

# New Polyol for Production of Rigid Polyurethane-Polyisocyanurate Foams, Part 2: Preparation of Rigid Polyurethane-Polyisocyanurate Foams with the New Polyol

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Received 28 July 2008; accepted 25 November 2008

DOI 10.1002/app.29998

Published online 22 June 2010 in Wiley InterScience (www.interscience.wiley.com).

**ABSTRACT:** The method of preparation, determination of foaming parameters, and methods for the determination of physicochemical properties of polyurethane-polyisocyanurate (PUR-PIR) foams prepared with the use of *N,N'*-di(methyleneoxy-2-hydroxyethyl)urea and boric acid derivatives are presented in this paper. It was found that application of the borate as a polyol component and simultaneously as a flame retardant in the recipe for production of PUR-PIR foams was very favorable. The foams prepared were characterized by reduced brittleness, higher

compressive strength and content of closed cells, as well as considerably lower flammability in comparison with standard foam. The results show that the new polyol prepared on the basis of *N,N'*-di(methyleneoxy-2-hydroxyethyl)urea and boric acid can be applied for production of rigid PUR-PIR foams, and it improves their physicochemical properties. © 2010 Wiley Periodicals, Inc. *J Appl Polym Sci* 118: 2250–2256, 2010

**Key words:** foam extrusion; oligomers; polyurethanes

## INTRODUCTION

Mass production of polyurethane plastics provokes escalation of requirements concerning both the products and technologies of their production. At present, producers of these plastics are concerned with the proecological activities. Elimination of toxic compounds and substances hazardous to the natural environment becomes a very significant problem.<sup>1</sup> A desired result is improvement of the raw material properties subjected to processing with simultaneous preservation of the suitable properties of products.

To comprehend specific properties of polyurethane foams, one should skillfully link the properties with foam structure, its macro- and microstructure, and with properties of polymer forming the skeleton of foam. On the other hand, the structure of specific foam is conditioned by the course of the production process and type of raw materials used. Taking advantage of the knowledge resulting from research studies carried out from several decades, the properties of foams can be controlled by the choice of technology and chemical or physical modifications of foam structure.<sup>2–6</sup>

The basic raw materials to produce polyurethanes are isocyanates and compounds containing active hydrogen atoms, particularly polyols, whose chains

are almost exclusively terminated with hydrogen groups, whereas isocyanates contain highly reactive isocyanate groups. They react both with compounds containing active hydrogen and under favorable conditions with themselves. Water, as a component containing hydroxyl groups, plays a significant role in production of foams. The presence of water enables formation of gaseous carbon dioxide, which acts as a blowing agent. It is also liberated in reaction to the isocyanate group with compounds containing carboxyl groups. Therefore, secondary reactions with mixtures containing unreacted or excessive isocyanate are possible. The new isocyanurate structures formed in the process significantly reduce flammability of the prepared rigid polyurethane foams.<sup>7–9</sup>

This study focused on the determination of possibilities to apply a new polyol (described in the first part of paper) containing active hydrogen atoms as well as boron and nitrogen atoms, reducing flammability, for preparation of rigid polyurethane-polyisocyanurate (PUR-PIR) foams.

## EXPERIMENTAL

### Raw materials

The polyether with the trade name of Rokopol RF-55 (product of oxypropylation of sorbitol),  $L_{OH} = 495.0$  mg KOH/g, produced by NZPO "Rokita," Brzeg Dolny, Poland, and Ongromat CR 30-20 (technical polyisocyanate whose main component is diphenylmethane 4,4'-diisocyanate, Budapest, Hungary),

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were used to prepare the rigid PUR-PIR foams. The density of Ongromat CR 30-20 at 25°C was 1.23 g/cm<sup>3</sup>, with viscosity of 200 mPa/s and content of NCO groups, 31.0%. Polyether and polyisocyanate were characterized according to the standards as follows: ASTM D 2849-69 and ASTM D 1638-70.

