# Appliance Rigid Foams Blown with Cyclopentane and Cyclopentane/Isopentane Blends

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# Appliance Rigid Foams Blown with Cyclopentane and Cyclopentane/Isopentane Blends

S. L. SCHILLING
*Bayer Corporation*
*100 Bayer Road*
*Pittsburgh, PA 15205-9741*

## INTRODUCTION

**The blowing agent** in rigid polyurethane foams serves at least two important purposes. One is to help give the foam a low overall thermal conductivity, and the other is to provide strength and dimensional stability to the foam by means of the internal cell pressure that it generates. As shown in Figure 1, about 60–65% of the heat transfer of a rigid foam can be directly attributed to the thermal conductivity of the cell gas, with the remainder coming from the thermal conductivity of the solid polymer and from infrared radiation. The thermal conductivity due to the cell gas can be improved not only by using blowing agents with lower thermal conductivities, but also by increasing the closed cell content of the foam, so that less of the cell gas is quickly lost and replaced with air. The radiation contribution to the foam's thermal conductivity is minimized by reducing the cell size of the foam. The easiest way to reduce the thermal conductivity contribution from the solid polymer is to lower the density of the foam so that there is less polymer present. However, reducing foam density may be counterproductive if it results in a larger cell size or more open cells.

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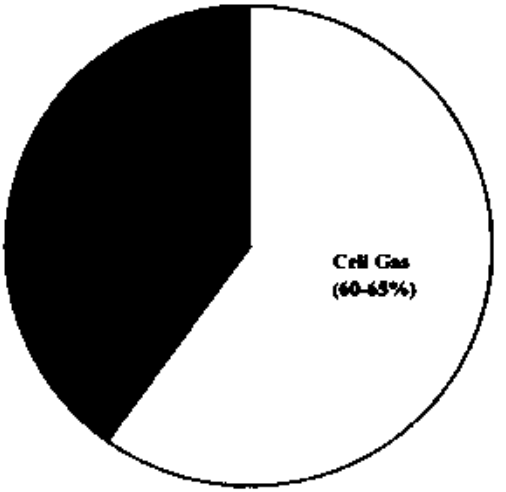


**Figure 1.** Typical contributions to the overall thermal conductivity of a rigid foam.

The second role the cell gas plays in closed cell rigid foams is to impart strength and dimensional stability to the rigid foam by means of the internal pressure it exerts. This is because the internal cell pressure acts to balance the external pressure exerted by the atmosphere. Other things being equal, foams with higher internal cell pressure will have a higher compressive strength than foams with low cell gas pressure. If the cell gas condenses, the cell gas pressure is reduced and must be compensated for by having stronger foam, which may require increasing the foam density.

Table 1 compares the physical properties of cyclopentane and

*Table 1. Comparison of cyclopentane and isopentane properties.*

| Name | Cyclopentane | Isopentane |
|---|---|---|
| Synonym | — | 2-methylbutane |
| Structure | $C_5H_{10}$ | $C_5H_{12}$ |
| CAS number | [287-92-3] | [78-78-4] |
| Molecular weight | 70.14 | 72.15 |
| Boiling point, °C | 49 | 28 |
| Liquid density at 25°C, g/ml | 0.751 | 0.620 |
| Heat of vaporization, cal/g | 97 | 82 |
| Flash point, °C | −37 | −57 |
| *k*-Factor @ 25°C, BTU-in/hr-ft²-°F (Reference [2]) | 0.078–0.086 | 0.093–0.102 |

isopentane. As this table shows, both blowing agents have similar molecular weights, so will provide about the same volume of gas for a given amount of blowing agent. However, cyclopentane has a significantly higher boiling point and higher heat of vaporization than does isopentane. As a result, cyclopentane must reach a higher temperature than isopentane before it begins to vaporize, and the vaporization of cycopentane consumes more heat than that of isopentane. Furthermore, because of its higher boiling point, cyclopentane is more susceptible than isopentane to condensation inside the cells at lower temperatures. These disadvantages of cyclopentane, however, are offset by its better *k*-factor and solubility in polyols.

There is some debate over the precise values for the vapor phase thermal conductivities of cyclopentane and isopentane [2]. Recently, it was even reported that cyclopentane's thermal conductivity approaches that of HCFC 141b at low temperatures [3]. However, it is generally accepted that the thermal conductivity of cyclopentane is significantly higher than that of HCFC 141b and significantly lower than that of isopentane or HFC 134a.

