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MATERIAL AND PROCESS DEVELOPMENT
OF POLYURETHANE FOAM, FY73-1

PDO 6984191, Quarterly Report

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Internal Distribution April 1973

Prepared for the
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**Kansas City
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Quarterly Report

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MATERIAL AND PROCESS DEVELOPMENT OF POLYURETHANE FOAM,
FY73-1

BDX-613-797, UNCLASSIFIED Quarterly Report, Internal Distribution
April 1973

Prepared by J. R. Fender, D/834, under PDO 6984191

This report summarizes a continuing evaluation of AVCO's ICU-36 isocyanurate material, a static compressive study in which foam and metal or wood composites are tested, early phases of a dynamic testing program, and a study of flame retardant additives. A final summarization of a past study of the effect of cure temperature on foam strength and shrinkage properties is also presented.

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THE BENDIX CORPORATION

KANSAS CITY DIVISION

KANSAS CITY, MISSOURI

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

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SUMMARY

Characterization and control of processes and properties of polyurethane foam encompasses many aspects. This project, therefore, deals with a number of continuing studies including evaluation of new materials, foam property characteristics, and effects of process changes and material system recompounding.

Work on new material evaluation has been recently directed toward isocyanurate materials. CPR 426 and AVCO ICU-36 have been studied and the ICU-36 has shown the most promise. A modification of the ICU-36 was evaluated this quarter and a reformulated CPR 426 foam system will be examined in the future. The major drawback with the materials evaluated to date is their extremely fast rise and gel time which severely limits their application.

Characterization of foam properties has included activity in static and dynamic compressive testing. The static tests were run on composite samples of foam/wood, foam/copper, and foam/aluminum as well as foam alone. Data from these tests are tabulated. Dynamic testing is just starting and reportable data are not yet available.

A study of flame retardant additives, Vircol 82 and antimony oxide, has shown that the flame retardance of foam systems in use at Bendix can be improved. This study also indicated, as expected, that polymeric isocyanate materials are easier to flame retard than toluene diisocyanate materials, but that the isocyanurate materials are more flame retardant than either.

Effect of cure time and temperature changes upon foam strength and dimensional properties is summarized. Review of the data indicates that general predictions of the strength/cure temperature relationships for molded-to-size foams are at best speculative and that each material application should be evaluated individually.

Future work will include continuation of the new material evaluation, especially of the modified CPR 426 material; static testing using the restraining compression fixture with emphasis placed on foam-wood composites; dynamic testing activity; review of new mold release agents; and evaluation of a Bendix prepared equivalent to materials described in Material Standard 2170553.

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DISCUSSION

SCOPE AND PURPOSE

The purpose of this project is to characterize current and newly developed polyurethane foam systems and to develop the processing techniques and limits, stabilization procedures, and shrinkage data required for use of these systems to make rigid polyurethane foam products.

PRIOR WORK

Evaluation of new materials over the past several quarters has been concentrated on isocyanurate foam systems. Both AVCO and CPR materials were evaluated, with AVCO ICU-36 showing the most promise. The main limiting factor in use of these materials is the very rapid reaction rate which presents handling problems and restricts the size of pour that can be made.

Both AVCO and CPR are working on improved systems. The AVCO material was reformulated to increase its compressive strength through the addition of 5 percent by weight of 0.002- to 0.003-cm-diameter, milled glass fibers. A reactive phosphonate ester flame retarding agent was also added to prevent smoldering after testing of evaluation samples submitted for the accident resistant container (ARC). The CPR material did not become available until late August and is not discussed in this report.

Energy absorbing characteristics of polyurethane foam have been studied during this fiscal year through static compressive testing as well as by dynamic techniques. Static results were determined by compression testing 5-inch-diameter (12.7 cm) by 7-inch-high (17.8 cm) samples to at least 50 percent deflection in a fixture that restrains the sample. A table of results similar to the more recent results appearing in this report was presented last quarter.

Other past activity which is continued includes investigation of flame retardant agents and foam system characterization to determine the effects of changes in mold preheat temperature, cure temperature, and cure time upon compressive strength, compressive modulus, foam shrinkage, and foam stabilization properties. Systems summarized in past reports were CPR 1040-10 (CPR Division of the Upjohn Company), Stafoam BC 1220 (Olin Corporation), and Rigifoam 6003-2.5A (Bendix Corporation.)

ACTIVITY AND ACCOMPLISHMENTS

New Material Evaluation

The AVCO ICU-36 System was modified by the vendor in an attempt to improve the compressive strength and to eliminate smoldering of the material which was experienced after extended 1800°F (983°C) temperature exposure tests run on candidate material specimens for the accident resistant container (ARC).

Milled glass fibers (approximately 0.002 cm in diameter and 0.5 cm long) were added to the material as a reinforcement to increase the compressive strength, and a reactive phosphonate ester was incorporated to give improved fire retardance. The reformulated system is designated Thermarest 5155.

