] The change in initiation time was small however, with a decrease of approximately 5 percent for each 10 percent increase in water content.

Time to Maximum Velocity. Again the times to maximum velocity were decreased, as it was for initiation time, about 5 percent for a 10 percent increase in water concentration.

Rise Time. The effect of resin water concentration on the rise time is the same as it was for intiation time and time to maximum velocity.

Maximum Velocity. The maximum velocity of all of the foams increased as the water concentration increased. The water-isocyanate reaction, besides creating a catalytic amine, yields the carbon dioxide required to expand the foam. Thus assuming no catalytic effect by the created amine, additional water yields more carbon dioxide per unit of time, thereby making the foam expand at a faster rate. Any catalytic effect of the amine would then add to this velocity change.

Maximum Height. As the water concentration of the resin increased, so did the final height of the foam. The foam will rise higher in the cup because of the lower free-rise density created by the additional water. Approximately a 5 percent increase in final height is recorded for each 10 percent increase in water content.

Rise-Rate Constant. The rise-rate constant of the polymeric isocyanate polyether polyol and toluene diisocyanate polyester polyol materials increased as the water concentration increased. This same response was not noted for the polymeric isocyanate-polyester polyol material. While the maximum velocity and maximum height of this foam increase as water was increased, and since the rise-rate constant is equal to the maximum velocity divided by the maximum height, this would indicate that the maximum velocity and height of the polymeric isocyanate-polyester polyol material was increased proportionately by increased water concentration and due to increased foam expansion only.

Had the reaction been catalyzed in some manner, not only the maximum velocity and maximum height, but also the rise-rate constant would have changed.

Foaming Time. The foaming time of the two affected materials were decreased about 5 percent by a 10 percent increase in water concentration.

Gel Time. The water concentration of the resin had a minor effect on the gel time of the foams. As the percent water increased, the gel time decreased slightly. However, the rise time and gel time changed at approximately the same rate, thereby negating the water concentration as a controller of the rise to gel ratio.

As was the case with component temperature, it is impossible to predict the quantitative changes that will occur in the rise-rate characteristics of all foam systems as the resin water concentration is varied. Again, the general trend is evident and most similar materials should conform to these patterns. The toluene diisocyanate foams, probably because the reactivity of toluene diisocyanate is greater than that of polymeric isocyanate, are more affected by changes in water concentration than are the polymeric isocyanate materials. The polymeric isocyanate-polyester polyol foam was not affected by changes in resin water concentration, except for differences in maximum velocity and maximum height due to density rather than reactivity changes. Since the reactivity of isocyanates with active hydrogen compounds is in the order of primary hydroxyl groups, water, and secondary hydroxyl groups,[7] the additional water would probably have accelerated the polyether materials more than the polyesters.

The resin-water content, while it does contribute to the rise-rate and gel time characteristics of the foam, is not nearly as significant a variable as is the component temperature. The control exercised over the resin component water concentration in present material specifications is more than sufficient to give reproducible rise-rate and gel time results. Rise-rate and gel time testing can only detect water concentration variations that are significantly outside the presently prescribed limits.

Rise-Rate and Gel Time Results Versus Mix Ratio

The following seven rise-rate variables of each of the foam systems were affected similarly by changes in the isocyanate content of the foam. See Figure 7.

Initiation Time. The initiation times of all the foam systems were increased by an increase in the mix ratio. In general, an increase from approximately 60 percent to 63 percent of the isocyanate in the total mixture results in about an 8 percent increase in the initiation time.

Time to Maximum Velocity. As was the initiation time, the time to maximum velocity is increased approximately 10 percent by a similar increase in the mix ratio.