## THEORETICAL CONSIDERATIONS

We used the method previously described by Raymond Yourd [4] to calculate the cell gas content of hypothetical foams made with various blowing agent combinations as a function of temperature. These foams all had a density of 2.00 pounds per cubic foot and were made with the same total level of blowing of 30 ml/g calculated at standard temperature and pressure. One advantage of this technique is that we can "prepare" hypothetical foams under conditions where it would be impossible to prepare actual foam samples because of solubility limitations or other constraints. Figures 2 and 3 show how the cell gas pressure and composition varies with temperature in one of these hypothetical foams. In this case, the foam was blown with water, cyclopentane, and isopentane such that 25% of total blowing came from $CO_2$ and the remaining 75% was from a 60:40 blend of cyclopentane:isopentane.

Above about 80°F no condensation occurs, so the pressure decreases with temperature according to the ideal gas law while the mole fraction of the gases remains unchanged. At about 80°F, cyclopentane begins to condense, which leads to a drop in its partial pressure as well as the total cell pressure. Also, as a result of cyclopentane's condensation, the mole fraction of cyclopentane in the vapor phase begins to decrease while the mole fractions of isopentane and $CO_2$ increase. This continues until

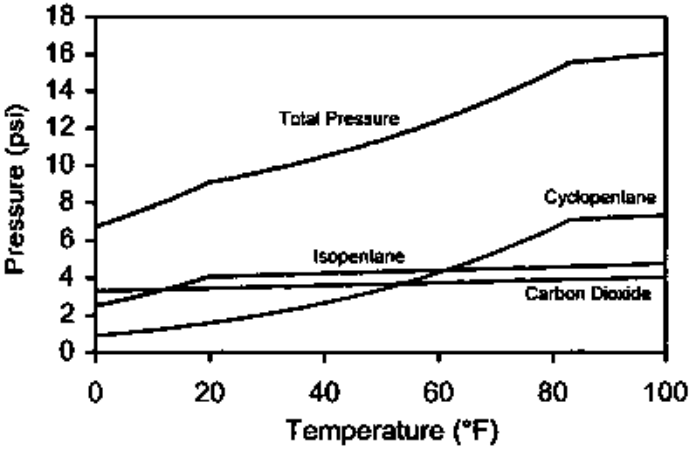


**Figure 2.** Cell gas pressure as a function of temperature.

about 20°F, at which point isopentane begins to condense. Because of the isopentane condensation, the cell pressure drops further and the mole fraction of isopentane begins to fall, with a corresponding increase in the $CO_2$ mole fraction.

From the cell gas composition, it is possible to calculate the thermal conductivity of the cell gas mixture from the thermal conductivities of the individual gases. Our model uses 25°C thermal conductivities of 0.084 and 0.097 BTU-in/hr-ft$^2$-°F for cyclopentane and isopentane, re-