Processing characteristics were virtually unchanged except that the fiber loaded resin material caused very high wear on machine pump seals and rapid leakage developed in two different machines used to dispense and mix the material.

The isocyanurate materials appear to offer an advantage in flame retardance but are hindered by their very rapid rise and gel rate which limits the ability to handle the material. Material poured from the foam machine is fully risen and is tack-free to the touch after only 30 seconds. Even with high output equipment, pouring into rising material results in the trapping of many random and large air voids.

No further work is planned with either the ICU-36 or Thermarest 5155 at this time. Some data, however, are still being collected and will be presented when available.

Static Compressive Study

A number of additional 5-inch-diameter (12.7 cm) by 7-inch-tall (17.8 cm) samples were tested in a restraining compression fixture to compare the relative strength of various material systems and combinations of materials. Data from these tests as well as several reference results reported last quarter are shown in Table 1.

Three materials were included in this quarter's evaluation. These were CPR 1056-20, a water blown toluene diisocyanate material; CPR 381-2E, a fluorocarbon blown polymeric isocyanate material; and AVCO ICU-36, an isocyanurate foam.

Reinforcing materials foamed in place inside the sample included 1-inch-diameter (2.5 cm) hardwood and redwood dowels, 1-inch (2.5 cm) redwood squares, 0.50-inch (1.3 cm) copper tubing, 0.75-inch (1.9 cm) aluminum conduit, and a special insert made of three 1-inch-thick (2.5 cm) redwood boards.

Table 1. Static Compression Test Results

Line	Sample Weight (g)	Load in Pounds (Kilograms) at Percent Deflection					*	Foam System	Reinforcing Material
		14.3	28.5	42.8	50	57.1			
1	656***	18,000 (8172)	24,000 (10896)	32,000 (14528)	39,500 (17933)	52,000 (23608)	72.8	1056-20	Redwood Squares
2	660***	20,500 (9307)	26,500 (12031)	34,700 (15754)	42,500 (19295)	59,000 (26786)	71.4	1056-20	Redwood Squares
3	707**	43,000 (19522)	44,000 (19976)	52,000 (23608)	61,000 (27694)	73,000 (33142)	--	1056-20	Redwood Squares
	717								20 lb/ft ³ (0.32 g/cc)
4	719**	39,700 (18024)	47,300 (21474)	53,000 (24062)	63,100 (28647)	-- --	--	4003-8	Hardwood Dowels
5	720**	44,000 (19976)	46,000 (20884)	57,000 (25878)	66,000 (29964)	82,000 (37228)	--	1056-20	Redwood Squares
6	720**	27,000 (12258)	21,000 (9534)	16,000 (7264)	24,000 (10896)	38,000 (17252)	--	381-2E	Aluminum Conduit
7	721**	40,000 (18160)	47,000 (21338)	56,000 (25424)	66,000 (29964)	84,000 (38136)	71.4	1056-20	Redwood Dowels (7 each)
8	722	26,400 (11986)	30,400 (13802)	39,800 (18069)	51,800 (23517)	-- --	--	4003-8	None
9	724	31,500 (14301)	36,000 (16344)	46,000 (20884)	58,000 (26332)	78,000 (35412)	--	1056-20	None
10	732**	19,000 (8626)	22,000 (9988)	19,000 (8626)	18,000 (8172)	22,000 (9988)	--	381-2E	Copper Tubing
11	733**	52,000 (23608)	54,000 (24516)†				--	1056-20	Special Redwood Insert
12	746***	8,500 (3859)	17,000 (7718)	35,000 (15890)	47,000 (21338)	70,000 (31780)	--	381-2E	Hardwood Dowels
13	784***	26,000 (11804)	34,000 (15436)	45,000 (20430)	58,000 (26332)	80,000 (36320)	68.6	1056-20	Redwood Dowels
14	803	87,500 (39725)	97,500 (44265)	100,000 (45400)	102,500 (46535)	100,000 (45400)	--	None	Redwood (Sandia Tested)
15	806**	60,000 (27240)††					--	1056-20	Special Redwood Insert
16	837***	4,300 (1952)	5,900 (2679)	8,000 (3632)	9,500 (4313)	12,000 (5448)	--	381-2E	Copper Tubing
17	846***	3,000 (1362)	5,500 (2497)	13,000 (5902)	17,000 (7718)	21,000 (9534)	--	381-2E	Aluminum Conduit
18	862**	62,000 (28148)	64,000 (29056)	80,000 (36320)	96,000 (43584)	125,000 (56750)	--	1056-20	Redwood Dowels
19	884	14,000 (6356)	31,500 (14301)	43,000 (19522)	58,000 (26332)	86,000 (39044)	--	ICU-36	None
	896								25 lb/ft ³ (0.40 g/cc)
20	917	16,000 (7264)	33,500 (15209)	46,500 (21111)	62,000 (28148)	96,000 (43584)	--	ICU-36	None
21	981***	41,000 (18614)	56,000 (25424)	92,000 (41763)	126,000 (57204)	200,000 (90800)	57.1	1056-20	Redwood Squares
22	1035***	4,300 (1952)	6,800 (3087)	9,300 (4222)	11,000 (4994)	13,000 (5902)	--	381-2E	Copper Tubing
23	1055**	85,000 (38590)	100,000 (45400)	126,000 (57204)	160,000 (72640)	-- --	54.3	1056-20	Redwood Dowels
24	1061***	48,000 (21792)	75,000 (34050)	108,000 (49032)	146,000 (66284)	-- --	51.4	1056-20	Hardwood Dowels
	1075								30 lb/ft ³ (0.48 g/cc)
25	1084**	93,000 (42222)	108,000 (49032)	132,000 (59928)	166,000 (75364)	-- --	54.3	1056-20	Redwood Squares
26	1085**	87,000 (39498)	104,000 (47216)	130,000 (59020)	161,000 (73094)	-- --	54.3	1056-20	Redwood Squares
27	1085**	80,000 (36320)	106,000 (48124)	134,000 (60835)	156,000 (70824)	200,000 (90800)	57.1	1056-20	Aluminum Conduit
28	1086	76,000 (34504)	92,000 (41768)	130,000 (59020)	180,000 (81720)	-- --	52.8	1056-20	None
29	1087**	79,000 (35866)	92,000 (41768)	120,000 (54480)	167,000 (75818)	-- --	52.8	1056-20	Hardwood Dowels
30	1101**	45,000 (20430)	48,000 (21792)	56,000 (25424)	63,000 (28602)	74,000 (33596)	--	1056-20	Copper Tubing
31	1107**	92,000 (41768)†††					--	1056-20	Special Redwood Insert
32	1187**	98,000 (44492)	115,000 (52210)	142,000 (64468)	184,000 (83536)	-- --	52.8	1056-20	Redwood Squares (7 each)
33	1200***	46,000 (20884)	58,000 (26332)	81,000 (36774)	104,000 (47216)	154,000 (69916)	--	1056-20	Aluminum Conduit