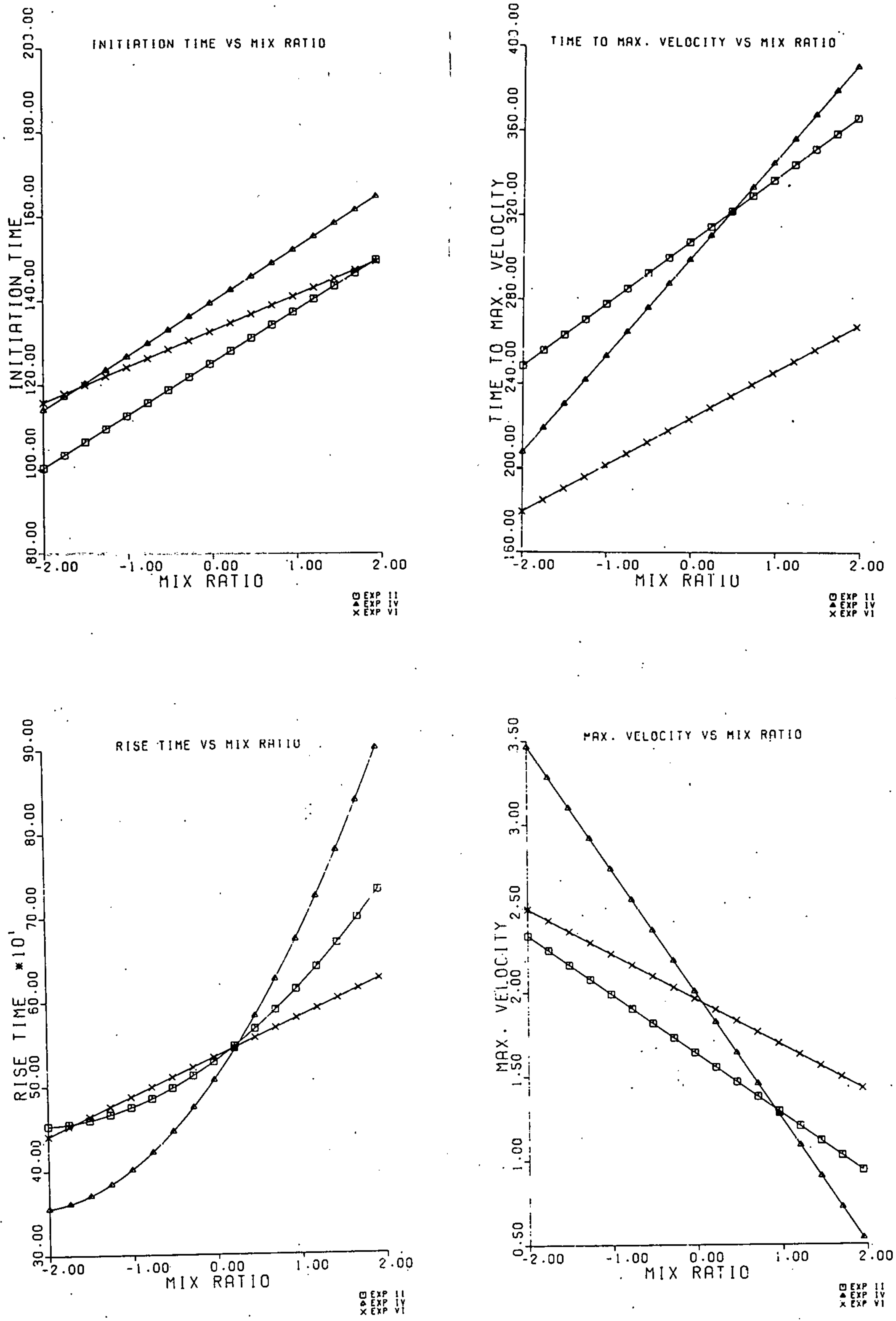


Figure 7. Rise-Rate and Gel Time Results Versus Mix Ratio (Reduced Computer Printouts.)

