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BDX-613-1836 (Rev.)

MATERIAL EVALUATION OF POLYURETHANE FOAM,
0.05 g/cm³ DENSITY

By J. R. Fender
Department 861

Published March 1978

Topical Report

Prepared for the United States Department of Energy
Under Contract Number EY-76-C-04-0613.

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**Kansas City
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Printed in the United States of America

Available From the National Technical Information Service, U.S. Department of Commerce, 5285 Port Royal Road, Springfield, Virginia 22161.

Price:	Microfiche	\$3.00
	Paper Copy	\$4.50

BDX-613-1836 (Rev.)
Distribution Category UC-25

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Topical Report
J. R. Fender, Project Leader

Project Team:
S. L. DeGisi
F. N. Larsen
T. E. Neet
B. C. Cole
L. E. Lacy
G. W. Griffith
R. W. Palmer
J. E. Anderson, Jr.

Communications Services

**Kansas City
Division**

MATERIAL EVALUATION OF POLYURETHANE FOAM, 0.05 g/cm³ DENSITY

BDX-613-1836 (Rev.), UNCLASSIFIED Topical Report, Published March 1978

Prepared by J. R. Fender, D/861

Rigifoam 6003-2, a Bendix manufactured CO₂-blown polyester polyurethane foam system, was chosen for use as the prime material for production of parts with a foam density of 0.05 g/cm³. Studies of the 6003-2 physical properties, chemical analysis, dimensional stability, moisture content, and the machining and gaging capabilities of this material are summarized. The next phase of this project will include the development of processing and stabilization techniques required to consistently fabricate quality foam billets which have no rejectable voids or streaks.

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The Bendix Corporation
Kansas City Division
P. O. Box 1159
Kansas City, Missouri 64141

A prime contractor with the United States Department of Energy under Contract Number EY-76-C-04-0613

CONTENTS

Section	Page
SUMMARY.	7
DISCUSSION	9
SCOPE AND PURPOSE.	9
PRIOR WORK	9
ACTIVITY	9
<u>Formulation</u>	10
<u>Chemical Analysis</u>	14
<u>Physical Properties</u>	19
<u>Dimensional Stability</u>	22
<u>Moisture Content</u>	28
<u>Machining and Gaging</u>	36
<u>Processing Techniques</u>	36
ACCOMPLISHMENTS.	37
FUTURE WORK.	38
APPENDICES	
A. TESTING RISE-RATE, RIGID URETHANE FOAM, DWG NUMBER 9952034	40
B. IONIC CHLORINE PROCEDURE	50
C. ASH CONTENT PROCEDURE.	52
DISTRIBUTION	54

ILLUSTRATIONS

Figure		Page
1	Compressive Strength of 6003-2, Tested Parallel to Rise.	21
2	Compressive Modulus of 6003-2, Tested Parallel to Rise.	22
3	Compressive Strength of 6003-2, Tested Perpendicular to Rise	23
4	Compressive Modulus of 6003-2, Tested Perpendicular to Rise	24
5	Tensile Strength of 6003-2, Tested Parallel to Rise.	25
6	Tensile Modulus of 6003-2, Tested Parallel to Rise.	26
7	Tensile Strength of 6003-2, Tested Perpendicular to Rise	27
8	Tensile Modulus of 6003-2, Tested Perpendicular to Rise	28
9	Ultimate Compressive and Tensile Strength Versus Temperature of 6003-2.	29
10	Dimensional Change With Time of Billets 330 mm in Diameter by 330 mm High	30
11	Effect of Machining on Dimensional Change With Time of Billets 330 mm in Diameter by 330 mm High	31
12	Changes in Dimensions and Weight of Stabilization Billets With Time	32
13	Changes in Dimensions With Time of Stabilization Billets 159 mm High	34
14	Equilibrium Water Content of 6003-2	36
15	Moisture Loss With Time of 6003-2, Stabilized at 59-Percent Relative Humidity When Exposed to Zero-Percent Relative Humidity	37

16	Moisture Pickup of 6003-2, Stabilized at Zero-Percent Relative Humidity When Exposed to 55-Percent Relative Humidity . .	38
17	Effect of Moisture on 6003-2 Billets.	39

TABLES

Number		Page
1	Amino Catalysts.	11
2	Formulations of Rigifoam 6003-2.	12
3	Effect of Catalyst on Compressive Strength . .	15
4	Effect of Catalyst on Tensile Strength	17
5	Elemental Analysis of Two Lots of Rigifoam 6003-2.	19
6	Analysis Data of Four Foam Samples	20

SUMMARY

This project was initiated to develop the production capability to pour, machine, and gage polyurethane foam parts with a density of 0.05 g/cm^3 . Specific areas to be studied include material formulation, chemical analysis, physical properties, dimensional stability, moisture content, machining and gaging techniques, and processing techniques.

Past experience with foams of less than 0.1 g/cm^3 density was limited to development activity. This work indicated problems with both voids and streaks in the material which would require control before the material could be successfully used in production.

A review of material properties data and literature, and analysis of the design requirements established the Bendix Rigifoam 6003 foam system as the prime material candidate for production application. Additional analysis revealed a potential compatibility problem between other components and foam containing the standard amine catalyst. To resolve this problem, the amine was removed from the formulation entirely, and processing characteristics and physical and chemical properties were evaluated.

This study showed little difference between the catalyzed and uncatalyzed foams. Therefore, the uncatalyzed version, designated Rigifoam 6003-2, became the baseline material for the remaining work on the project.

Other formulations that appear to be successful in preventing amine outgassing from the finished foam material include substitution of dimethylethanolamine (DMEA) for the N-methylmorpholine (NMM) and incorporation into the formulation of various resins containing catalytic nitrogen within their structure.

Chemical analysis of the 6003-2 material was as expected for a polyurethane foam material and provided the information required to establish a specification limit of 300 parts per million (ppm) for ash content of production foam material.