Test samples 5 inches (12.5 cm) in diameter by 7 inches (17.8 cm) high.

*Percent deflection at which 200,000 pound (90800 kg) total load is obtained.

**Test load applied parallel to axis of reinforcement.

***Test load applied perpendicular to axis of reinforcement.

† 56,000 (25424) peak at 1.5 inches (3.81 cm).

†† 60,500 (27467) peak at 1.1 inches (2.79 cm); test

discontinued at 1.6 inches (4.06 cm).

††† 95,000 (43130) peak at 1.2 inches (3.05 cm).

Samples prepared for testing with the load direction parallel to the reinforcement axis were molded-to-size using five reinforcing members except for two special samples produced with seven for comparison. Figure 1 shows a sample before testing along with two which have been tested. It is interesting to note the lack of damage to the tested samples. Figure 2 also shows compressed samples. The left sample contains hardwood dowels, the center copper conduit, and the right aluminum conduit. The fact that the wood compressed and the metal tubes showed a honeycomb-like column failure without catastrophic failure of the sample was somewhat surprising.

The samples tested perpendicular to the reinforcing were prepared by molding a billet containing 24 reinforcing units. From each of these billets, four samples containing 12 reinforcing members were machined. One billet yielding samples with only 8 copper tubes was also produced.

Figure 3 shows specimens representative of this type of sample before and after testing.

One problem experienced in preparation of billets containing reinforcement was cracking of the foam especially in the higher density (0.5 g/cc) parts. This may result from shrinkage stresses caused by differential dimensional change in the gelling and curing foam material and the reinforcing material which is reacting basically to changes induced by only temperature change. Attempts to eliminate the problem are not being pursued at this time. Compressive results on these cracked samples were in the same general range as those expected of uncracked material.

A review of the data in Table 1 indicated the following conclusions.

- Squares and dowels give approximately the same results (Lines 5 and 7).
- Seven reinforcing units instead of five did not appreciably change strengths (Lines 25 and 32).
- The load transmits through the foam matrix into the reinforcement and if the reinforcement is weaker, the test values obtained reflect mainly the strength of the reinforcing material. Lines 16 and 22 give essentially the same values for different weight samples.
- Metal tubes did not appear to increase strength and in some cases actually reduced it.
- Special inserts give high initial strength but loose strength at failure, possibly from lack of column strength (Lines 11, 15, and 31).

Text continued on page 18.

Figure 1. Samples Tested Parallel to Reinforcement Axis

Figure 2. Compression Tested Samples

Figure 3. Samples Tested Perpendicular to Reinforcement Axis

- Wood alone has very high strength (Line 14).
- ICU-36 material (Lines 19 and 20) is not as strong as other materials tested (Lines 8 and 9).