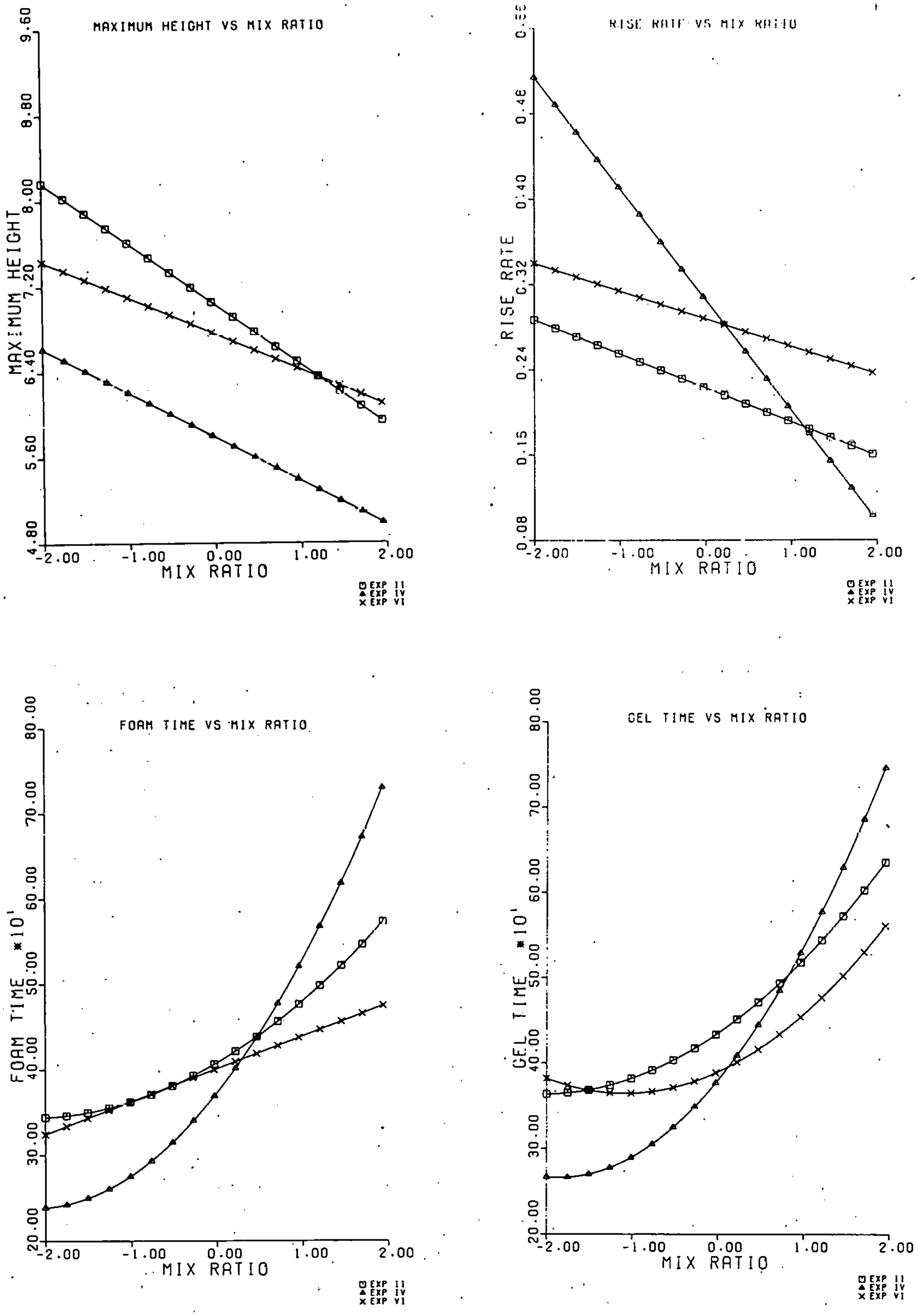


Figure 7 Continued. Rise-Rate and Gel Time Results Versus Mix Ratio

Rise Time. As was related previously, the rise time is also increased about 10 percent as the mix ratio is increased the referenced amount.

Maximum Velocity. As expected, the maximum velocities of the foams decrease, about 20 percent, as the mix ratio is increased.

Maximum Height. The maximum heights of the foams decrease about 7 percent as the mix ratio is increased.

Rise-Rate Constant. The rise-rate constants follow the trend and decrease about 15 percent with the increased mix ratio.

Foaming Time. An increase in the mix ratio causes the foaming time to lengthen about 15 percent for an increase of from 60 percent to 63 percent isocyanate in the total foam.

Gel Time. Changes in mix ratio will significantly affect the gel times of the foams, with lower mix ratios yielding shorter gel times. However, as with the percent water, the gel time and rise time change at the same rate, also eliminating the mix ratio as a means of controlling the rise to gel ratio.

Again, it is impossible to predict the exact effect of mix ratio changes on the rise-rate and gel time characteristics of the foam. Most similar foam systems should, however, conform to the general trends. The mix ratio changes, besides altering the isocyanate-to-resin ratio, also change the concentration of water and catalyst in the foaming system. These will, as explained, have their effect on the total foam system reactivity.

The mix ratio, which is a more significant variable than the water concentration, is not as influential as the component temperature in its effect on the rise-rate and gel time properties of the foams. Present material specifications usually instruct that the mix ratios be maintained within ±1 percent of the indicated values. The percentage is normally with reference to the mix weights and, as such, the actual mix ratio variations possible are sufficiently less than the changes indicated by the levels in the study. Thus, present material specifications should provide adequate control on mix ratios for rise-rate and gel time testing purposes.

Rise-Rate and Gel Time Results Versus Material Reactivity

The catalyst concentration in the resin is the most significant variable affecting the rise-rate and gel time characteristics of the foams. However, different catalyst concentrations and variations on these concentrations were necessary to obtain satisfactory rise-rate curves for each of the foam systems. As such, a direct comparison of the different materials is not applicable. Figure 8, keeping in mind that the levels +2 through -2 do not represent equivalent catalyst concentrations, show comparative graphs of the results. By comparing these figures with the variable levels indicated in Tables 3, 5, and 7 the effect of changes in the amount of catalyst in the foam system can be seen.

The catalyst concentration in the foam was the major factor influencing the rise-to-gel ratio of the material. Increasing the catalyst level naturally caused the foam to gel faster, that is, yielded shorter gel time. However, as the gel times decreased, the rise time decreased even faster. Thus, catalyst concentration appears to be the important variable for adjusting the rise to gel ratio of foams.

Materials specifications do not attempt to control the catalyst concentrations of the foam system they represent. Rise-rate testing can detect small changes in the catalyst concentrations, especially in polyester polyol and toluene diisocyanate foams, and as such would be a good test for establishing control over this formulation variable.

Physical Properties, Compressive Strength

The compressive strength of the polymeric isocyanate-polyether polyol material was evaluated using test specimens cut from 20 pounds per cubic foot (320 kg/m^3) nominal density, 6- by 6- by 1 inch test blocks. The 3 pounds per cubic foot (48 kg/m^3) nominal density, free-rise billets used for the rise-rate determinations were also evaluated. All of the foam samples were cured for 8 hours at 325°F. The average compressive strengths obtained are shown in Tables 14 through 18.