Determination of Rigifoam 6003 physical properties proved the material to be capable of meeting requirements. Strengths were determined by testing 0.0000164 m^3 samples in both tension and compression. Some of the conclusions of this study follow.

- Density has a pronounced effect upon physical properties. The strengths of material with a density of 0.06 g/cm^3 are nearly double those of 0.04 g/cm^3 material.

- Strengths obtained in the parallel-to-foam-rise direction are consistently higher than those obtained in the perpendicular-to-rise direction.
- Tensile and compressive strengths show some loss as a result of increasing test temperature, but both values are adequate up to 54°C.

Dimensional stability measurements made on cylinders 300 mm in diameter by 330 mm high and on contoured stabilization blanks 273 mm in diameter by 159 mm high indicated that shrinkage continued to take place over the entire testing period, which was up to 230 days. The shrinkage was more rapid at first and reduced with time. These data were used to establish a 60-day minimum stabilization time for the foam stabilization blank.

Moisture caused the foam to grow, and the effect appeared to be rapid and reversible. Moisture pickup appeared to be more rapid than did moisture loss.

Evaluation of machining and gaging capabilities indicate that no special handling, tools, or procedure will be required.

Future activity will include a continuation of stabilization data, evaluation of expediting stabilization by oven post cure, and review of the effect of variation of the resin-to-isocyanate ratio. In addition, a machine mixing process will be developed.

DISCUSSION

SCOPE AND PURPOSE

The purpose of this project is to develop the production capability to pour, machine, and gage polyurethane foam parts with a density of 0.05 g/cm^3 . This report discusses the activity surrounding the evaluation of Bendix Rigifoam 6003-2.

PRIOR WORK

Prior work with foam in the low-density range specified was performed approximately 10 years ago. This prior effort supported laboratory paid orders and progressed far enough to identify problem areas requiring further concentrated development effort.

ACTIVITY

A review of material properties data and literature, and analysis of design requirements led to the conclusion that Bendix Rigifoam 6003 would be the best available candidate material for production parts of polyurethane foam with a density of 0.05 g/cm^3 . Work on this project was, therefore, started with Rigifoam 6003-2.5A as the prime material for study. The project plan was established to evaluate the 6003 foam system in the following areas.

- Formulation,
- Chemical Analysis,
- Physical Properties,
- Dimensional Stability and Moisture Content,
- Machining and Gaging, and
- Processing Techniques.

Summaries of progress and activity in each of these areas follow.

Formulation

Very soon after initiation of activity on this project it was discovered that a potential compatibility problem existed in an assembly as a result of traces of amine introduced by the foam parts. The amine, N-methylmorpholine (NMM), is added to the resin component of the foam system and serves as a catalyst for the foaming reactions. The amine remains with the foam material after foaming is complete.

Outgassing tests on the Rigifoam 6003-2.5A, per MS 2170576, using the mass spectrometer, proved the presence of amine in the sample but were not quantitative.

Solvent extraction procedures and subsequent analysis by gas chromatography to resolve NMM from the solvent mixture gave good experimental results. Dimethylformamide (DMF) was found to swell the foam and promote disintegration during extraction. DMF also extracted NMM more completely and rapidly than other solvents investigated. Benzene used as an internal standard improved precision. This test method established an average NMM content of the 6003-2.5A foam of 1200 ppm.

Several methods of removing the amine from the foamed material were tried. Heating the foam sample in a vacuum oven for 16 hours at 60°C did not significantly decrease the NMM content. Samples of Rigifoam 6003-2.5A which had been stored in desiccated containers for 235 days were found to contain 1040 ppm NMM. Oven aging at 80°C in an air-circulating oven gave only slight reduction to 975 ppm.

It was concluded that loss of NMM from the foam occurred slowly and that acceleration of its removal by thermal or vacuum treatment would be ineffective.

Elimination of amine outgassing was approached by three different routes. Complete elimination of the amine catalyst from the formulation was one approach. The second method was replacement of the catalyst by a reactive amine catalyst, dimethylethanolamine, and the third was an incorporation of amine-containing polyethers into the formulation. These last two approaches would chemically bind the amine into the foam structure. The catalytic materials investigated are listed in Table 1.

BKC 1101 resin, a pentaerythritol/E-caprolactone polyester, and BKC 1201 T-Component, a toluene diisocyanate/BKC 1101 prepolymer are the principal components in the foam. The water and surfactant contents were maintained at the same levels as in Rigifoam 6003-2.5A. The catalytic material concentrations were adjusted by trial to match the rise-rate constant within

Table 1. Amino Catalysts

Designation	Manufacturer	Type	Hydroxyl Number
Poly G 357 SA	Olin Chemicals	Polyether	350
Pluracol 595	BASF Wyandotte	Polyether	415
Pluracol 355	BASF Wyandotte	Polyether	455
Poly G 71-530	Olin Chemicals	Polyether	530
Poly G 70-600	Olin Chemicals	Polyether	600
Dimethylethanolamine	Union Carbide	Aminoalcohol	630

±5 percent, as determined per 9952034 (Appendix A) for Rigidfoam 6003-2.5A. The formulations investigated, together with the rise-rate characteristics and mechanical strength properties, are given in Table 2. To facilitate comparison of the mechanical properties, the data have been normalized to a density of 51 kg/m³.

The rise-rate characteristics of Rigidfoam 6003-2.5A with a resin component containing 0.4 parts-by-weight (pbw) of NMM can be duplicated by substitution of 0.2 pbw of dimethylethanolamine (DMEA) for the NMM. With the aminopolyethers, significantly shorter initiation times and times to maximum velocity were obtained. The noncatalyzed material is, as expected, a slower reacting system, having longer initiation time, rise time, and time to maximum velocity as well as reduced maximum height, maximum velocity, and rise-rate constant. Heating of the noncatalyzed components significantly reduced the initiation time, rise time, and time to maximum velocity. An increase in maximum velocity was obtained, accompanied by a decrease in maximum height. It was concluded that heating of the components increased the urethane reaction at a faster rate than the blowing reaction.