Work will continue with static testing, especially on wood-foam composites.

Determining the Dynamic Properties of Polyurethane Foam

Impact testing to determine the dynamic properties of various foam systems will be done using 2.5-inch-diameter (6.25 cm) samples from 1 to 8 inches long (2.54 to 20.32 cm). Projectiles ranging in weight from 1.89 to 12.60 pounds (858 to 5720 grams) will be impacted into the samples at velocities between 50 and 300 feet (15.24 and 91.44 meters) per second.

Acceleration of the actuator thrust column at firing pressures from 500 psi to 2000 psi (3.45 to 13.79 mPa) increments has been recorded. Integration of these records has provided velocity/displacement information necessary to control impact velocity. Impact velocity will be controlled by selecting the proper actuator pressure and adjusting the gap between the sample and the projectile prior to a test. Final velocity measurement will be determined from magnetic sensors installed every 0.25 inch (0.62 cm) along the projectile/sample guide tube which is attached to the actuator thrust column.

Six preliminary shots have been made on foam samples to evaluate hardware and the effectiveness of the magnetic sensors. Any further testing has been postponed until completion of the total instrumentation system. Assembly of electrical interfacing is underway to permit determination of foam deflection, stress, and energy absorbed by entering a punched paper tape record of each test into the CDC 6600 computer. The paper tape record represents the analog signal from the projectile accelerometer converted to a digital format. Evaluation of the total instrumentation system should begin by September 11, 1972.

Flame Retardant Additives

This portion of the project was initiated to determine if fire retardant characteristics could be imparted to foam systems conventionally used at Bendix without degrading the foam polymer. The initial work, reported previously, provided a cursory examination of the fire retardant characteristics imparted to the foams by two additives, Vircol 82 (Mobil) and antimony oxide. The experiments were all conducted on low density, 2 pounds per cubic foot (0.032 g/cm^3), free rise foam. Studied during this quarter is the effect of these additives on both the burning rates and compressive strengths of foam from higher density, 5 to 20 pounds per cubic foot, (0.08 to 0.32 g/cm^3) molded billets.

Three foam systems, CPR 1204-3C, BKC Thermalthane 4003-8, and AVCO ICU-36, were evaluated at densities of 5 to 20 pounds per cubic foot (0.08 to 0.32 g/cm³) for their fire-retardant characteristics. The AVCO ICU-36 material, an isocyanurate modified polyurethane foam containing no fire-retardant additive, was tested as received from the vendor. The other foams were modified by the addition of antimony oxide or Vircol 82 as shown in Table 2.

Formulations 1 and 2 depict proposed 10 and 20 percent additions of antimony oxide to the foams. However, the antimony oxide was not tested with the CPR 1024-3C material because of viscosity and blending problems with the material. With the BKC Thermalthane 4003-8 foam, the antimony oxide functioned satisfactorily. Formulations 3 and 4 represent 5 and 10 percent additions of Vircol 82 to the foam. These mixtures for the CPR 1024-3C material were abandoned because the resulting foam blocks contained severe yellow streaks indicating separation of the fire-retardant additive. These same problems were not evident with the 4003-8 formulation. Formulations 5 and 6 also contained 5 and 10 percent additions of Vircol 82 (hydroxyl number 192). However, the mix ratios for these formulations were adjusted to account for the reactive sites of the fire-retardant additive. Test blocks made from these mixtures with either the CPR 1024-3C or BKC Thermalthane 4003-8 foams were produced with no difficulty.

Compressive strength test coupons, 1-inch (2.54 cm) cubes, and 6 by 2 by 0.5 inch (15.24 by 5.08 x 1.25 cm) fire retardant test samples were cut from test blocks made from AVCO ICU-36, CPR 1024-3C, formulations 5, 6, and 7, and all BKC Thermalthane 4003-8 formulations. The cubes were compression tested at either room temperature or 275°F (135°C). These results are listed in Tables 3 through 6.

For evaluating the effect of the fire-retardant additives on the compressive strength of the foams Tables 3 and 4, covering the BKC Thermalthane 4003-8, present the most convenient results to study since these data present all of the formulations. Also, these test results are not as erratic as those obtained with the CPR 1024-3C material. There is apparently no effect of the Vircol 82 on the room temperature compressive strength of the foam (Table 3). The antimony oxide, on the other hand, appears to have weakened the material. However, it must be remembered that the antimony oxide is a powder and, as such, does not become part of the polymer structure but is, instead, a filler. The true density of the foam is thus less than the values indicated. In fact, by adjusting the sample density to consider only the foam and not the filler, the compressive strength of the specimens slightly exceeds the strength of the unfilled samples at the same foam density. This conforms to the results obtained on the other filled polyurethane foams. These same judgements apply to the antimony oxide filled materials at the 275°F (135°C) test temperature.

Text continued on page 25.