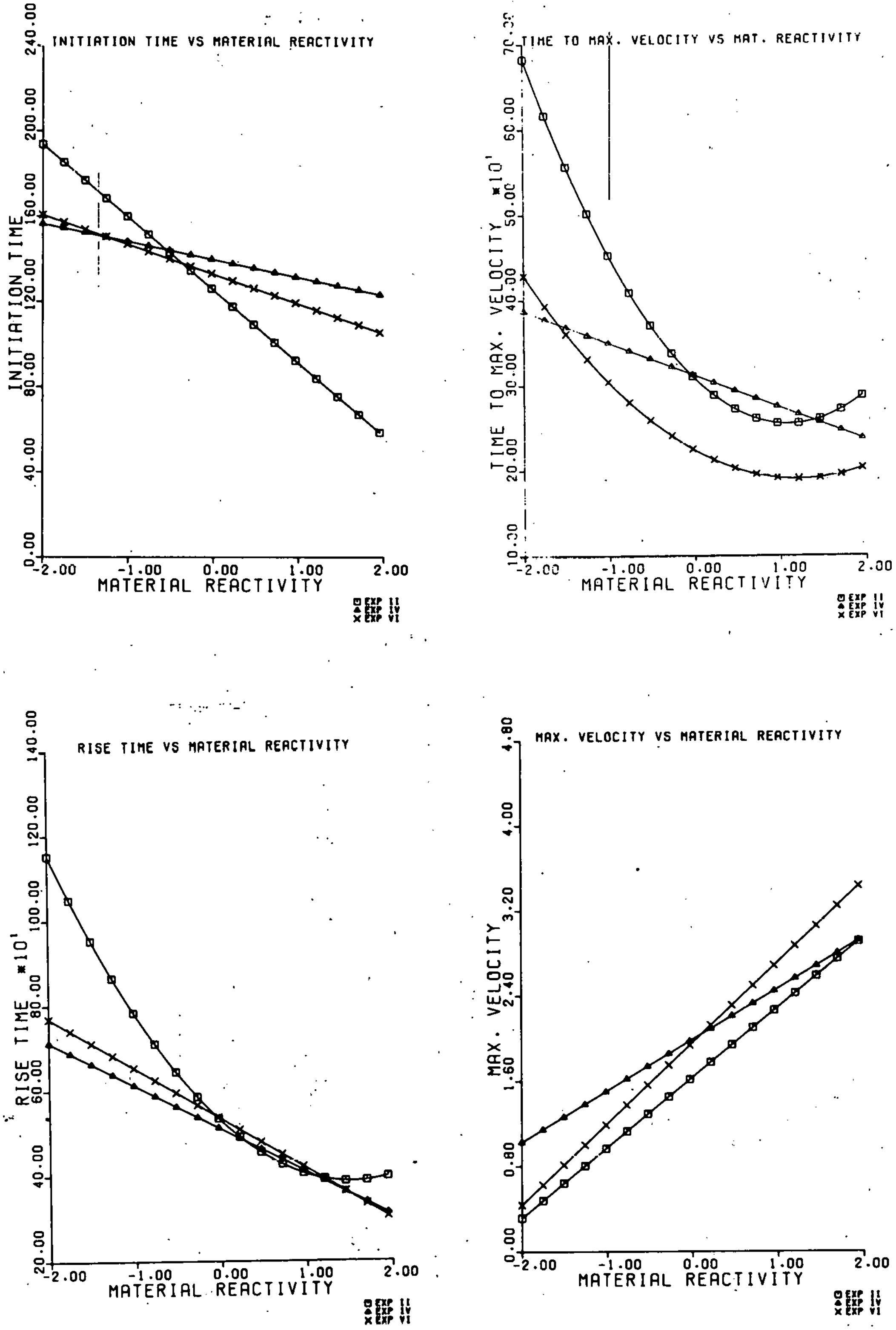


Figure 8. Rise-Rate and Gel Time Results Versus Material Reactivity (Reduced Computer Printouts.)