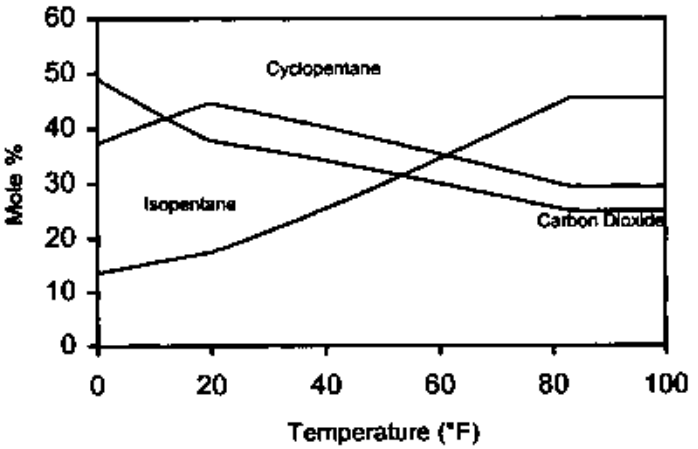


**Figure 3.** Cell gas composition as a function of temperature.

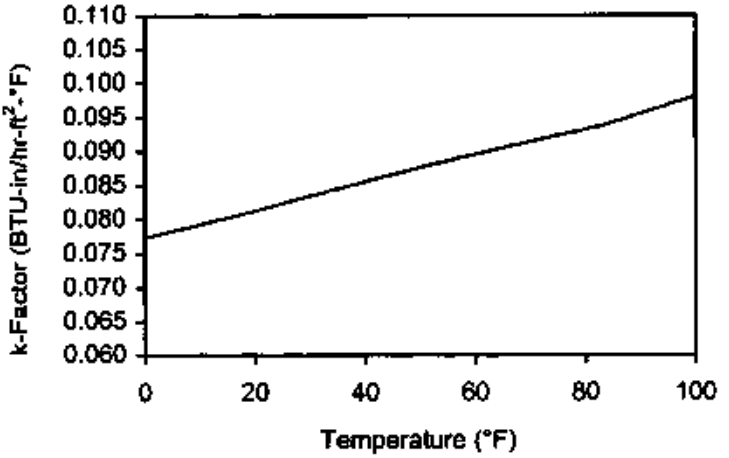


**Figure 4.** Cell gas *k*-factor as a function of temperature.

spectively [5,6]. These values were selected from the many that have been suggested because they gave the best prediction of actual foam *k*-factors. Figure 4 shows a graph of thermal conductivity versus temperature for the hypothetical foam we have been discussing.

Using our model in conjunction with a statistical experimental design, we explored the effects of both the amount of blowing from $CO_2$ and the cyclopentane:isopentane ratio in the hydrocarbon blend used to make the foam. The blowing from $CO_2$ was varied from 0 to 50% of the total, with the remainder being made up of cyclopentane:isopentane blends which ranged from 100% cyclopentane to a mixture of 60% cyclopentane and 40% isopentane. The total amount of blowing and the foam density were again held constant at 30 ml/g and 2.00 pounds/cubic foot, respectively. The total cell gas pressure and cell gas thermal conductivity were then determined at both 35° and 75°F.

The results we obtained were analyzed using JMP® software, and the results were summarized in the contour plots shown in Figures 5 and 6. In these plots, the percent of blowing from $CO_2$ is given on the horizontal axis and the percent cyclopentane in the cyclopentane:isopentane blend used is on the vertical axis. Figure 5 shows contour plots of total cell gas pressures calculated at 35°F and 75°F. These plots show that the total cell pressure increases with both $CO_2$ level and the amount of isopentane present in the blend, which is to be expected. Closer observation shows that at 35°F, the cell pressures are considerably less than at 75°F. This is due to both the temperature effect on the ideal gas law and the condensation effect that was discussed earlier.