Table 2. Material Formulations (Parts by Weight)

1024-3C Formulation							
Components	1	2	3	4	5	6	7
CPR 1024-3C R-Component	100.0	100.0	100.0	100.0	100.0	100.0	100.0
Antimony Oxide	40.0	80.0					
Vircol 82			20.0	40.0	20.6	42.8	
CPR 1024-3C T-Component	300.0	300.0	300.0	300.0	313.2	327.4	300.0
4003-8 Formulation							
Components	1	2	3	4	5	6	7
BKC 4003-8 R-Component	100.0	100.0	100.0	100.0	100.0	100.0	100.0
Antimony Oxide	32.9	65.8					
Vircol 82			16.5	32.9	17.0	35.0	
BKC 4008-8 T-Component	229.0	229.0	229.0	229.0	239.5	250.4	229.0

Table 3. Compressive Properties of BKC 4003-8 at Room Temperature

Flame Retardant Additive*	Percent	Density		Compressive Properties					
				10 Percent Deflection		Yield		Modulus	
		lb/ft ³	g/cm ³	psi	mPa	psi	mPa	psi	mPa
Antimony Oxide	10	10.0	0.16	305.6	2.08	307.3	2.08	7261	50.06
Antimony Oxide	20	10.0	0.16	237.8	1.65	247.6	1.72	5884	40.57
Vircol 82	5	10.0	0.16	339.9	2.34	346.3	2.40	7733	53.32
Vircol 82	10	10.0	0.16	332.0	2.30	340.0	2.35	7905	54.50
Vircol 82**	5	10.0	0.16	324.9	2.20	344.7	2.38	7639	52.67
Vircol 82**	10	10.0	0.16	327.1	2.25	334.0	2.31	7373	50.84
None		10.0	0.16	323.8	2.20	331.3	2.31	7843	54.08
Antimony Oxide	10	20.0	0.32	1029.8	7.10	1049.0	7.25	25206	173.79
Antimony Oxide	20	20.0	0.32	910.7	6.30			21728	149.81
Vircol 82	5	20.0	0.32	1258.5	8.67	1273.2	8.80	30096	207.51
Vircol 82	10	20.0	0.32	1233.5	8.50			29327	202.20
Vircol 82**	5	20.0	0.32	1224.7	8.45			28859	198.98
Vircol 82**	10	20.0	0.32	1248.7	8.62			29335	202.26
None		20.0	0.32	1253.3	8.64	1267.0	8.75	28241	194.71
<p>*Parts by weight added to BKC 4003-8 Foam System.</p> <p>**Additional isocyanate added to the foam system to account for the reactive sites on the additive.</p>									

Table 4. Compressive Properties of BKC 4003-8 at 275°F (135°C)

Flame Retardant Additive*	Percent	Density		Compressive Properties				
				10 Percent Deflection		Yield	Modulus	
		lb/ft ³	g/cm ³	psi	mPa		psi	mPa
Antimony	10	10.0	0.16	160.6	1.10	-----	3985	27.48
Antimony	20	10.0	0.16	126.0	0.87	-----	3097	21.35
Vircol 82	5	10.0	0.16	179.1	1.23	-----	4704	32.43
Vircol 82	10	10.0	0.16	153.5	1.05	-----	4083	28.15
Vircol 82**	5	10.0	0.16	178.6	1.23	-----	4804	33.12
Vircol 82**	10	10.0	0.16	162.8	1.12	-----	4161	28.69
None		10.0	0.16	199.1	1.37	-----	5056	34.86
Antimony Oxide	10	20.0	0.32	601.2	4.15	-----	14296	98.57
Antimony Oxide	20	20.0	0.32	506.7	3.50	-----	12237	84.37
Vircol 82	5	20.0	0.32	620.6	4.30	-----	15719	108.38
Vircol 82	10	20.0	0.32	537.7	3.72	-----	13696	94.43
Vircol 82**	5	20.0	0.32	607.4	4.20	-----	14969	103.21
Vircol 82**	10	20.0	0.32	512.8	4.22	-----	15688	108.17
None		20.0	0.32	708.8	4.90	-----	16785	115.73

*Parts by weight added to BKC 4003-8 foam system.