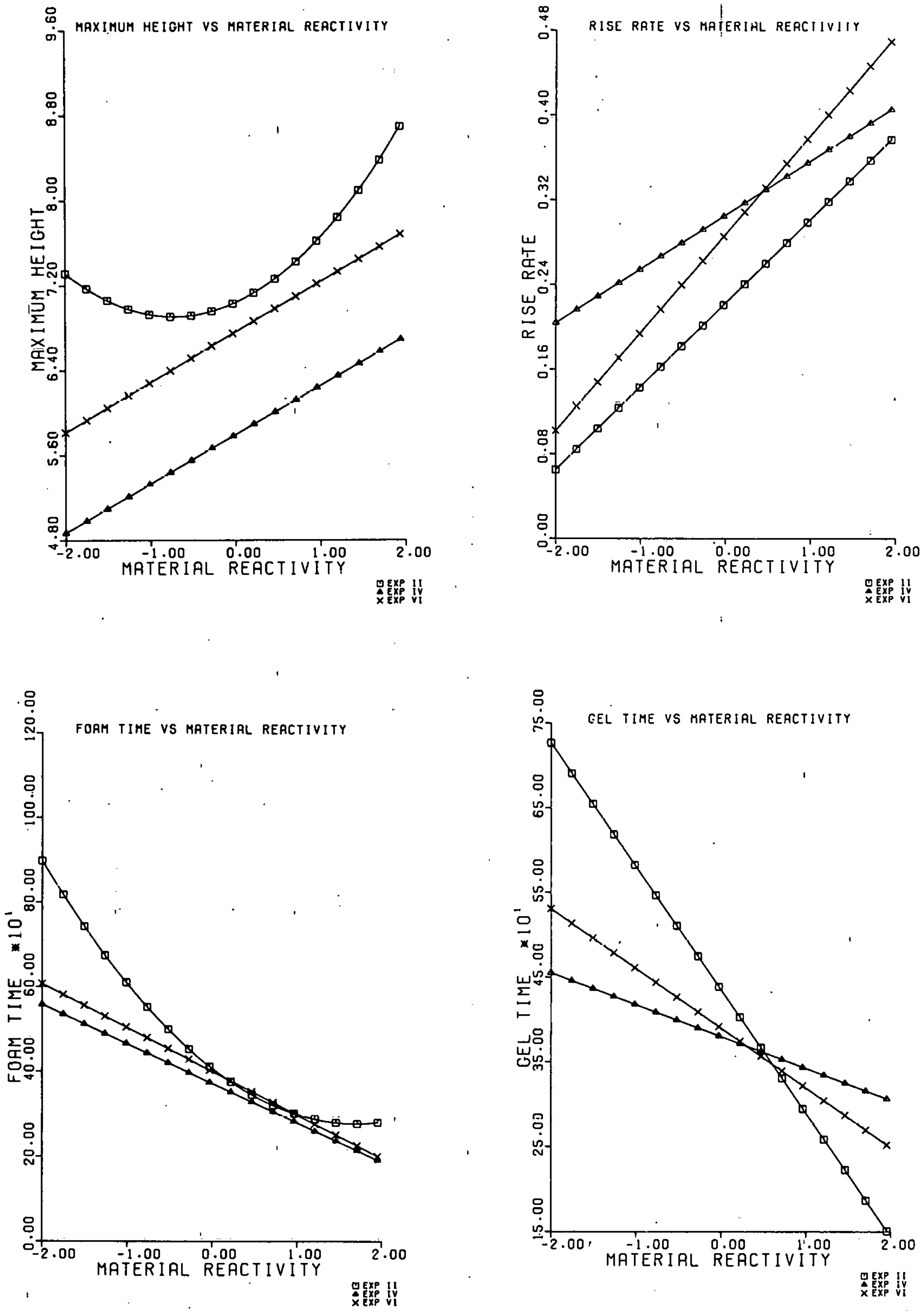


Figure 8 Continued. Rise-Rate and Gel Time Results Versus Material Reactivity

Test Blocks. Specimens 1.129 inches (28.68 mm) in diameter and 1.000 inch (25.40 mm) high were cut from the blocks, and compression tested at both room temperature and 325°F. The average compressive strength results for the various lines of Experiments 1 and 2 are listed in Tables 14 and 15, respectively. All values have been normalized to a density of 20 pounds per cubic foot.

Only two of the variables appeared to have any significant effect on the data. A low component temperature of 60°F (15°C) drastically reduced the compressive strength of the foam at the 325°F test temperature. This is, as yet, unexplained, but could very well be due to a compatibility problem or incomplete mixing caused by the lower reactivity of the material at this temperature. In Experiment 2, the compressive strength, especially at the elevated temperature, is reduced by a decrease in the mix ratio, (less isocyanate). This is caused by the creation of a less rigid polymer because of the low, 0.9 isocyanate index. Other strength differences which may appear are more readily attributable to test block density gradients than to the variables themselves. However, since only two of the variables were affecting the results, and then mostly only at elevated temperatures, this portion of the experiment was deleted on the remaining experiment on the basis that the effect of mix ratio and component temperature on compressive strength could be investigated later if warranted.

Free-Rise Samples. One-inch cube test samples were cut from the core of each of the billets used for rise-rate determinations. These specimens were then compression tested, both parallel and perpendicular to the direction of foam rise. The compressive strengths recorded on these tests, as shown in Tables 16 and 17, were so low that any attempt at comparison would be masked, since most of the differences are well within the range of the experimental error. This portion of the work was also abandoned for the rest of the experiments.

Text continued on page 54.

Table 14. Averages and Standard Deviations for Responses in Experiment 1--Test Blocks