Anhydrous potassium acetate in the form of 33% solution in diethylene glycol (Catalyst-12, POCh Gliwice, Poland) and DABCO 33LV'' (triethyleneamine produced by Hüls, Germany) in the form of 33% solution in dipropylene glycol were applied as catalysts in foam composition. The polysiloxanepolyoxyallyleneoxydimethylene surface-active agent "Sillicone L-6900" (Witco Corp., Sweden) was used as a stabilizer of the foam structure. The porophor was carbon dioxide formed in reaction to water with isocyanate groups. Moreover, tri(2-chloro-1-methyl-ethyl) phosphate ("Antiblaze TMPC," Albright and Wilson, United Kingdom) was introduced into the foam compositions.

The compound obtained in reaction to *N,N'*-di(methyleneoxy-2-hydroxyethyl)urea and boric acid was applied as a modifying agent for production of foams, and it was named borate.

#### Determination of recipe for PUR-PIR foams

The basis for the determination of recipe for preparation of the rigid PUR-PIR foams was the value of hydroxyl number of polyols, i.e., Rokpol RF-55 and *N,N'*-di(methyleneoxy-2-hydroxyethyl)urea.

The content of auxiliaries (catalysts, unreactive flame retardants, and surface-active agents), which, as a rule, do not contain hydroxyl groups, was determined parts by weight per 100 parts by weight of polyol.

In the next step, the amount of water indispensable to obtain foam of the assumed density was determined by making use of calculations presented in paper.<sup>1</sup>

All the aforementioned substances after being mixed in calculated amounts constituted polyol pre-mix, which was mixed with isocyanate to form rigid PUR-PIR foams.

The amount of isocyanate was determined by taking into account the ratio of isocyanate to hydroxyl groups, which for the rigid PUR-PIR foams was 3 : 1. Calculated amounts of isocyanate were increased by isocyanate mass indispensable for reaction with water in which gas foaming the reaction mixture was liberated, CO<sub>2</sub>.

#### Synthesis of rigid PUR-PIR foams in molds of 18 dm<sup>3</sup>

The rigid PUR-PIR foams were produced, according to the receipts mentioned above, in such a manner that the weighted amount of polyisocyanate was

introduced into one polypropylene vessel (1 dcm<sup>3</sup>), and oligomerol with auxiliary substances were put into another one. Oligomerol was thoroughly mixed with all the components using an electric stirrer (1800 rpm). Polyisocyanate was merged with polyol component and stirred precisely for about 15 s. It was then poured into a form, and expanding of foam was observed. An open form was applied in our studies to allow the foam to expand freely. The form was made from steel (3 mm), and its internal dimensions were 25 cm × 25 cm × 30 cm.

In the first stage, the standard foam without the new polyol added was prepared, and the foams containing from 0.1 to 0.4 of chemical equivalent of the prepared borate were obtained.

During synthesis of PUR-PIR foams, the course of the reaction mixture foaming was monitored by measurement of the respective production times, i.e., start time and times of expanding and gelation.

Start time was measured by a timer, from the moment when all the components were mixed to the moment when so called "creamy state" was attained. It was visualized by the beginning of the foam expansion.

Time of expanding was measured by a timer, from the moment when all the components were mixed to the moment when the foam attained maximum volume.

Gelation time was measured by a timer, from the moment when all the components were mixed to the moment when free surface of foam ceased to stick to a clean glass rod.

Recipes for preparation of foams are listed in Table I.

#### Directions of studies on the properties of rigid PUR-PIR foams

Polyurethanes are segment polymers consisting of the rigid and flexible segments. The rigid segments have an effect on mechanical and thermal strength and high elasticity module, whereas the elastic ones influence softness, flexibility, high values of ultimate elongation, and resistance at low temperature. It was noticed that there is a close dependence between properties of polyurethanes and their physical and chemical structures.

Directions of studies on properties of rigid polyurethane foams are determined by their physical (density, water absorptivity, flammability), mechanical (compressive strength, brittleness), electrical (heat conduction), and chemical (ageing, content of closed cells) properties.