**Additional isocyanate added to foam system to account for the reactive sites of the additive.

Table 5. Compressive Properties Of CPR 1024-3C

Percent Vircol 82 Additive*	Density		Test Temperature (°F)	Compressive Properties					
				10 Percent Deflection		Yield		Modulus	
	lb/ft ³	g/cm ³		psi	mPa	psi	mPa	psi	mPa
5	7.5	0.120	Room	211.1	1.41	232.2	1.60	5727	39.49
10	7.5	0.120	Room	204.4	1.37	219.8	1.50	5478	37.77
None	7.5	0.120	Room	223.3	1.53	230.0	1.59	5986	41.27
5	15.0	0.240	Room	717.2	4.95			20632	142.25
10	15.0	0.240	Room	724.8	5.00	724.1	5.00	22697	156.49
None	15.0	0.240	Room	687.9	4.72	700.9	4.80	22211	153.14
5	7.5	0.120	275**	61.9	0.43	69.2	0.48	2194	15.13
10	7.5	0.120	275**	33.5	0.22	42.0	0.29	817	5.63
None	7.5	0.120	275**	87.6	0.60	98.0	0.67	3239	22.33
5	15.0	0.240	275**	324.6	2.22	328.0	2.25	11909	82.11
10	15.0	0.240	275**	97.5	0.67	0.0	0.0	2701	18.62
None	15.0	0.240	275**	219.1	1.50	233.2	1.62	6998	48.25
*Formulation adjusted to account for the reactive sites of the additive. **275°F = 135°C									

Table 6. Compressive Properties Of AVCO ICU-36

Test Temperature	Density		Compressive Properties					
			10 Percent Deflection		Yield		Modulus	
	lb/ft ³	g/cm ³	psi	mPa	psi	mPa	psi	mPa
Room 275°F (135°C)	5	0.084	48	0.32	53	0.36	1561	10.76
	5	0.083	25	0.17			709	4.89
Room 275°F (135°C)	12.5	0.20	280	1.92	266	1.82	8499	58.60
	12.5	0.20	119	0.80			3303	22.77

The foams containing the Vircol 82, on the other hand, were weakened by the elevated temperature testing. This reduction is probably caused by the introduction of the lower hydroxyl number resin into the polymer.

The fire retardant specimens were evaluated using a horizontal burning test in accordance with Underwriters Laboratories Subject Test 94 and a vertical test utilizing a 3-second ignition time. For the horizontal test, the specimens were placed on a wire screen with the 6 by 2 inch (15.24 by 5.08 cm) surface in the vertical direction. The lower 2 inch (5.08 cm) end was ignited by a flame for three seconds. Since the entire surface of the specimen was charred, only the time to "flame-out" was recorded (Table 7).

These data show that the polymeric isocyanate based BKC Thermalthane 4003-8 foam is inherently more fire-retardant than the toluene diisocyanate based CPR 1024-3C material. While both fire-retardant additives did improve the BKC 4003-8 foam, their effect on this product was not nearly as dramatic as the increased fire-retardance for the CPR 1024-3C material obtained by using the Vircol 82.

For a true comparison of fire retardance characteristics, neither the burn time nor burn distance should be considered separately. For example, the CPR 1024-3C plus 5 percent Vircol material burned for 322 seconds and 5.56 inches (14.1 cm), while the straight CPR 1024-3C burned for only 224 seconds, but the entire sample was consumed by the fire. Obviously, the burn time was shortened because the material burned much more rapidly.

Thus, the conventional polyurethane foam systems used at Bendix can be made fire-retardant. However, there will probably be some effect on the physical properties of the foam produced. If fire-retardation is deemed necessary, each application should be judged separately to ensure that the foam will still perform as required.

Cure Temperature Study

A series of experiments has been performed to determine the effect of mold preheat temperature, cure temperature, and cure time on the physical properties of three polyurethane foam systems in molded-to-size applications. Statistical analyses of the compressive strength and shrinkage data for the individual foams systems were discussed in prior quarterly reports (Rigifoam 6003-2.5A (Bendix) in the FY71-3 report, Stafoam BC 1220 (Olin) in the FY71-4 report, and CPR 1040-10 (CPR) in the FY72-1 report). The data from the studies have been examined collectively to determine if general conclusions can be drawn for the response of molded-to-size foams to changes in mold

Table 7. Results of Burn Tests

Base Material	Flame Retardant Additive*	Percent	Density		Horizontal Test			Vertical Test
			lb/ft ³	g/cm ³	Burn Time** Seconds	Burn Distance		Burn Time Seconds
						Inches	cm	
1024-3C	Vircol 82**	5	9.41	0.15	322	5.56	14.12	114
1024-3C	Vircol 82**	10	9.17	0.14	30	1.42	3.61	52
1024-3C	None		8.77	0.14	224†	6.00†	15.24	174*
1024-3C	Vircol 82**	5	18.04	0.29	132	3.92	9.96	218
1024-3C	Vircol 82**	10	17.79	0.28	37	1.44	3.66	19
1024-3C	None		17.17	0.27	491†	6.00†	15.24	301*
4003-8	Antimony Oxide	10	9.40	0.15	13	0.78	1.98	12
4003-8	Antimony Oxide	20	8.67	0.14	14	0.64	1.63	7
4003-8	Vircol 82	5	9.37	0.15	18	0.90	2.29	5
4003-8	Vircol 82	10	9.47	0.15	18	0.92	2.34	2
4003-8	Vircol 82	5	9.04	0.14	14	0.72	1.83	6
4003-8	Vircol 82	10	9.66	0.15	29	1.18	3.00	2
4003-8	None		8.50	0.14	25	1.20	3.05	36
4003-8	Antimony Oxide	10	18.93	0.30	17	0.51	1.30	3
4003-8	Antimony Oxide	20	17.89	0.28	15	0.17	0.43	0
4003-8	Vircol 82	5	19.37	0.31	35	1.10	2.79	1
4003-8	Vircol 82	10	19.18	0.31	37	0.80	2.03	1
4003-8	Vircol 82**	5	18.67	0.30	30	0.78	1.98	1
4003-8	Vircol 82**	10	19.35	0.31	41	0.74	1.88	1
4003-8	None		18.56	0.30	42	1.03	2.62	47
Avco ICU-36	None		4.82	0.08	11	0.95	2.41	12
Avco ICU-36	None		12.30	0.20	10	0.95	2.41	16