Compressive strength, compressive modulus, and tensile strength determined on all the formulations are also shown in Table 2. The compressive properties for the polyether-catalyzed materials are, in general, slightly lower than those of the NMM-catalyzed materials. The noncatalyzed and DMEA-catalyzed material have essentially the same strength and modulus as the NMM-catalyzed material. The foam blocks from which these data were obtained were molded in small molds (200 by 200 by 80 mm) with direction of rise in a 200-mm direction. This may have resulted in some cell distortion and density nonuniformity with a detrimental effect on the mechanical strength. To eliminate these effects, foam billets were cast in large molds (200 by 200 by 350 mm), and

Table 2. Formulations of Rigifoam 6003-2

Components	Parts by Weight							
Distilled Water	6.7	6.7	6.7	6.7	6.7	6.7	6.7	6.7
L5320	2.0	2.0	2.0	2.0	2.0	2.0	2.0	2.0
Surfactol 365	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0
BKC 1101 Resin	100.0	100.0	100.0	96.0	95.0	90.0	85.0	85.0
Poly G 357 SA	---	---	---	---	---	---	15.0	---
Poly G 71-530	---	---	---	---	---	---	---	15.0
Poly G 70-600	---	---	---	4.0	---	---	---	---
Pluracol 355	---	---	---	---	5.0	---	---	---
Pluracol 595	---	---	---	---	---	10.0	---	---
N-methyl- morpholine	0.4	---	---	---	---	---	---	---
Dimethylethanol- amine	---	---	0.2	---	---	---	---	---
BKC 1201 Prepolymer	276.2	275.2	275.7	277.4	275.5	272.2	267.1	274.4
Rise-Rate Characteristics per 9952034, 32 kg/m ³ , Class A, 25°C								
Initiation Time(s)	94	110*	96	78	75	78	77	82
Rise Time(s)	281	360*	285	276	264	285	233	303
Time to Maximum Velocity(s)	162	185*	160	145	140	139	136	135
Max. Height (m)	0.111	0.076*	0.110	0.110	0.100	0.107	0.112	0.104
Max. Velocity (m/s)	0.067	0.027*	0.065	0.070	0.061	0.061	0.066	0.059
Rise-Rate Constant	0.60	0.35*	0.59	0.63	0.61	0.57	0.59	0.58

Table 2. Formulations of Rigifoam 6003-2

Mechanical Properties at 24 ±6°C, Normalized to 51 kg/m ³								
Compressive								
Strength (kPa)	345	345	379	317	290	276	290	352
Compressive								
Modulus (kPa)	7653	7377	8687	6964	5998	5792	5998	7171
Tensile								
Strength (kPa)	517	524	579	648	552	593	579	552

*At 32°C

specimens were cut from the core of the billets. Foam catalyzed with NMM (Rigifoam 6003-2.5A), noncatalyzed foam (Rigifoam 6003-2), and foam catalyzed with DMEA were included in this test series.

The compressive and tensile data are shown in Tables 3 and 4, respectively. These data indicate that the mechanical strength of the noncatalyzed Rigifoam 6003-2 is equal to that of NMM catalyzed Rigifoam 6003-2.5A. Rigifoam 6003 which is catalyzed with DMEA appears to be somewhat stronger than NMM catalyzed Rigifoam 6003-2.5A.

Samples of NMM-catalyzed Rigifoam 6003-2.5A, noncatalyzed Rigifoam 6003-2, DMEA-catalyzed Rigifoam 6003, and Rigifoam 6003 catalyzed with each of the amino-polyethers were subjected to mass spectrometer outgassing tests. The only samples which outgassed any detectable amine catalyst were the NMM-catalyzed Rigifoam 6003-2.5A samples. These data support the conclusion that amine catalysts containing reactive hydroxyl groups are chemically bound into the polyurethane structure and will not migrate out into the surrounding atmosphere.

Since the noncatalyzed Rigifoam 6003-2 was shown to have mechanical properties equal to those of the NMM-catalyzed material, and no amine outgassing could be detected with the noncatalyzed material, Rigifoam 6003-2 was selected as the material for the polyurethane foam. Rigifoam 6003-2, mixed by using batch-making techniques, is currently being used in production.

Chemical Analysis

Elemental analysis of foam samples from two lots of Rigifoam 6003-2 produced data typical of polyurethane materials, which are shown in Table 5. Spectroscopic analysis of the ash from these lots showing trace elements is included in the data.

Table 6 gives the results of analysis of four additional foam billets. Numbers shown are average values of multiple samples. Billets X012 and X020 were poured from catalyzed 6003, and X050 and X056 from uncatalyzed material.

The laboratory procedures used for determining ionic chlorine and ash content are included as Appendix B and Appendix C, respectively.

Sulfur and phosphorus were below detection limits in all samples.

It was concluded from this activity that the 6003 system meets the chemical requirements and the studies provided the information required to establish a 300 ppm ash content limit for the foam material.