An apparent density of the foams tested was determined by the use of the cubical samples (side of 50 mm, ISO 845-1988 standard), and it was expressed as a ratio of foam mass to its geometric

TABLE I  
Recipes for Preparation of Rigid Polyurethane-Polyisocyanurate Foams

	Unit	Standard	EG1	EG2	EG3	EG4
Rokopol RF-55	Chem. eq.	1	0.9	0.8	0.7	0.6
	g	55.67	50.99	45.32	39.66	33.9
Borate	Chem. eq.	0	0.1	0.2	0.3	0.4
	g	0	5.57	11.15	16.73	22.3
Silikon L6900	g	4.6	4.6	4.6	4.6	4.6
DABCO	g	2.8	2.8	2.8	2.8	2.8
Catalyst 12	g	6.5	6.5	6.5	6.5	6.5
Antiblase TMCP	g	34.6	34.6	34.6	34.6	34.6
Water	Chem. eq.	0.7	0.7	0.7	0.7	0.7
	g	3.15	3.15	3.15	3.15	3.15
Ongromat 30-20	Chem. eq.	3.7	3.7	3.7	3.7	3.7
	g	250.1	250.1	250.1	250.1	250.1

volume. Prior to cutting out the samples used for the determination of apparent density, the foams were seasoned for 24 h at room temperature. The samples were then cut out accurate to 0.1 mm and weighted accurate to 0.1 g.

Determination of water absorptivity was carried out according to DIN 53433 standard. The method consisted of measurement of the buoyant force of a sample (150 mm × 150 mm × 25 mm) immersed in distilled water for 24 h. The method is applied for all rigid porous plastics which do not react with water and are insoluble in water. Water absorbability is a ratio of the absorbed water volume to the initial sample volume expressed in percentage.

The flammability of the obtained PUR-PIR foams was tested by two methods:

1. According to PN-78 C-05012 standard horizontal test. The method consisted of determination of velocity of the flame superficial propagation along the sample (150 mm × 50 mm × 13 mm) placed horizontally and kindled at one end. The velocity of the flame superficial propagation is defined as the velocity of the flame front shifting along the surface of the flammable material tested.

The tested foam sample was placed on a horizontally fixed net and the burner with a flame was put to one of the ends for 60 s. A transverse line was marked at the distance of 125 mm from the sample end to which the burner was applied. The distance of the flame front shifting and the time necessary to reach the marked transverse line by the front of the flame were measured. If the ignited foam went out before flame reached a control mark, the foam was defined as self-extinguishing. The ability of foam to self-extinguish can also be evaluated by measurement of the length of the burned segment. In the case when foam was not extinguished, we determined the mean time of burning of the tested segment or

velocity of flame propagation (in mm/s) on the basis of distance traversed by the front of flame in the defined time.

2. According to ASTM D 3014-73 standard simplified chimney test (Butler's vertical test). The apparatus used to determine the flammability according to the vertical test consisted of a vertical column (300 mm × 57 mm × 54 mm) whose three walls were made from a metal plate and the fourth one, which was a movable pane. Six foam samples (150 mm × 19 mm × 19 mm) were tested. Before burning, the samples were weighted accurate to 0.0001 mm, and they were then placed inside a chimney. A pane was installed, and a flame from the burner, supplied with a gas: propane-butane, was brought into contact with the sample for 10 s. The burner was then removed, and the times of the sample's free burning and retention (residue after burning) were measured by stopwatch in a vertical test. Retention was calculated from eq. (1):

$$R_e = \frac{m}{m_0} \times 100\% \quad (1)$$

where  $R_e$  is retention;  $m_0$  is mass of sample before burning (g); and  $m$  is mass of sample after burning (g).

Compressive strength was determined by the use of general-purpose strength machine (Instron 5544). The peel and flanks of foam were cut off, and the cubic samples were cut out (side of 50 mm ± 1 mm). The samples were then subjected to compressive strain by 10%, according to the direction of foam expansion.

Brittleness of foams was determined using standard apparatus according to the ASTM C-421-61 standard. It was calculated as a loss in mass (in percent) of 12 foam cubes (side of 25 mm) in relation to their initial mass. The apparatus applied to determine the brittleness of PUR foams was a cubical box (190 mm × 197 mm × 197 mm) made from oak

wood and rotating about an axis with a speed of 60 rpm. The box was filled with 24 oak cubes (20 mm × 20 mm × 20 mm).

The loss in foam mass (%), i.e., measure of its brittleness, is expressed by eq. (2):

$$K = \frac{m_1 - m_2}{m_1} \times 100\% \quad (2)$$

where  $m_1$  is mass of shaped foam before test; and  $m_2$  is mass of shaped foam after test.