Line No.	Design Matrix				Percent Response						
	X1	X2	X3	X4	6,	10	ULT	MOD	6,	10	MOD
					Room Temperature				325°F		
					Y11	Y12	Y13	Y14	Y15	Y16	Y17
1	-1	-1	-1	-1	923.7	899.5	933.3	32453.	329.1	347.2	14969.
					23.5	27.2	24.6	1377.	60.8	42.9	1961.
2	+1	+1	-1	-1	880.5	837.1	886.7	35041.	363.4	372.6	19061.
					29.1	28.6	28.6	2007.	11.8	11.8	632.
3	+1	-1	+1	-1	920.9	895.7	928.8	36562.	386.0	400.5	17925.
					9.5	6.9	8.4	1669.	8.9	4.6	2350.
4	+1	-1	-1	+1	875.6	842.5	892.6	34146.	348.0	363.0	18880.
					47.0	37.3	38.4	2587.	21.0	20.8	771.
5	-1	+1	+1	-1	917.4	899.4	923.4	33693.	327.7	343.2	15667.
					16.6	13.8	14.6	1975.	66.3	55.9	3617.
6	-1	+1	-1	+1	926.4	928.5	947.2	32730.	275.6	308.6	12424.
					10.7	9.6	12.1	1360.	68.3	64.6	2745.
7	-1	-1	+1	+1	935.8	913.3	944.2	33488.	364.5	365.8	17243.
					20.5	10.4	22.5	1561.	8.1	14.9	1167.
8	+1	+1	+1	+1	857.3	841.7	876.1	32897.	362.6	378.3	19148.
					25.7	26.0	26.4	1054.	10.0	7.5	644.
9	0	0	0	0	868.4	858.3	870.4	33785.	330.4	346.3	18699.
					21.9	21.3	19.9	1324.	18.0	17.1	1627.
10	0	0	0	0	861.0	860.1	871.8	32579.	331.8	348.0	18751.
					11.5	12.5	12.4	1440.	6.1	5.9	1416.
11	+2	0	0	0	Not Available						
12	-2	0	0	0	877.5	867.9	895.2	30419.	69.6	83.9	1996.
					35.6	37.7	29.0	3161.	12.3	11.6	457.
13	0	+2	0	0	846.8	833.0	848.2	32370.	313.8	330.7	17694.
					10.9	9.3	9.8	1294.	5.6	3.8	1351.
14	0	-2	0	0	854.8	856.9	874.2	31851.	315.1	330.2	17415.
					15.0	15.0	17.3	801.	9.2	9.2	2338.
15	0	0	+2	0	865.4	860.4	874.5	32511.	336.2	351.7	19026.
					11.8	7.6	9.8	1905.	9.5	8.1	1123.
16	0	0	-2	0	916.9	880.0	923.5	34175.	349.5	359.9	16535.
					9.6	19.7	10.2	991.	4.4	3.8	1575.
17	0	0	0	+2	867.8	842.2	874.0	31636.	337.2	350.5	16709.
					12.6	11.0	15.0	1127.	10.6	8.8	1442.
18	0	0	0	-2	938.4	914.2	944.5	34367.	345.2	360.6	18115.
					31.9	23.3	25.5	2713.	16.2	14.1	1353.

Table 15. Averages and Standard Deviations for the Responses in Experiment 2--Test Blocks

Design Matrix					Percent Response						
					6	10	ULT	MOD	6	10	MOD
					Room Temperature				325°F		
Line No.	X1	X2	X3	X4	Y11	Y12	Y13	Y14	Y15	Y16	Y17
1	-1	-1	-1	-1	942.5	932.5	955.2	33668.	206.6	221.4	8591.
					31.8	26.3	28.9	2319.	29.1	26.0	2037.
2	+1	+1	-1	-1	830.5	842.0	881.8	23783.	391.4	337.3	16306.
					25.9	29.2	24.2	3657.	19.4	24.6	1692.
3	+1	-1	+1	-1	877.3	875.1	906.1	27989.	361.5	363.3	17675.
					16.7	23.6	20.5	1935.	13.4	13.2	1644.
4	+1	-1	-1	+1	980.5	936.6	984.1	35751.	413.1	430.2	21539.
					11.2	10.5	10.4	1895.	5.1	5.4	1228.
5	-1	+1	+1	-1	880.5	863.4	886.1	32993.	168.0	182.5	6759.
					14.8	13.8	13.0	1914.	22.7	18.0	1645.
6 *	-1	+1	-1	+1	936.7	891.7	949.1	36206.	67.7	88.8	1605.
					20.0	17.0	20.3	1872.	14.4	12.1	656.
7 *	-1	-1	+1	+1	945.6	909.9	965.2	36418.	52.1	70.7	1190.
					3.9	5.7	3.9	1197.	11.5	12.1	327.
8	+1	+1	+1	+1	Not Available						
9	0	0	0	0	874.5	860.5	876.5	34120.	340.0	354.3	20021.
					5.3	5.0	5.8	684.	2.7	2.8	854.
10	0	0	0	0	894.5	865.5	901.2	32827.	338.5	350.5	15318.
					14.9	10.4	12.0	1909.	7.8	7.9	1332.
11	+2	0	0	0	994.4	959.9	1004.5	34366.	449.4	467.4	18497.
					18.4	18.9	12.3	2596.	16.0	18.8	2294.
12	-2	0	0	0	896.4	878.5	918.5	35220.	161.5	182.4	6578.
					5.9	19.9	8.6	1100.	13.6	10.5	693.
13	0	+2	0	0	873.2	856.4	888.9	34905.	360.8	375.2	19355.
					8.0	17.1	10.7	1095.	10.9	8.3	867.
14	0	-2	0	0	897.5	877.0	903.0	35042.	396.2	411.2	20204.
					22.8	20.0	24.4	1587.	14.0	13.2	1303.
15	0	0	+2	0	878.4	865.6	893.6	32326.	335.6	349.6	15556.
					16.8	5.7	5.4	1386.	6.9	3.8	644.
16	0	0	-2	0	888.4	866.3	905.9	33172.	333.9	347.9	16343.
					20.0	19.3	47.9	1308.	7.1	6.4	687.