Table 3. Effect of Catalyst on Compressive Strength

Material	Perpendicular to Rise			Parallel to Rise		
	Density (g/cm ³)	Compressive		Density (g/cm ³)	Compressive	
		Strength (kPa)	Modulus (kPa)		Modulus (kPa)	Strength (kPa)
Rigifoam 6003-2 (No Catalyst)	0.0462	255	6047	0.0466	5743	255
	0.0530	386	8412	0.0473	6267	303
	0.0474	303	6267	0.0490	6895	331
	0.0458	269	5743	0.0502	7653	359
	0.0502	331	7171	0.0490	7033	324
	0.0518	372	7860	0.0506	7860	359
	0.0529	379	7860	0.0492	7998	365
	0.0514	345	7171	0.0492	7308	352
	0.0490	310	6633	0.0487	7033	331
	0.0497	324	7033	0.0466	5943	290
Average	0.0494	327	7019	0.0486	6971	327
Rigifoam 6003-2.5A (N-methylmorpholine Catalyst)	0.0505	352	7653	0.0478	7308	317
	0.0465	276	6385	0.0510	8619	379
	0.0473	283	6385	0.0498	7998	352
	0.0532	379	8205	0.0508	8825	379
	0.0503	345	7653	0.0470	7171	290
	0.0510	359	7860	0.0498	8205	359
	0.0463	269	6157	0.0513	9101	379
	0.0530	372	7998	0.0510	8619	379
	0.0506	331	7308	0.0500	8412	359
	0.0502	338	7308	0.0498	8205	359
Average	0.0499	330	7295	0.0498	8246	355

Table 3 Continued. Effect of Catalyst on Compressive Strength

Material	Perpendicular to Rise			Parallel to Rise		
	Density (g/cm ³)	Compressive		Density (g/cm ³)	Compressive	
		Strength (kPa)	Modulus (kPa)		Modulus (kPa)	Strength (kPa)
Rigifoam 6003	0.0505	393	9101	0.0494	9584	386
(Dimethylethanolamine	0.0470	310	7308	0.0473	9308	359
Catalyst)	0.0498	372	8412	0.0495	9860	386
	0.0484	352	8205	0.0462	8825	345
	0.0468	283	6502	0.0508	9860	400
	0.0498	352	7860	0.0495	9584	386
	0.0497	331	7515	0.0511	9308	400
	0.0498	338	7860	0.0511	9584	407
	0.0510	400	9101	0.0511	9584	400
	0.0513	400	9584	0.0498	10135	386
Average	0.0494	353	8143	0.0496	9563	385

Table 4... Effect of Catalyst on Tensile Strength

Material	Perpendicular to Rise			Parallel to Rise		
	Density (g/cm ³)	Tensile Strength (kPa)	Tensile Modulus (kPa)	Density (g/cm ³)	Tensile Strength (kPa)	Tensile Modulus (kPa)
Rigifoam 6003-2 (No Catalyst)	0.0471	474	9791	0.0506	554	15996
	0.0526	555	15169	0.0468	545	15858
	0.0484	521	13652	0.0494	545	16892
	0.0470	465	11928	0.0495	536	16548
	0.0482	490	12893	0.0495	579	17582
	0.0471	478	10342	0.0495	520	16823
	0.0498	482	12618	0.0506	552	17237
	0.0494	452	11583	0.0506	592	15858
	0.0470	418	8963	0.0502	521	16892
	0.0527	465	17237	0.0494	517	16892
Average	0.0489	480	12418	0.0496	546	16658
Rigifoam 6003-2.5A (N-methylmorpholine Catalyst)	0.0478	448	12411	0.0479	601	16548
	0.0489	500	14479	0.0495	598	16754
	0.0484	505	12273	0.0492	647	17789
	0.0513	563	14824	0.0497	653	17582
	0.0494	454	12549	0.0524	607	16961
	0.0486	453	12066	0.0522	634	18961
	0.0527	473	15169	0.0486	558	17099
	0.0510	463	13927	0.0514	644	17926
	0.0492	517	14341	0.0524	586	17789
	0.0478	487	11859	0.0495	607	18961
Average	0.0495	486	13390	0.0503	614	17637

Table 4 Continued. Effect of Catalyst on Tensile Strength

Material	Perpendicular to Rise			Parallel to Rise		
	Density (g/cm ³)	Tensile Strength (kPa)	Tensile Modulus (kPa)	Density (g/cm ³)	Tensile Strength (kPa)	Tensile Modulus (kPa)
Rigifoam 6003-2.5	0.0479	496	13790	0.0479	577	24132
(Dimethylethanolamine	0.0492	465	15513	0.0478	607	19995
Catalyst)	0.0484	498	12962	0.0486	618	20340
	0.0511	645	18961	0.0479	590	18961
	0.0490	593	15169	0.0519	658	20340
	0.0502	494	14824	0.0522	663	18961
	0.0482	531	13445	0.0514	628	20340
	0.0479	467	11376	0.0486	630	24821
	0.0502	538	12824	0.0514	545	19857
	0.0514	674	17926	0.0474	603	20202
Average	0.0494	540	14679	0.0495	612	20795

Table 5. Elemental Analysis of Two Lots of
Rigifoam 6003-2

Major Constituents	Lot 449	Lot 452
	Percent	
Carbon	59.58	59.97
Hydrogen	6.06	6.03
Nitrogen	9.62	10.21
Oxygen (By Difference)	24.74	23.79
<hr/>		
Trace Elements	Parts Per Million	
Ash Content	73	82
Silicon	21	52
Manganese	0.03	0.02
Magnesium	0.19	0.43
Tin	ND*	0.33
Chromium	0.06	0.04
Iron	1.2	0.41
Aluminum	0.61	0.3
Calcium	1.2	1.6
Copper	0.07	0.09
Zinc	0.17	0.15
Titanium	0.1	0.07
Lead	0.04	0.14
Nickel	0.07	0.05
<hr/>		
*None Detected		

Physical Properties

Studies have been conducted to evaluate the effect of changes in foam density, foam rise direction, and test temperature on compressive and tensile strength and modulus.

Compressive tests were run, using 0.0000164 m^3 samples, 28.7 mm in diameter by 25.4 mm high, machined from various locations within the foam billet. Figures 1 through 4 were prepared from data obtained by testing samples from billets targeted at 0.04, 0/05, and 0.06-g/cm^3 density.