*Parts by weight added directly to foam system.

**Reformulated to account for the reactive sites on the additive.

***Burn time in seconds is the time the specimen burned after the flame was removed.

Horizontal samples were ignited for 60 seconds while vertical specimens were ignited for 3 seconds.

†Sample completely consumed

preheat temperatures and cure cycles. In addition, related data on localized shrinkage, coefficient of thermal expansion, foam pressure, and foam exotherm has been analyzed.

Effects on Compressive Strength

Rigifoam 6003-2.5A and Stafoam BC 1220 were found to respond similarly to changes in preheat temperature. An increase in mold preheat temperature resulted in an increase in apparent compressive strength. The response of CPR 1040-10, however, was that increases in mold preheat temperatures resulted in decreases in apparent compressive strength. This response of CPR 1040-10 was entirely unexpected as it had been generally experienced that increases in mold preheat temperature decrease the density gradient across molded urethane foams thus resulting in higher core densities and resultant increased compressive strength. It has been subsequently determined in studies conducted under another PDO that CPR 1040-10 also responds abnormally to catalyst concentration in that increases in catalyst result in decreased core density and reduced apparent compressive strength. CPR 1040-10 is the only polyurethane foam system studied at Bendix which responds to mold preheat and catalyst in this way.

An increase in cure temperature for CPR 1040-10 resulted in an increase in apparent compressive strength, at room temperature. CPR 1040-10 has very low compressive strength at elevated temperatures, therefore, it was not tested at other than room temperature. Cure temperature was found to have no significant effect on the room temperature compressive strength of Stafoam BC 1220. With increased cure temperatures an increase in the compressive strength at elevated temperature occurred. For Rigifoam 6003-2.5A the response of compressive strength to increased cure temperatures was opposite to that expected. At both room temperature and elevated temperature the compressive strength decreased with increasing cure temperatures. The different responses of the three materials indicates that general predictions of the strength/cure temperature relationships for molded-to-size foams are at best speculative and that each candidate material should be subjected to cure tests.

Effects on Shrinkage

The three materials reacted similarly with respect to shrinkage responses. Mold preheat temperature did not significantly affect shrinkage. As the cure temperature is increased, the overall shrinkage per inch increases. The shrinkage can be divided into two phases. The first phase, the initial or in-the-mold shrinkage, increases as the cure temperature increases and then levels off at higher cure temperatures. Figure 4 shows the effect of cure

Figure 4. Effect of Cure Temperature on Initial Shrinkage of Urethane Foam

temperature on initial shrinkage of CPR 1040-10 molded to 20 pounds per cubic foot (0.32 g/cm^3) block density. The second phase, post-molding shrinkage, is unaffected by cure temperature. Thus when shrinkages of a series of test blocks or parts cured over a range of temperatures is determined periodical and plotted as in Figure 5, a family of parallel curves results. The vertical separation of the curves decreases with increased time as the block stabilizes. The same relationships have been observed for all foams studied to date.

In addition to the effect of cure temperature on overall shrinkage, a second shrinkage-cure temperature relationship has been observed. As the cure temperature is increased the propensity to produce localized shrinkage areas increases. In addition, the severity of the localized shrinkage defect becomes greater.

Effects on Coefficient of Thermal Expansion

Molded test blocks of CPR 1040-10 were cured at 77°F, 120°F, 165°F, 205°F, 275°F, 200°F, and 325°F (25°, 49°, 74°, 96°, 135°, 149°, and 163°C respectively). Three rods approximately 0.5-inch (1.2 cm) diameter by 4.000 (10.1 cm) ± 0.005 inches (.012 cm) long were machined from each block. Coefficient of thermal expansion tests were performed on the specimens at each of four temperature ranges, 77° to 120°F, 77° to 165°F, 77° to 205°F, and 77° to 220°F (104°C). The upper test temperature level, 220°F, is a limitation imposed by the thermal capability of the test equipment. The same test specimens were used for all test temperature ranges from 120°F to 220°F. The data obtained from the test series is summarized in Table 8.