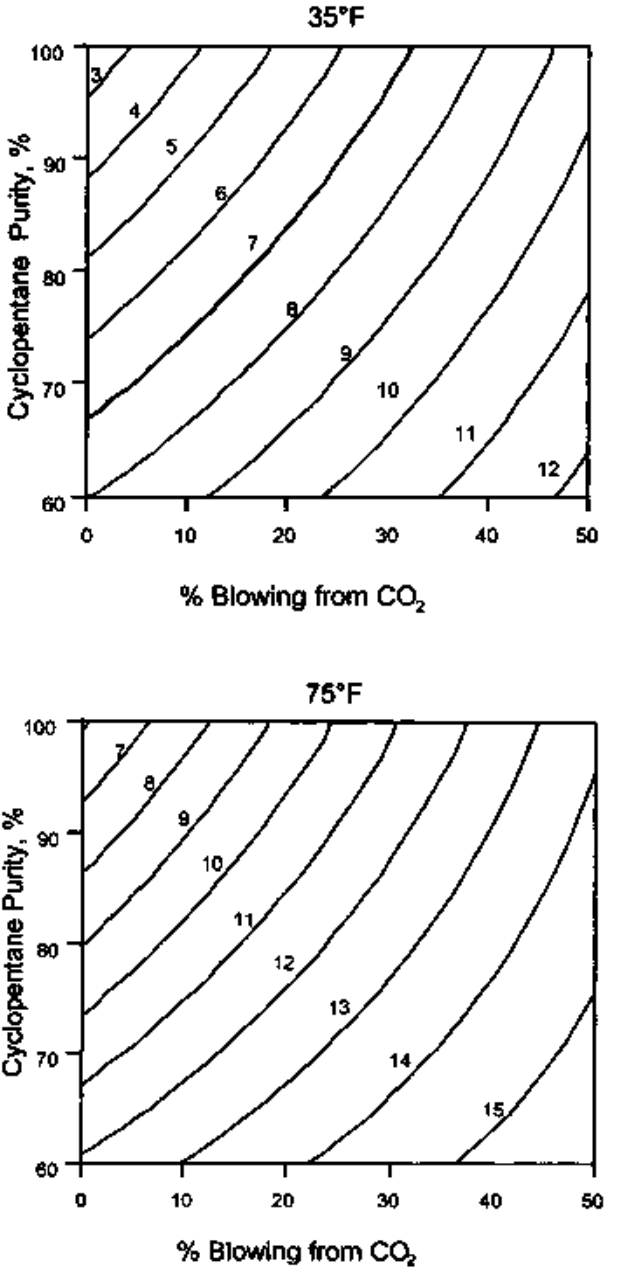


**Figure 5.** Contour plot of cell pressure in psi at 35°F and 75°F as a function of cyclopentane:isopentane ratio and blowing from carbon dioxide.

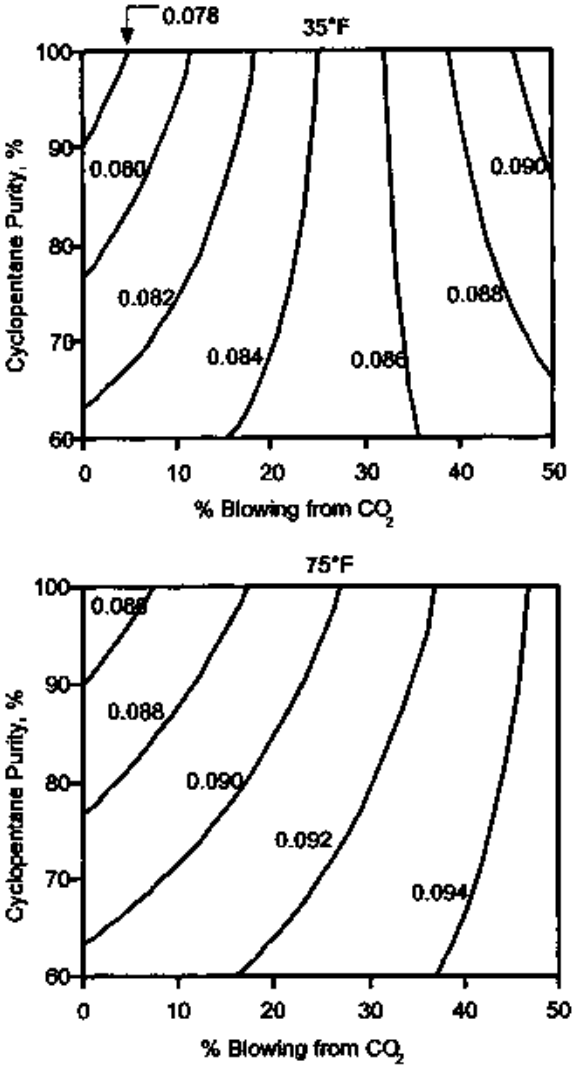


**Figure 6.** Contour plots of cell gas $k$-factor in BTU-in/hr-ft$^2$-°F at 35°F and 75°F as a function of cyclopentane:isopentane.

Figure 6 shows the corresponding contour plots for the cell gas $k$-factors at 35° and 75°F. At both temperatures, the minimum $k$-factor is obtained by using cyclopentane as the only blowing agent because it has the lowest individual thermal conductivity of the three gases. At both temperatures, the gas thermal conductivity increases with increasing $CO_2$

levels. However, at 35°F, when less than about 30% of the blowing comes from $CO_2$, increasing the i-pentane content of the hydrocarbon blend increases the thermal conductivity. With higher $CO_2$ levels, the effect of the c-/i-pentane ratio switches, and the *k*-factor decreases with increasing isopentane levels.

At 75°F, the *k*-factor increases with both the amount of blowing from $CO_2$ and the amount of isopentane being used over the entire region we examined. However, the effect of the cyclopentane:isopentane ratio does become smaller at higher $CO_2$ levels, suggesting that a switch similar to the one seen at 35°F might occur at even higher $CO_2$ levels.

Before moving on to our experimental results, it needs to be pointed out that these results refer to only the initial cell gas composition. In foams with identical structures (density, cell size, closed cell content, etc.) one would expect to see these same trends in actual foam samples. However, as discussed earlier, foam structure accounts for a large part of the thermal conductivity of a foam, and therefore can have a significant impact on the overall *k*-factor. Furthermore, because carbon dioxide is generated by the reaction of water with isocyanate, changing the amount of blowing from $CO_2$ also results in a change in the polymer structure, which can affect foam properties.