Table 6. Analysis Data of Four Foam Samples

Chemical	Samples			
	X012	X020	X050	X056
	Content (ppm)			
Ash	194	190	93	38
Total Chlorine	46	125	105	71
Ionic Chlorine	15	Not Tested	5	4
Sulfur	BD*	BD	BD	BD
Phosphorus	BD	BD	BD	BD
Silicon	79	66	47	18
Manganese	0.04	0.05	BD	0.01
Iron	0.8	1.7	0.8	0.4
Magnesium	1.7	1.8	1.0	0.4
Chromium	0.12	0.2	BD	0.01
Nickel	BD	0.36	BD	BD
Aluminum	0.7	1.2	0.7	0.3
Calcium	2.9	4.7	2.6	0.8
Titanium	0.5	0.2	0.2	BD
Copper	0.06	0.29	0.22	0.07
Zinc	0.44	1.2	0.26	0.17
Lead	0.51	0.35	0.13	0.06
Boron	1.2	3.1	5.1	0.8
Potassium	4.1	3.5	0.17	0.9
Sodium	2.3	3.6	6.9	3.4
*Below Detection				

Tensile properties were determined from specimens similar to the compressive specimens prepared for testing by bonding between two aluminum end adapters, using a room temperature curing rigid epoxy system mixed in the ratio of 10 pbw Epibond 104 and 1 pbw Hardener 951, manufactured by Furane Plastics, Inc. Tensile properties are shown in Figures 5 through 8.

Figure 1. Compressive Strength of 6003-2, Tested Parallel to Rise

It can be seen from these data that strength and modulus are highly dependent upon foam density, and that the strength and modulus of 0.06-g/cm³ material are approximately double those of 0.04-g/cm³ material. These illustrations also show that parallel-to-rise properties are consistently higher than those tested perpendicular to rise, a condition that is classical with urethane foam materials. It is postulated that this condition arises from elongation of the foam cells during foam rise, giving reduced strengths as the cells become less spherical.

These data have been used to reduce the testing required in qualifying the production foam billets. Since minimum tensile strength is the acceptance criteria, it was decided to

Figure 2. Compressive Modulus of 6003-2, Tested Parallel to Rise

test in only the weakest direction (perpendicular-to-rise), knowing that if these samples meet the minimum strength requirement, the strongest (parallel-to-rise) samples would always meet the requirement, making their actual testing unnecessary.

To determine the effect of temperature upon ultimate tensile and compressive strength, 60 each 0.0000164 m³ test specimens were taken from the test specimen area of a series of foam billets. Ten samples each were tested to determine compressive and tensile strengths at room temperature, at 54°C, and at -40°C. The results on Figure 9 show that tensile strength is affected less than compressive strength and that neither shows a major loss in strength up to the 54°C maximum testing temperature.

Dimensional Stability

Previous studies have shown that polyurethane foam billets and molded-to-size parts continue to shrink over a period of time. The exact nature of this shrinkage is dependent upon many factors including material density, part configuration, and environment.

Figure 3. Compressive Strength of 6003-2, Tested Perpendicular to Rise

Several of the factors involved in stabilization of the 6003-2 foam system have been studied and are described in the following sections.

The first dimensional stability study was made by taking periodic measurements of the height and diameter of 330 by 330 mm billets, rough machined from the center of 0.05 g/cm³ 6003-2 cast billets, 508 mm high by 457 mm in diameter. Two flats were cut on the diameter to allow repeatability of measurement. The billets were measured in the dry inspection area (less than 15-percent RH corrected to 20°C) and stored in desiccated containers between measurements. Figure 10 depicts the shrinkage of two representative billets for 200 days after rough machining. There appears to be

Figure 4. Compressive Modulus of 6003-2, Tested Perpendicular to Rise

a change in rate at about 130 days and a relatively stable condition after approximately 170 days. Total shrinkage is approximately the same for the height and diameter measurements.

Figure 11 traces the change in billet height of three additional billets for 75 days. These billets were then machined into sections 159 mm high by 273 mm in diameter, and measurements were reinitiated after machining. It was surprising to note the change in shrinkage rate of the smaller sized billet. This change in rate would indicate that more rapid stabilization would be possible by using the smaller billet size.

A second study was started with the smaller billet which included a scooped out area in the center to remove as much material as possible to establish a stabilization blank from which both parts could be machined. Each large billet produces two stabilization blanks with the top blank labeled A and the bottom B. Figure 12 shows the dimensional change with time of parts 90A and 90B, a representative billet, and the average dimensional change and weight loss of three additional billets from the same original material lot. Weight loss and dimensional change give amazingly similar curves. Change becomes less with time, although the parts were still losing weight and shrinking after 70 days.

Figure 5. Tensile Strength of 6003-2, Tested Parallel to Rise

Figure 6. Tensile Modulus of 6003-2, Tested Parallel to Rise

Figure 7. Tensile Strength of 6003-2, Tested Perpendicular to Rise

Figure 13 includes data from a second series of foam billets. It can be seen that this series exhibits less shrinkage than did the first group. It can also be seen that the stabilization blanks from the top of the billet, labeled "A", shrink more than the blanks from the bottom of the billet, labeled "B". After the initial differing shrinkage rate, however, the movement appears to be essentially the same.

The following additional studies are under way, but there is not enough data at this time to allow conclusions to be drawn.

Figure 8. Tensile Modulus of 6003-2, Tested Perpendicular to Rise

- A study of expediting stabilization by elevated temperature post cure. Twenty-four hour post cures of 94 and 120°C were chosen for study.
- Variations in the resin to isocyanate component ratio to give isocyanate index levels of 105 to 120.

Moisture Content

Moisture is known to affect polyurethane foams, with moisture increases causing dimensional growth, and moisture losses causing

Figure 9. Ultimate Compressive and Tensile Strength Versus Temperature of 6003-2

Figure 10. Dimensional Change With Time of Billets 330 mm in Diameter by 330 mm High

Figure 11. Effect of Machining on Dimensional Change With Time of Billets 330 mm in Diameter by 330 mm High

Figure 12. Changes in Dimensions and Weight of Stabilization Billets With Time

Figure 12 Continued. Changes in Dimensions and Weight of Stabilization Billets With Time

Figure 13. Changes in Dimensions With Time of Stabilization Billets 159 mm High

shrinkage. Samples of 6003-2 material were tested to determine characteristics of moisture pickup and loss of this material. Figures 14, 15, and 16 were plotted from data obtained by moisture analyzer tests of very small samples (2 to 2.5 cm³).