The softening point (as a thermal resistance under compressive stress) was determined by the use of cubical samples (side of 20 mm), the direction of foam expansion longwise, in accordance with the DIN 53424 standard. The foam samples were subjected to the action of compressive load of 24.52 kPa per hour at a temperature of 50°C. Temperature at which the sample was compressed by 2 mm was accepted as the softening point.

Thermal resistance of foams was determined under dynamic conditions in air atmosphere at the heating rate of 5°/min within the range of temperature from 20 to 800°C. Tests were carried out using a derivatograph produced by MOM Budapest (Paulik-Paulik-Erdey).

Heat conductivity of foams was defined by determination of the thermal conductance  $\lambda$ . The dimensions of the foam samples were 200 × 200 longwise 25 mm and the apparatus FOX 200 (Lasercomp) was used for testing. It allowed us to determine the  $\lambda$  value within the range from 20 to 100 mW/(m K). The method consists of the determination of quantity of heat flowing through the given material in the time unit during steady heat flow at a constant temperature difference on opposite sides of sample.

Changes in the linear dimensions of foams were determined after 48 h of thermostating at a temperature of 120°C (393 K) by using the cubical samples (side of 50 mm). Samples of foams were measured in the direction of the foam expansion longwise. Changes in linear dimensions were calculated by eq. (3):

$$\Delta l = \frac{l - l_0}{l_0} \times 100\% \quad (3)$$

where  $l_0$  is length of sample before thermostating (mm); and  $l$  is length of sample after thermostating (mm).

The loss in mass of foams was determined after 48 h of thermostating at temperature of 120°C (393 K) by using the cubical samples (side of 50 mm). The loss in mass of foams was calculated by eq. (4):

$$\Delta m = \frac{m_0 - m}{m_0} \times 100\% \quad (4)$$

where  $m_0$  is mass of sample before thermostating (g); and  $m$  is mass of sample after thermostating (g).

Changes in volume of the foams were determined after 48 h of thermostating at a temperature of 120°C (393 K) by using the cubical samples (side of 50 mm). Samples were measured in the direction of the foam expansion longwise. Change in volume was calculated by eq. (5):

$$\Delta V = \frac{V - V_0}{V_0} \times 100\% \quad (5)$$

where  $V_0$  is volume of sample before thermostating; and  $V$  is volume of sample after thermostating.

The content of the closed cells was determined in accordance with PN-ISO 4590:1994 standard by method II (dimensions of samples: 100 mm × 30 mm × 30 mm). The method consisted of determination of the relative pressure drop (earlier calibrated for the volume standards) from the differences in indications on the scale of a manometer whose one arm was opened to atmosphere. The method is appropriate for the determination of closed cell content in rigid porous plastics expressed in percentage.

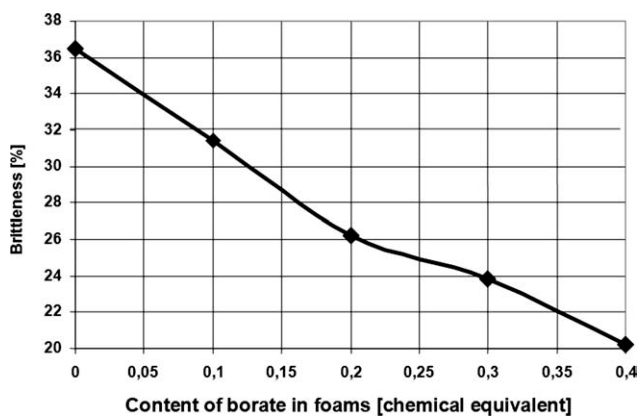
## RESULTS AND DISCUSSION

The rigid PUR-PIR foams were prepared by a one-stage method with the use of boric compound obtained, i.e., *N,N'*-di(methyleneoxy-2-hydroxyethyl)urea and boric acid derivative. The ratio of isocyanate (NCO) to hydroxyl (OH) groups was 3 : 1. Carbon dioxide liberated in reaction of PMDI with water was a foaming gas in these systems.

A progressive introduction (in the amount of 0.1 of chemical equivalent) of boroorganic compound into foam composition resulted in elongation of processing parameters such as start time, time of expansion, and time of gelation in comparison with standard foam. Thus, it can be stated that the boric compound obtained was less reactive in respect to PMDI, in comparison with commonly applied polyol.