## HANDMIX EVALUATIONS

To determine if actual foam samples exhibited the same effects as the hypothetical foams discussed above, three series of handmix experiments were performed. In the first, we varied the relative amounts of blowing from carbon dioxide and cyclopentane while holding the total amount of blowing constant. In the second, we again varied the relative amounts of blowing from carbon dioxide and cyclopentane, but this time we adjusted the total amount of blowing in an attempt to obtain the same foam density. In the final set of handmix experiments, we varied the ratio of cyclopentane:isopentane while maintaining the same amount of blowing from $CO_2$ and the same total blowing level.

### Experimental

All handmix evaluations were performed in a laboratory equipped to safely handle flammable blowing agents. The general procedure used was as follows: The masterbatch (consisting of polyol, surfactant, catalyst, water, and pentane) was prepared, weighed into a container, and the temperature adjusted to 20.0 ± 0.1°C. The isocyanate was wet tarred

into a separate container, and its temperature also adjusted to 20.0 ± 0.1°C. The isocyanate was then added to the masterbatch and vigorously stirred using an air mixer for 5 seconds before being poured into the desired container or mold. To measure gel times, a total of 250 g material was poured into a box and repeatedly touched with a thin stick until the foam adhered to the stick and a string formed as the stick was pulled away.

To prepare panels, a 25″ high × 14″ wide × 2″ thick mold with a detachable lid was used. The mold was heated to 120°F before the desired amount of material was poured into the mold and allowed to rise. The minimum fill density is determined by foaming an amount of material and allowing it to rise above the top of the open mold. The excess material is trimmed off and the resulting foam is weighed. The minimum fill density is then calculated by dividing this weight by the calculated volume of the mold. Test panels are prepared in a similar manner, except that the mold's lid is attached before the rising foam reaches the top. The foam is left in the mold for 3.5 minutes before being removed and cured overnight at room temperature before being cut and tested.

## Effect of Water Level on Cyclopentane Blown Rigid Foams with the Same Total Amount of Blowing

These experiments were run varying the $CO_2$:pentane ratio, but keeping the total ml/g of blowing the same. Since we wanted to examine the effect of low $CO_2$ levels with high pentane levels, we used a total blowing level that was much lower than normal to ensure problems with cyclopentane solubility would not be encountered. This resulted in foams with densities that are much higher than normal, but which should still show the effects of varying the $CO_2$:pentane ratio. In all of these experiments, pure cyclopentane was used and the catalyst level for each formulation was adjusted to give approximately the same gel time. Information on the formulations used in these experiments and the resulting foam properties are given in Table 2.

Two interesting observations can be made concerning these experiments. First, as the amount of blowing from $CO_2$ is reduced, more catalyst is required to obtain the same gel times. The reason for this, we believe, is twofold. First, with less water present, less heat is generated from its reaction with the isocyanate causing the temperature to be lower. The second reason is that when the $CO_2$ level is low, more energy is expended vaporizing the larger amount of cyclopentane that is present. Both of these effects work to reduce the temperature the foam reaches and increase the amount of catalyst needed. We should also

*Table 2. Formulation data and results for experiments used to examine the effect of $CO_2$ level on cyclopentane blown rigid foams with the same total blowing.*

| $CO_2$:Cyclopentane Ratio | High | Medium | Low |
|---|---|---|---|
| Formulation Data, pph: | | | |
| Masterbatch | 100 | 100 | 100 |
| Water in masterbatch | 1.4 | 1.0 | 0.6 |
| Cyclopentane in masterbatch | 8.9 | 9.9 | 10.8 |
| Catalyst in masterbatch | 2.4 | 2.8 | 3.8 |
| % Blowing from $CO_2$ | 40 | 30 | 20 |
| Isocyanate | 117 | 110 | 102 |
| Foam Data: | | | |
| Cream time, seconds | 10 | 10 | 9 |
| Gel time, seconds | 44 | 44 | 41 |
| Minimum fill density, lb/ft$^3$ | 2.42 | 2.51 | 2.83 |

point out that each formulation was run at the same index. However, as the water level decreased, so did the amount of isocyanate used because it was no longer needed to react with the water.

Even though each formulation had the same gel time and total amount of blowing, the minimum fill density increased when the amount of blowing from $CO_2$ was reduced. Again, this is most likely related to the lower temperature that is reached in the foams as the cyclopentane:water ratio is increased. Because the density differences in this series of experiments made direct comparison of physical properties difficult, we ran the following set of experiments.