Figure 14 shows the equilibrium moisture pickup of samples exposed to controlled relative humidity conditions until constant water content was detected by the moisture analyzer. Figure 15 depicts the moisture loss of a sample stabilized at 59-percent RH as the result of exposure to zero-percent RH, and Figure 16 shows the moisture gain of a sample stabilized at zero-percent RH when the sample was exposed to 55-percent RH.

The following are conclusions drawn from this study.

- Moisture pickup is much more rapid than is moisture loss.
- Foam samples stabilized and stored at 15-percent RH, or less, contain less than 0.8-percent moisture.

From this information, a 1-percent maximum water content became a specification limit for the foam product.

To determine the effect of moisture on an actual stabilization blank foam billet, billet numbers 90A and 90B were introduced to a constant 54-percent RH atmosphere. Billet 90A was exposed for 1 hour, and billet 90B for 3 hours. After exposure, the billets were weighed and measured. The results are plotted in Figure 17 as a 20-day section of the Figure 12 stabilization curve. This study confirms that moisture pickup is more rapid than moisture release. Because of the time dependency of moisture pickup both billets were exposed to 48-percent RH for 24 hours. Remeasurement and weight changes are also shown in Figure 17.

The following are conclusions drawn from this study.

- The part weight and dimensional change curves both show the same trends.
- Long exposures produce larger weight increases and dimensional change. Billet 90A after 24 hours exposure is actually larger than its measurement at the start of the study.
- Although moisture pickup is more rapid than moisture loss, the parts returned to the original dimensional loss curve in a relatively short time, as shown by comparing the exposed billets with the curve of billets 89, 91, and 92. The moisture pickup is thought to be a condition which is retarded by the closed cell nature of the foam; thus, short exposures predominately affect the foam surface.

Figure 14. Equilibrium Water Content of 6003-2

Machining and Gaging

Evaluation of machining and gaging capabilities indicates that no special handling, tools, or procedures will be required. Band sawing can easily and quickly be used for rough cutting operations, and single point tape controlled techniques provide very good machined surfaces.

Gaging by use of light pressure (25 g) probes gives repeatable results and does not damage the foam surface. One sample was rotated under a probe for over 200 cycles and no evidence of dimensional change or probe track was seen.

Processing Techniques

Batch-mixing techniques were employed for all of the billets used in the studies summarized in this report.

Figure 15. Moisture Loss With Time of 6003-2, Stabilized at 59-Percent Relative Humidity When Exposed to Zero-Percent Relative Humidity

ACCOMPLISHMENTS

A summary of the accomplishments of this project to date follows:

- Methods of eliminating amine outgassing and resulting compatibility problems were investigated. A material formulation, Rigifoam 6003-2, containing no amine catalyst, was chosen for production use.
- Chemical analysis data, which showed the Rigifoam 6003-2 to be acceptable for the requirements, were used to establish a 300-ppm ash content limit for production.
- Physical property determinations also showed the Rigifoam 6003-2 to be an acceptable candidate for production. This work provided information to allow reduction of production tensile testing requirements by testing in the minimum strength direction only.
- Dimensional change with time measurements taken over a long period showed a continued shrinkage. This shrinkage was

Figure 16. Moisture Pickup of 6003-2, Stabilized at Zero-Percent Relative Humidity When Exposed to 55-Percent Relative Humidity

rapid at first, became less with time, and was small when compared with the part dimensional tolerances. This study was used to establish a 60-day minimum aging time for the foam stabilization blanks and to eliminate concern for a first-in/first-out procedure at the assembly site.

- Moisture causes dimensional changes in the part. These changes are rapid and reversible. Moisture pickup is faster than is moisture loss.
- Machining and gaging studies show that no special tools, fixtures, or procedures should be required for fabrication and inspection of foam parts with a density of 0.05 g/cm³.

FUTURE WORK

Stabilization studies in work will continue, and several new studies will be started. The new studies will be aimed at reducing the stabilization time through heat-aging and material ratio changes.

Mixing studies will be started as soon as the foam metering and mixing machine on order is received to develop a process which will minimize streaks and voids in the product.

Figure 17. Effect of Moisture on 6003-2 Billets

Appendix A

TESTING RISE-RATE, RIGID URETHANE FOAM,
DWG NUMBER 9952034

Drawing Call-Out: Rise-Rate characteristics of rigid urethane per 9952034-00.

41

	DWG CLASSIFICATION LEVEL	
<p>1. <u>GENERAL.</u></p> <p>1.1 <u>Scope.</u> This specification covers the procedure for measuring the rise-rate characteristics of rigid urethane foams using a rise-rate transducer, computer, and dual channel recorder amplifier.</p> <p>2. <u>DOCUMENTS.</u></p> <p style="padding-left: 40px;">NOT APPLICABLE.</p> <p>3. <u>REQUIREMENTS.</u></p> <p>3.1 <u>Equipment.</u> The following equipment or its equivalent is required.</p> <ul style="list-style-type: none"> a. Fluidyne Model 112 Rise-Rate measuring system and manufacture instruction manual. (See 6.5) b. Chart, roll, 5-inch, 0-100 graduations 100 foot length. c. 85 ounce paper container, 6 inches high, 7 5/8 inches O.D. top, 5 3/4 inches O.D. base. d. 32 oz. paper container, 6 inches high, 4 inches O.D. top, 3 1/4 inches O.D. base. e. 2 inch Conn Blade (see 6.4) f. 3 inch Conn Blade (see 6.4) g. 2 inch x 2 inch x 1/2 inch foam float weighing less than 2.5 grams. <p>3.2 <u>Equipment Setup and Calibration.</u> Setup and calibrate the equipment as instructed using the instruction manual. The Rise Range selector setting (H) and the Rate Range selector setting (R) shall be adjusted such that the maximum possible reading will be obtained on their respective charts (without going off scale) when the sample is evaluated.</p> <p>3.3 <u>Mixing of Material.</u></p> <p>3.3.1 <u>Free Rise Density Mix.</u> The nominal free rise density of the materials, in pounds per cubic foot, shall be determined in the following manner.</p> <p>3.3.1.1 weigh an 85 oz. paper container to the nearest gram.</p> <p>3.3.1.2 Mix a sufficient quantity of material, by the method specified in the process specification and the appropriate material specification, such that when the material is poured into the 85 oz. paper container and allowed to foam without restraint, the foamed material will completely fill the container, but not exceed the top of the container by more than two (2) inches.</p>		
ISSUE	B	DWG CLASSIFICATION LEVEL
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5	5	SHEET 2 OF