Apparent densities of the foams prepared with addition of borates are higher than that of standard foam (36.96 kg/m<sup>3</sup>), and they increase as the amount of the new polyol is increased in foam composition, i.e., from 52.32 kg/m<sup>3</sup> for EG1 foam (containing 0.1 of chemical equivalent) to 71.41 for EG4 foam (containing 0.4R of the new polyol).

The boroorganic compounds applied to produce the rigid PUR-PIR foams behave in foam composition similarly to the common cross-linking agents, causing the foam structure to become more ordered. Therefore, on the basis of the brittleness determinations, it was found that addition of boroorganic compounds to foam composition resulted in reduction of the foam brittleness. For the standard foam, it was equal to 36.5%. Addition of compound containing boron caused reduction of brittleness from 31.41%



**Figure 1** Dependence between foam brittleness and content of boroorganic compound in foam composition.

for EG1 foam containing 0.1 of chemical equivalent to 20.2% for EG4 foam containing 0.4 of chemical equivalent of this polyol.

An increase in the content of the boroorganic compound in the foam composition resulted in considerable reduction of foam brittleness (Fig. 1).

The mechanical compressive strength of the foams prepared is very high. Increase in the compressive strength of the foams prepared was observed as the content of the new compound was increased in foam composition. A close directly proportional dependence between density and ultimate compressive strength of the foams was observed. It was equal to 237.5 kPa for standard foam. For other foams with progressively reduced amounts of the industrial oligomerol (Rokopolu RF-55) at the cost of the same quantity of boroorganic compound, the mechanical compressive strength was within the range of 241.9 kPa for EG1 foam containing 0.1 of chemical equivalent of borate to 398.7 kPa for EG4 foam containing 0.4 of chemical equivalent of borate. The results allowed us to state that introduction of the new polyol into the premix resulted in significantly better ultimate compressive strength of the foams obtained (Fig. 2).

The content of closed cells was determined in the materials under study. It was found that the new polyol had an effect on quantity of closed cells. PUR-PIR foams containing new polyol were characterized by a distinct increase in content of closed cells in comparison with standard foam. Content of closed cells was 83.4% for standard foam and within the range of 84.1% for EG1 foam to 97.1% for EG4 foam containing 0.1 of chemical equivalent of borate. Application of boroorganic compounds allowed us to obtain foams with a content of closed cells above 90% (foams: EG2, EG3, and EG4).

Analysis of results showed that the softening point of the foams containing boroorganic compounds was insignificantly lower in comparison with standard foam. The softening point was reduced from

230°C for standard foam to 218°C for EG4 foam containing 0.4 of chemical equivalent of new borate.

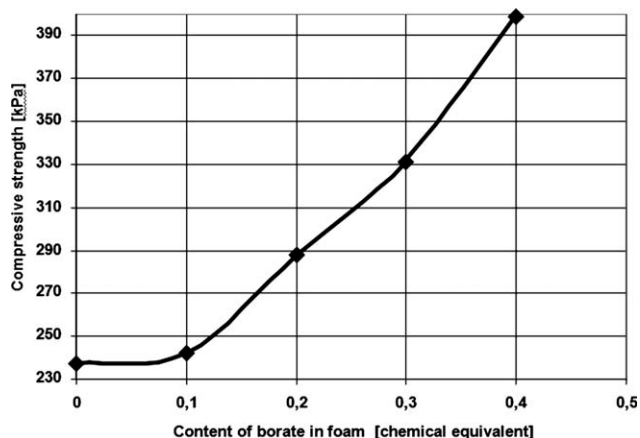
Moreover, water absorptivity was determined for the new foams. It was found that water absorptivity decreased as the quantity of the new polyol in foam composition was increased. Minimum decrease was observed for EG1 foam (1.7%) and maximum decrease for EG4 foam (0.61%), respectively. It can result from a smaller quantity of closed cells in EG1 foam in comparison with EG4 foam. For standard foam, it was equal to 1.8% v/v and for other foams it decreased to about 0.87% v/v.

Studies on stability of the linear dimensions, changes in volume, and loss in foam mass after thermostating for 48 h at temperature of 120°C showed that borate applied had no effect on properties mentioned above within the range of experiments.