* Temperature was greater than 325°F specified.

Table 16. Averages and Standard Deviations for the Responses in Experiment 1--Free-Rise Specimens

Design Matrix					Percent Response					
Line No.	X1	X2	X3	X4	6	10	MOD	6	10	MOD
					Parallel to Rise			Perpendicular to Rise		
					Y1	Y2	Y3	Y4	Y5	Y6
1	-1	-1	-1	-1	48.0	55.8	1182.	33.5	40.6	742.
					3.4	2.8	172.	1.2	1.3	28.
2	+1	+1	-1	-1	Not Available					
3	+1	-1	+1	-1	43.5	51.3	974.	31.2	39.1	590.
					1.6	1.7	47.	2.4	1.4	64.
4	+1	-1	-1	+1	36.8	47.4	758.	27.7	35.9	584.
					1.6	1.8	36.	1.7	1.5	48.
5	-1	+1	+1	-1	48.2	54.8	1154.	31.9	39.4	695.
					4.2	3.3	129.	2.7	3.0	63.
6	-1	+1	-1	+1	42.4	51.4	936.	29.9	37.3	646.
					3.0	2.0	94.	.9	.8	21.
7	-1	-1	+1	+1	Not Available					
8	+1	+1	+1	+1	40.2	50.0	861.	29.0	36.7	633.
					2.0	2.0	67.	1.1	1.0	31.
9	0	0	0	0	45.9	52.4	1120.	31.0	39.0	703.
					2.6	1.6	80.	2.0	1.6	43.
10	0	0	0	0	Not Available					
11	+2	0	0	0	38.8	47.2	898.	30.3	37.5	659.
					1.1	.8	45.	1.1	1.1	32.
12	-2	0	0	0	43.8	53.2	957.	30.2	38.9	676.
					2.6	3.0	73.	.8	1.4	19.
13	0	+2	0	0	43.0	49.9	964.	31.1	38.8	682.
					2.0	1.2	43.	1.1	1.1	28.
14	0	-2	0	0	41.9	49.3	927.	30.1	38.3	660.
					1.2	1.6	30.	1.9	2.0	41.
15	0	0	+2	0	44.8	50.1	1021.	31.6	38.5	698.
					1.4	1.1	44.	2.6	2.1	58.
16	0	0	-2	0	41.0	49.6	899.	31.4	40.1	692.
					1.9	1.4	58.	.6	.9	20.
17	0	0	0	+2	40.6	48.6	868.	27.3	34.7	600.
					2.5	1.9	70.	3.1	2.5	67.
18	0	0	0	-2	46.2	54.9	1038.	36.1	44.0	770.
					2.2	1.2	56.	1.0	2.0	25.

Table 17. Averages and Standard Deviations for the Responses in Experiment 2--Free-Rise Specimens

Design Matrix					Percent Response					
Line No.	X1	X2	X3	X4	6	10	MOD	6	10	MOD
					Parallel to Rise			Perpendicular to Rise		
					Y1	Y2	Y3	Y4	Y5	Y6
1	-1	-1	-1	-1	38.9	49.3	820.	28.6	38.2	618.
					1.5	1.1	35.	1.4	1.7	39.
2	+1	+1	-1	-1	42.9	50.8	967.	34.9	44.0	756.
					2.3	1.3	73.	1.2	2.2	31.
3	+1	-1	+1	-1	42.7	51.1	977.	34.7	42.9	763.
					2.3	2.6	85.	3.0	2.8	69.
4	+1	-1	-1	+1	46.1	56.4	1074.	33.3	41.3	732.
					1.3	2.5	52.	1.5	1.7	51.
5	-1	+1	+1	-1	41.3	48.5	861.	30.7	39.0	658.
					2.0	2.4	70.	1.9	2.1	63.
6	-1	+1	-1	+1	48.4	60.1	1102.	27.7	36.4	625.
					4.2	4.5	108.	2.3	2.8	54.
7	-1	-1	+1	+1	43.3	57.4	963.	27.3	35.6	590.
					3.2	4.2	66.	1.8	2.5	43.
8	+1	+1	+1	+1	44.5	56.0	1000.	31.6	40.9	688.
					1.6	2.3	48.	1.5	1.6	35.
9	0	0	0	0	42.9	52.1	944.	29.9	38.7	641.
					1.7	1.2	55.	3.3	2.6	73.
10	0	0	0	0	Not Available					
11	+2	0	0	0	45.3	53.2	990.	36.8	46.6	788.
					1.6	2.3	37.	1.6	2.0	45.
12	-2	0	0	0	42.7	55.1	895.	25.8	33.0	585.
					4.3	5.1	94.	3.4	2.8	155.
13	0	+2	0	0	42.3	50.9	937.	31.7	39.8	688.
					1.6	1.2	48.	1.3	.9	34.
14	0	-2	0	0	43.9	51.3	995.	31.7	39.6	705.
					2.2	1.0	59.	.7	.6	25.
15	0	0	+2	0	43.9	51.7	997.	32.2	39.6	696.
					3.2	1.8	75.	1.0	1.2	24.
16	0	0	-2	0	42.6	51.0	947.	32.9	41.0	729.
					1.7	1.4	49.	1.2	1.4	37.
17	0	0	0	-1	42.7	49.7	957.	31.7	39.8	680.
					2.8	1.5	70.	1.9	1.8	36.