	DWG CLASSIFICATION LEVEL	
<p>3.3.1.3 Remove the excess material such that the material left in the container will be flush with the top and completely fill the container.</p> <p>3.3.1.4 Weigh the container and its contents to the nearest gram.</p> <p>3.3.1.5 To determine the nominal free rise density of the material, use the following equation:</p> $D = (W_F - W_i) (0.0212)$ <p>Where D = the nominal free rise density of the material, lbs/cu ft.</p> <p>W_F = the weight of the foam and the container, grams.</p> <p>W_i = the weight of the container, grams.</p> <p>3.3.2 <u>Rise Rate Mix.</u> Mix the materials in the ratio specified in the appropriate Material Specification. Use the weights, containers and mixing conditions as shown in Table 1 for the applicable nominal densities as determined per 3.3.1.</p>		
TABLE 1		
Nominal Free-Rise Density, lbs/ft ³	2 4 6 8	10 12 15 20
Mix Weight, grams*	300 400 500 600	700 800 950 1200
Pour Weight, grams	100 200 300 400	500 600 750 1000
Mixing Container	±5 ±10 ±10 ±10	±10 ±10 ±15 ±15
	32 oz paper Container	85 oz paper Container
Mixing Blade	2-inch single Conn	3-inch single Conn
<p>*All weights specified are plus or minus 1%.</p> <p>3.3.3 Any material with a nominal free-rise density other than that specified in Table 1 shall use the conditions for the nominal free-rise density nearest to it, i e, material with a 3.3 pounds per cubic foot nominal free-rise density shall be treated as if the nominal free-rise density was 4 pounds per cubic foot. Materials having a nominal free rise density half way between two values listed in Table 1 shall use the lower of the two values listed; i.e, material with a 7 pounds per cubic foot nominal free rise density shall be treated as if the nominal free-rise density was 6 pounds per cubic foot.</p> <p>3.3.4 The mix speed shall be 1,500 ± 100 rpm in the material.</p>		
ISSUE	B C	DWG CLASSIFICATION LEVEL
		UNCLASSIFIED
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		14213
		DWG NUMBER
		9952034
		SHEET 3 OF

	DWG CLASSIFICATION LEVEL																							
<p>3.3.5 The temperature of the two components of the material at initiation of mixing shall be as specified in the applicable specification or drawing. If no temperature is specified, the temperature of the material shall be $77 \pm 2^{\circ}\text{F}$.</p> <p>3.3.6 The class of the material is as designated in the applicable specification or drawing. The mix times for the various classes of material shall be as given in Table 2.</p> <p style="text-align: center;">TABLE 2</p> <table style="margin-left: auto; margin-right: auto;"> <thead> <tr> <th style="text-align: left;"><u>Class of Material</u></th> <th style="text-align: left;"><u>Mix Time, Seconds*</u></th> </tr> </thead> <tbody> <tr><td>A</td><td>30</td></tr> <tr><td>B</td><td>45</td></tr> <tr><td>C</td><td>60</td></tr> <tr><td>D</td><td>75</td></tr> <tr><td>E</td><td>90</td></tr> <tr><td>F</td><td>120</td></tr> <tr><td>G</td><td>150</td></tr> <tr><td>H</td><td>180</td></tr> <tr><td>J</td><td>210</td></tr> <tr><td>K</td><td>240</td></tr> </tbody> </table> <p style="text-align: center;">*All times stated are plus or minus 3 seconds.</p> <p>3.4 <u>Rise-Rate Machine Operation.</u></p> <p>3.4.1 Pour the proper weight of material as designated in Table 1 into an 85 oz paper container. A time lapse of no greater than 20 seconds is permitted from cessation of mixing until recording operations on the rise-rate machine have begun. Operate the equipment as instructed using the instruction manual.</p> <p>3.4.2 Allow the equipment to operate until the change in the rise height chart is less than 1 unit during a 2 minute time period.</p> <p>3.4.3 The number of pours for each sample shall be as specified in the applicable specification or drawing.</p> <p>3.5 <u>Records.</u> The following data shall be recorded:</p> <p>3.5.1 Initiation Time (Ti): The time interval from start of mixing to the time when 2 percent of the maximum foam height occurs (see 6.1).</p>			<u>Class of Material</u>	<u>Mix Time, Seconds*</u>	A	30	B	45	C	60	D	75	E	90	F	120	G	150	H	180	J	210	K	240
<u>Class of Material</u>	<u>Mix Time, Seconds*</u>																							
A	30																							
B	45																							
C	60																							
D	75																							
E	90																							
F	120																							
G	150																							
H	180																							
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		SHEET 4 OF																						