## Effect of Water Level on Cyclopentane Blown Rigid Foams with the Same Density

In order to get a clearer understanding of the effects of varying the amount of blowing from $CO_2$, we adjusted the formulations for the medium and low ratio experiments above in an attempt to obtain the same foam densities. We did this by increasing the amount of total blowing while maintaining the same cyclopentane:water ratio. These new formulations are shown in Table 3 along with the physical properties of the foams obtained.

Looking at these results, we see that again, a higher catalyst level is needed to obtain the same reactivity when less water is present. Even though the total amount of blowing was increased by 3 and 11% for the medium and low water level experiments, respectively, these minimum

*Table 3. Formulation data and results for experiments used to examine the effect of $CO_2$ level on cyclopentane blown rigid foams with the same density.*

| $CO_2$:Cyclopentane Ratio | High | Medium | Low |
|---|---|---|---|
| Formulation Data, pph: | | | |
| Masterbatch | 100 | 100 | 100 |
| Water in masterbatch | 1.4 | 1.0 | 0.7 |
| Cyclopentane in masterbatch | 8.9 | 10.3 | 12.1 |
| Catalyst in masterbatch | 2.4 | 2.8 | 3.8 |
| % Blowing from $CO_2$ | 40 | 30 | 20 |
| Isocyanate | 117 | 110 | 102 |
| Total blowing, ml/g | 21.1 | 21.8 | 23.4 |
| Foam Data: | | | |
| Cream time, seconds | 10 | 10 | 9 |
| Gel time, seconds | 44 | 44 | 41 |
| Minimum fill density, lb/ft$^3$ | 2.42 | 2.54 | 2.56 |
| Packed density, lb/ft$^3$ | 2.64 | 2.66 | 2.70 |
| Core density, lb/ft$^3$ | 2.32 | 2.31 | 2.29 |
| 35°F $k$ factor, BTU-in/hr-ft$^2$-°F | 0.138 | 0.137 | 0.141 |
| 75°F $k$ factor, BTU-in/hr-ft$^2$-°F | 0.150 | 0.148 | 0.153 |
| Closed cell content, % | 88.1 | 87.2 | 84.6 |
| Average cell diameter, μm | 218 | 208 | 284 |
| Parallel compressive strength, psi | 54.2 | 61.9 | 56.9 |
| Perpendicular compressive strength, psi | 28.4 | 22.9 | 23.2 |

fill densities were still slightly higher than the foam with the high water level. The panels we evaluated were prepared near the same density by varying the amount the panels were packed to give foams with very similar core densities.

The $k$-factors appear to show a minimum at the mid-water level; however, the difference between the high- and mid-water level foams is not statistically significant (standard deviations were ±0.001). It is clear however, that the foam with the lowest amount of blowing from $CO_2$ had the highest $k$-factors. This is contrary to what was predicted by our model and can be attributed, at least in part, to the larger cell size and greater open cell content of this foam.

Our model predicts that the foam with the higher $CO_2$ content would have the greater internal cell pressure, which might be manifested as a higher compressive strength. Indeed, the high $CO_2$ foam does exhibit the highest perpendicular compressive strength of the three. However, this is not the case for the parallel compressive strength, which is highest for foam blown with the medium $CO_2$ level formulation.

## Effect of Cyclopentane:Isopentane Ratio on Foams with the Same Blowing Level

In this set of experiments, we used a single formulated polyol with the same level of blowing, varying only the ratio of cyclopentane:isopentane used. These experiments used a higher blowing level that is more typical of appliance rigid foam formulations than the previous experiments. The formulations used and the physical properties that were measured are given in Table 4.

All of these foams used the same catalyst level and had similar gel times. However, as the isopentane level increased, we observed a drop in the cream time that we attribute to the lower boiling point of isopentane. We did not see any significant changes in density going from 100% cyclopentane to a 60:40 blend of cyclopentane:isopentane. Also, we did not observe any significant change in the compressive strength as would be predicted by our model.