ACCOMPLISHMENTS

Of the eight material and processing variables studied, component temperature, mix time, mix speed, water concentration, mix ratio, mix quantity, pour weight, and material reactivity, only four--the component temperature, water concentration, mix ratio, and material reactivity--significantly altered the rise rate and gel time characteristics of the materials. While the exact effect of these variables on all foam systems is not predictable from this work, the trend has been established and all similar materials should conform to these patterns.

Component temperature is one of the most significant variables. To obtain reproducible rise-rate and gel time results, the component temperature of the materials should be maintained at least within 2° F (1°C) of the specified values.

The water concentration of the resin is of minor significance in rise-rate testing. A moderately large change in the amount of water in the foam produces a relatively small change in the rise-rate and gel time results. Present material specifications provide sufficient control over the resin-water concentrations to yield reproducible rise-rate results. Rise-rate testing can only detect water concentration variations that are significantly outside the presently prescribed limits.

The mix ratio, while a more significant variable than the water concentration, is not as influential as the component temperature in its effect on the rise-rate properties of foams. Present material specifications usually instruct that the mix ratios be maintained within 1 percent of the indicated values. This percentage is normally with reference to the mix weights and, as such, the actual mix ratio variations possible are sufficiently less than changes represented by the levels in the study. Thus, present material specifications should provide adequate control on mix ratios for rise-rate testing purposes.

The material reactivity (catalyst concentration) is the most significant variable affecting the rise-rate characteristics of the foams. However, all of the foam systems were not equally affected by equivalent change in catalyst concentration. Toluene diisocyanate and polyester polyol foams are generally more susceptable to catalyst level changes than are polymeric isocyanate and polyether polyol foams. In any case, rise-rate testing may provide an efficient tool for checking the presently undefined catalyst level in urethane foam systems

The effect of the four referenced variables on the gel time of the foam was similar to the effect those variables had on the rise-rate characteristics; specifically, the time to maximum velocity and rise time. Three of these variables produced gel time changes equivalent to the changes in rise-rate characteristics. Only one variable, the material reactivity or catalyst concentration, caused the gel time to change at a different rate than the rate of change experienced by the rise-rate characteristics, thereby indicating that the rise-to-gel ratio of foams can be significantly altered by proper catalyst and catalyst level selection.

The compressive strength of the foam was not significantly affected by six of the eight variables. Only changes in the mix ratio and component temperature caused the compressive strength to change. As the mix ratio decreased, the compressive strength, especially at elevated temperatures, decreased. This was caused by the less rigid polymer developed as the percentage of isocyanate, or the isocyanate index, decreased. A low component temperature of 60°F (15°C) drastically reduced the compressive strength of the foam at 325°F (160°C) test temperature. This might be caused by a compatibility problem or incomplete mixing of the components due to the lower material reactivity at this temperature.

FUTURE WORK

A brief examination of the effect of low component temperatures on the physical properties of urethane foam is recommended. This study should include not only the effect of the component temperature on these properties but also the reason or cause for such an effect.

An evaluation of the effect of various catalysts and catalysts concentration on the rise-to-gel ratio of urethane foams should definitely be undertaken.

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REFERENCES

[1] Material Specification 2170402: Polyurethane Foam, Rigid, 4 Lb/Ft^3 Bendix Corporation, Kansas City Division, (October, 1969).

[2] Material Specification 2170562-2170568: Polyurethane Foam, Rigid, Heat Resistant, Bendix Corporation, Kansas City Division, (June, 1970).

[3] Process Development Order 6989016: Evaluation of the Repeatability of the Polytron Rise-Rate Machine, Bendix Corporation, Kansas City Division, (January, 1969).

[4] Roger Jennings, "Instrumental Analysis of the Performance of Rigid Urethane Foaming Systems," Journal of Cellular Plastics, Vol. 5, No. 3, (May through June, 1969).

[5] Process Specification 9952034: Testing Rise-Rate, Rigid Urethane Foam, Bendix Corporation, Kansas City Division, (July, 1969), pp 159-172.

[6] Calvin J. Benning, "Plastic Foams, Volume I, Chemistry and Physics of Foam Formation," John Wiley & Sons, Inc, New York, N. Y., 1969, pp 119-121.

[7] J. M. Buist and H. Gudgeon, Advances in Polyurethane Technology, New York: John Wiley and Sons, p 8, (1973).

[8] R. K. Traeger, "Physical Properties of Rigid Urethane Foams," Journal of Cellular Plastics, Vol. 3, No. 9, (September, 1967), pp 405-418.