	DWG CLASSIFICATION LEVEL	
<p>3.5.2 Rise Time (Tr): The time interval from start of mixing to the time when 95 percent of the maximum foam height occurs (see 6.1).</p> <p>3.5.3 Time to Maximum Velocity (T_{V_m}): The time interval from start of mixing to the time that maximum velocity occurs (see 6.2).</p> <p>3.5.4 Maximum Height (X_m): The final foam height; equal to the rise range factor (H)/100 x Maximum Height Ordinate (see 6.1).</p> <p>3.5.5 Maximum Velocity (V_m): Equal to rate range factor (R)/100 x Maximum Velocity Ordinate chart reading (see 6.2).</p> <p>3.6 <u>Rise Rate Constant</u>. The rise rate constant (C) shall be calculated as follows:</p> $C = \frac{V_m}{X_m} \quad (\text{see 6.3})$ <p>SECTIONS 4 AND 5 NOT APPLICABLE</p>		
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		SHEET 5 OF

6. NOTES.

6.1 Example of Rise Recorder curve and calculations for initiation time and rise time.

ISSUE	A	B			DWG CLASSIFICATION LEVEL	SIZE	CODE IDENT	DWG NUMBER
					UNCLASSIFIED	A	14213	9952034
								SHEET 6 OF

6.2 Example of Rate Recorder Curve.

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

$T_{V_m} = 187$ seconds

6.3 $C = V_m / X_m$
 $= (2.9) / (6.3)$
 $= 0.460$ inches/minute/inch

6.4 Conn is the trade name for a mixing blade manufactured by the Conn Company, Warren, Pennsylvania.

ISSUE	A	B	DWG CLASSIFICATION LEVEL	SIZE	CODE IDENT	DWG NUMBER
			UNCLASSIFIED	A	14213	9952034
						SHEET 7 OF

DWG CLASSIFICATION LEVEL

6.5 Fluidyne Instrumentation
3685 Mount Diablo Boulevard
Lafayette, California 94549

6.5.1 Picture of Rise-Rate Machine.

TRANSDUCER

RECORDER
AMPLIFIER

COMPUTER

RECORDER

ISSUE	A	B		DWG CLASSIFICATION LEVEL	SIZE	CODE IDENT	DWG NUMBER
				UNCLASSIFIED	A	14213	9952034
						SHEET	8 OF

6.6 Definitions of Symbols.

X_o = 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 at Start of Mixing, seconds
 T_i = Initiation Time, seconds
 T_r = Rise Time, seconds
 T_{V_m} = Time to Maximum Velocity, seconds
 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

6.7 This specification is designed to serve as a guide for the operation of a "Rise-Rate Machine." All mixing conditions, weights, etc. should be performed exactly as stated herein unless the appropriate Material Specification for the material in question requires that different parameters be used. Should this be the case, the operator must record on chart rolls the mixing conditions, weights, etc., that differ from those required by this Process Specification.

ISSUE	A	B				DWG CLASSIFICATION LEVEL	SIZE	CODE IDENT	DWG NUMBER
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								SHEET 9	OF

Appendix B

IONIC CHLORINE PROCEDURE

Appendix B

IONIC CHLORINE PROCEDURE

Break sample into small pieces, using care to prevent contamination. Accurately weigh material sample, 1 to 1.5 grams, into a 50-cc beaker.

Add 15 cc of absolute ethanol, and gently heat on a hotplate for 10 minutes minimum. Decant the ethanol from the sample into a 30-cc beaker. Wash the sample with 5 cc of ethanol, and add wash solution to decanted liquid in 30-cc beaker. Reduce the total liquid volume to about 10 cc by gentle heating.

Add 5 cc of distilled water followed by 2 cc of 1 normal nitric acid. Then add 1 cc of gelatin reagent (6.0 grams of Knox gelatin, 0.1 gram of thymol blue, and 0.1 gram of crystal thymol diluted to 1 liter with hot water).

Fill beaker to the 25-cc mark with ethanol. Two blanks should be carried through the entire procedure.

Titrate all samples and blanks on a Buchler-Cotlove chloridometer, using the low range (highest sensitivity). Convert the result to ppm chlorine via calibration procedures described in the operating instructions for the Cotlove chloridometer.

Appendix C

ASH CONTENT PROCEDURE

Appendix C

ASH CONTENT

Cut a sample, 15 grams minimum, into chunks approximately 50 mm, and weigh accurately. The sample is then decomposed in a platinum or well-glazed porcelain crucible on a hot plate. Add one chunk of sample at a time to the crucible, and wait until it is decomposed before adding more. This procedure prevents loss of sample from overloading the crucible. Since platinum is very thermally conductive, these crucibles shall be shielded from direct drafts in order to get good degradation. When the sample has been sufficiently reduced on the hot plate, transfer it to a clean muffle furnace. Start at approximately 300°C, and raise the temperature in 50-degree intervals over a period of 4 to 5 hours to 650°C. Hold at 650°C until no evidence of carbon char remains in the crucibles. This usually requires about 2 to 4 hours. Alternatively, the sample can be brought to 500°C in 50-degree stages, left overnight, and then brought to 650°C in the morning for several hours. The sample should be cooled in a dry area or a desiccator. Quantitatively transfer the ash sample into a small accurately weighted vial, and then reweigh. Calculate the ash content in parts per million, based on the transferred weight of the ash.

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BDX-613-1836 (Rev.)

MATERIAL EVALUATION OF POLYURETHANE FOAM,
0.05 g/cm³ DENSITY, J. R. Fender, D/861, UNCLAS
Topical, March 1978

Rigifoam 6003-2, a Bendix manufactured CO₂-blown polyester polyurethane foam system, was chosen for use as the prime material for production of parts with a foam density of 0.05 g/cm³. Studies of the 6003-2 physical properties, chemical analysis, dimensional stability, moisture content, and the machining and gaging capabilities of this material are summarized. The next phase of this project will include the development of processing and stabilization techniques required to

PLASTICS: Polyurethane Foam

MATERIAL EVALUATION OF POLYURETHANE FOAM,
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Topical, BDX-613-1836 (Rev.), March 1978

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consistently fabricate quality foam billets
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