The data for coefficient of linear thermal expansion indicate that at given test temperature, as the cure temperature increases, the coefficient decreases. When the cure temperature exceeds the test temperature, the coefficient becomes constant. At each cure temperature the coefficient decreases until the high end of the test temperature range exceeds the cure temperature. The coefficient then increases rapidly. The increase should be termed an apparent coefficient of expansion as an irreversible change occurs when the test temperature exceeds the cure temperature. That the change is irreversible can be seen in Table 8 by the increase in length of the specimen after completion of the test. The decrease noted for those specimens cured at higher temperatures is attributed to the normal shrinkage since the blocks had not been completely stabilized before machining. The data from the coefficient of expansion tests support our previous assertions that molded-to-size parts should be cured at a temperature above the anticipated use temperature to eliminate potential catastrophic failures.

In addition to performing the coefficient of expansion tests, density determinations

Figure 5. Shrinkage of Stafoam 410

Table 8. Effect of Cure Temperature on Thermal Expansion of CPR 1040-10

Cure Cycle	Thermal Expansion, in. /in/°F x 10 ⁶ (cm/cm/°C x 10 ⁶)				Average Final Change in Length* (in. /in.)	Average Specimen Density	
	77 to 120°F (25 to 48.9°C)	77 to 165°F (25 to 73.9°C)	77 to 205°F (25 to 96.1°C)	77 to 220°F (25 to 104°C)		lb/ft ³	g/cm ³
77°F (25°C) for 4 Days	39.5 (71.1)	72.2 (130.0)	159.9 (287.8)	159.5 (287.1)	+0.0250	19.207	0.308
120°F (48.9°C) for 8 Hours	39.4 (70.9)	35.3 (63.5)	112.6 (202.7)	126.6 (227.9)	+0.0160	20.636	0.331
165°F (73.9°C) for 4 Hours	38.5 (69.3)	34.3 (61.7)	44.5 (80.1)	61.5 (110.7)	+0.0058	20.759	0.333
205°F (96.1°C) for 4 Hours	38.2 (68.8)	35.3 (63.5)	33.6 (60.5)	38.2 (68.8)	-0.0015	20.948	0.336
250°F (121.1°C) for 4 Hours	38.3 (68.9)	35.0 (63.0)	30.3 (54.5)	27.7 (49.9)	-0.0007	21.324	0.342
275°F (135°C) for 4 Hours	38.2 (68.8)	34.6 (62.3)	30.3 (54.5)	27.0 (48.6)	-0.0012	21.188	0.339
300°F (148.9°C) for 4 Hours	37.9 (68.1)	35.2 (63.4)	30.6 (55.1)	25.3 (45.5)	-0.0017	22.020	0.353
325°F (162.8°C) for 4 Hours	38.2 (68.8)	35.2 (63.4)	30.2 (54.4)	25.1 (45.2)	-0.0018	21.421	0.343
*Determined by measuring specimens at 77°F (25°C) before and after completion of coefficient test.							

were made on each specimen. The data in Table 8 indicates that cure temperature results in increased density of the specimens taken from the core. The increase in density that can be attributed to the shrinkage caused by cure is readily calculated. The difference in linear shrinkage of 77°F (25°C) and 325°F (163°C) cures for CPR 1040-10 is 0.020 inches in 6 inches (0.05 cm in 15.24 cm) or 0.33 percent. The volumetric or cubical coefficient of shrinkage is three times the linear shrinkage. Thus the volume decrease from increased cure should be approximately 1.0 percent. Since the pour weight is constant, the density which is inversely related to volume should increase 1 percent. The actual percentage increase in density equals $2.2/19.2$ or 10.9 percent. The increase in density must therefore result from a redistribution of foam material in the test block. The increase in density is probably responsible for a portion of the increase in compressive strength related to cure temperature.

Effects on Foam Pressure and Exotherm

In an effort to gain further insights into the cure temperature-shrinkage relationship, a 6 by 6 by 1 inch (15.24 by 15.24 by 2.54 cm) test block was equipped with a thermocouple and a pressure transducer to monitor changes during the pouring and cure of CPR 1040-10. The data obtained from the pressure transducer was erratic. Distortion of the transducer body was the apparent cause. The temperature data obtained indicated that an exotherm of 155°F (68°C) would be obtained with CPR 1040-10 in a one-inch (2.54 cm) cross section at 21 lb/ft³ (0.34 g/cm³) molded density. The maximum temperature obtained during cure was approximately 10°F (5.6°C) higher than the oven temperature.

FUTURE WORK

New material evaluation will continue with the isocyanurate foam materials. Major effort will be with the modified CPR 426 now designated DX152-36-40.

Static testing using the restraining compression fixture will continue with emphasis placed on wood-foam composites.

Samples for dynamic testing are being prepared and initial test data should be available for reporting next quarter.

Other continuing activity includes a mold release study and the evaluation of a Bendix prepared foam system equivalent to the systems described by Material Standard 2170553.