The *k*-factors at both 35° and 75°F were affected, however. These *k*-factors increased by 0.007–0.008 BTU-in/hr-ft$^2$-°F on going from the pure cyclopentane to the 60:40 blend. These increases in *k*-factor are larger than would be expected from our hypothetical foam experimental design. Since all the foams had the same density and closed cell content, these differences from the theoretical values can most likely be attrib-

*Table 4. Formulation data and results for experiments used to examine the effect of cyclopentane:isopentane ratio on cyclopentane blown rigid foam.*

| Cyclopentane:Isopentane Ratio | 100:0 | 80:20 | 60:40 |
|---|---|---|---|
| Formulation Data, pph: | | | |
| Masterbatch | 100 | 100 | 100 |
| Cyclopentane in masterbatch | 12.1 | 9.8 | 7.4 |
| Isopentane in masterbatch | — | 2.4 | 4.9 |
| Isocyanate | 122 | 122 | 122 |
| Foam Data: | | | |
| Cream time, seconds | 10 | 9 | 7 |
| Gel time, seconds | 42 | 41 | 44 |
| Minimum fill density, lb/ft$^3$ | 1.78 | 1.78 | 1.77 |
| Packed density, lb/ft$^3$ | 1.96 | 1.96 | 1.97 |
| Core density, lb/ft$^3$ | 1.73 | 1.73 | 1.72 |
| 35°F *k* factor, BTU-in/hr-ft$^2$-°F | 0.134 | 0.138 | 0.141 |
| 75°F *k* factor, BTU-in/hr-ft$^2$-°F | 0.146 | 0.151 | 0.154 |
| Closed cell content, % | 88.0 | 88.0 | 87.7 |
| Average cell diameter, µm | 188 | 218 | 213 |
| Parallel compressive strength, psi | 35.8 | 42.3 | 38.1 |
| Perpendicular compressive strength, psi | 19.5 | 21.5 | 19.6 |

uted to differences in the foams' cell size, which was smallest in the foam made using pure cyclopentane.

## MACHINE EVALUATIONS

### Experimental

We prepared foams on a Hennecke HK-550 high-pressure foam machine using a Hennecke MQ-18-2 mixhead. The liquid output was approximately 60 lb/minute at a pour pressure of 1500 psig. A Bosch panel mold was used to prepare these panels. This mold is 20 cm wide by 5 cm thick by 200 cm long and was held at 120°F and filled while in the vertical position.

### Bosch Panels

Bosch panels were prepared using the formulation for pure cyclopentane and the 80:20 cyclopentane:isopentane blend listed in Table 4. The results of evaluation of these panels are given in Table 5.

These results show the typical differences one would expect going from a handmix to a high-pressure machine-made foam. These include a faster gel time and higher densities. In addition, foam prepared on the high-pressure machine gave lower *k*-factors, a higher closed cell content, and smaller cell size than the handmix foam. Also, the Bosch panel samples showed smaller differences between the parallel and perpendicular compressive strengths, indicating less orientation of the cells. This last result is somewhat surprising considering the longer distance the foam

*Table 5. Evaluation results of Bosch panel foams samples prepared using a high-pressure foam machine.*

| Cyclopentane:Isopentane Ratio | 100:0 | 80:20 |
|---|---|---|
| Gel time, seconds | 25 | 30 |
| Minimum fill density, lb/ft$^3$ | 1.98 | 1.87 |
| Packed density, lb/ft$^3$ | 2.18 | 2.07 |
| Core density, lb/ft$^3$ | 1.89 | 1.83 |
| 35°F *k* factor, BTU-in/hr-ft$^2$-°F | 0.132 | 0.135 |
| 75°F *k* factor, BTU-in/hr-ft$^2$-°F | 0.142 | 0.146 |
| Closed cell content, % | 90.1 | 89.3 |
| Average cell diameter, µm | 147 | 137 |
| Parallel compressive strength, psi | 32.6 | 31.2 |
| Perpendicular compressive strength, psi | 21.9 | 21.2 |

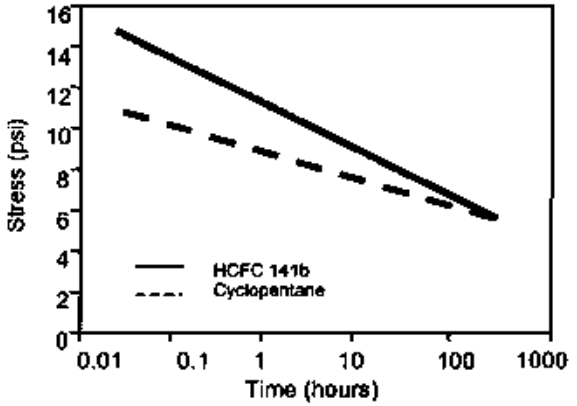


**Figure 7.** Comparison of the compressive creep behavior of a cyclopentane with an HCFC 141b blown foam.

has to flow and the shorter gel times in the Bosch panel as compared to the handmix panels.

In the Bosch panels, unlike the handmix panels, foam made with the cyclopentane:isopentane blend had lower minimum fill, freeze stable, and core densities than the foam made with pure cyclopentane. This can be attributed to the higher vapor pressure of the isopentane. Additionally, like the handmix panels, foams blown with the pure cyclopentane gave lower *k*-factors than the cyclopentane:isopentane blend.

The compressive creep behavior of the foam made using pure cyclopentane on the high-pressure machine was evaluated using the method described previously by Raymond Yourd [4]. Results of this evaluation are given in Figure 7 along with results of a typical HCFC 141b/PMDI system.

As this figure shows, the cyclopentane blown foam is more susceptible to compression by heavy loads than typical HCFC 141b blown foams. However, the slope of stress versus the logarithm of time required to reach 5% compression is less steep for the cyclopentane foam. This indicates that the cyclopentane blown foam is less susceptible to creep caused by small loads over the long term.

## Results of Refrigerators Foamed with Cyclopentane

Typical refrigerator cabinets and doors have been foamed with cyclopentane in trials with a major appliance manufacturer on Bayer Corporation's state-of-the-art foaming equipment in Pittsburgh. Unfortunately, we are not able to report on the specific results of those

trials at this time. However, we can report that the system processed well in both cabinets and doors. Furthermore, the performance of this foam in cabinets was generally comparable to its performance in Bosch panels with one exception; the cabinets gave a lower minimum fill density. We compensated for this by foaming the cabinets at a higher packing level, even though the foam was determined to be freeze stable at a 10% packing level.

## CONCLUSIONS

The results of these studies show that both the amount of blowing from carbon dioxide and the ratio of cyclopentane:isopentane play an important role in the processing and properties of rigid foams. However, theoretical calculations based on the cell gas composition alone were not good predictors of actual foam properties. Furthermore, calculations based only on cell gas composition do not provide any insight into the reactivity and density differences we observed when we varied the $CO_2$:cyclopentane ratio.

Increasing the blowing obtained from carbon dioxide by increasing the water level used in the masterbatch has several beneficial effects. First, it reduces the amount of catalyst needed to obtain a given gel time and also reduces the foam density obtained for a given blowing level. Both of these effects can reduce the cost of using a system for appliance insulation. A somewhat surprising result was that increasing the $CO_2$ blowing level did not significantly increase the $k$-factor of handmix foams as predicted by our calculations. On the contrary, the $k$-factor actually decreased on going from the low water to the high water formulation. Reasons for this could be the higher open cell content and larger cell size obtained with the low water formulations.

We thought that increasing the amount of isopentane used in the cyclopentane:isopentane blend might result in lower density foams or at least give foams with a higher compressive strength as a result of a higher internal cell pressure, as predicted by our theoretical calculations. Neither effect, however, was clearly observed in the handmix foams although they might be more evident in Bosch panels or cabinets. What was clearly observed, however, is that increasing the ratio of isopentane:cyclopentane resulted in foams with significantly higher $k$-factors. This was predicted by our calculations for foams with low water levels, but just the opposite was predicted for high water levels. Besides the cell gas composition, the one obvious difference between these foams is the cell size, which increases significantly with decreasing water levels.

Finally, we showed that the handmix results translated well to machine results, with the critical property of *k*-factor actually improving in Bosch panels.

## ACKNOWLEDGEMENTS

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## BIOGRAPHY

### Steven L. Schilling

Dr. Steven L. Schilling is a Development Scientist in the Technical Insulation Department of the Polyurethanes Division of Bayer Corporation. After receiving a Ph.D. in Physical Organic Chemistry from Duke University in 1984, he joined Mobay Corporation (now Bayer Corporation) as a member of the Process Improvement Laboratory in New Martinsville, West Virginia. There he worked to improve the processes

for manufacturing toluene diisocyanate (TDI) and polymeric methylene diphenyl isocyanate (pMDI) before moving to the Research Department in 1988. While in the Research Department, he was primarily concerned with developing new polyether polyols and methods for manufacturing them. He has been in the Technical Insulation Department since 1997. His current interests include developing improved insulating rigid foams using third generation blowing agents and understanding structure/property relationships in appliance rigid foams.