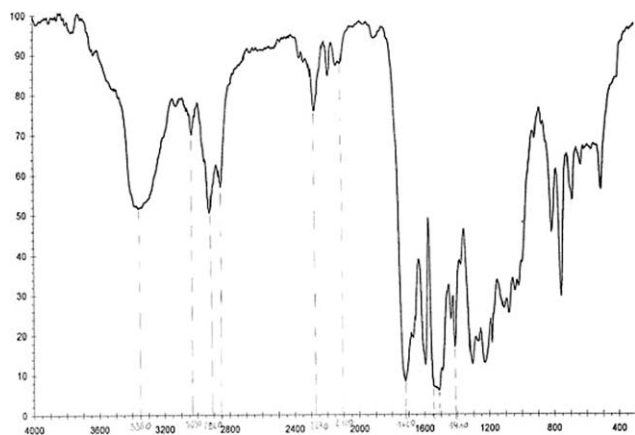
To confirm the presence of group characteristic for foams, the foams were ground in a Janicki's mill and then analyzed by the IR spectroscopy method by the use of the KBr technique. The IR spectrum of PUR-PIR foam containing 0.4 of chemical equivalent of borate is presented in Figure 3. The Thermogram of foam containing 0.4 of chemical equivalent of borate is presented in Figure 4.

It was found that the foams prepared were PUR-PIR ones, since the vibration bands corresponding to urethane group ( $2180\text{--}1710\text{ cm}^{-1}$ ) and isocyanurate ring ( $1710\text{--}1680$  and  $1410\text{ cm}^{-1}$ ) were observed in spectra.

To achieve more fully the characteristics of thermal resistance, all the foams prepared were subjected to thermogravimetric analysis under dynamic conditions in air atmosphere at a heating rate of  $5^\circ\text{C}/\text{min}$  at the range of temperatures from 20 to  $800^\circ\text{C}$ . On the basis of TG (change in mass) and DTG (derivative of change in mass) curves, the characteristics were determined as follows: temperature of the first change in mass of foam ( $^\circ\text{C}$ ), extrapolated temperature of the



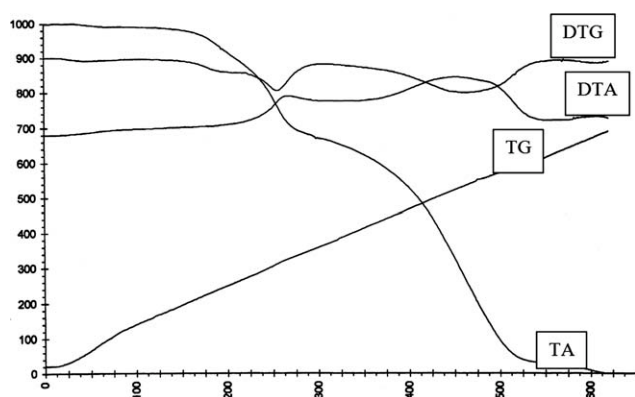
**Figure 2** Dependence between compressive strength of foams and content of boroorganic compounds.



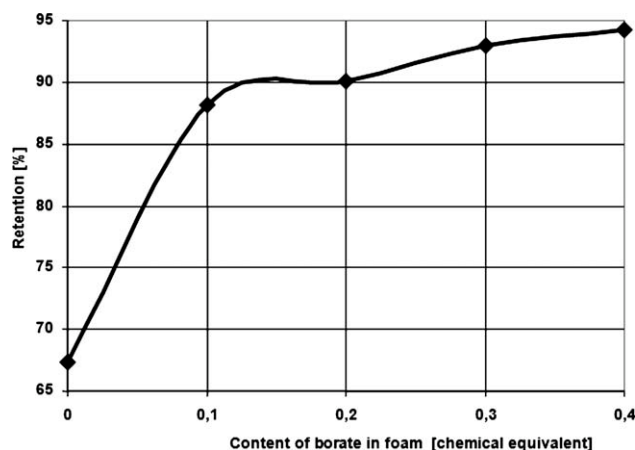
**Figure 3** IR spectrum of PUR-PIR foam containing 0.4 of chemical equivalent of borate.

main loss in mass ( $^{\circ}\text{C}$ ), and temperature of the highest rate of loss in mass ( $^{\circ}\text{C}$ ), (extreme on DTG curve).

The first loss in foam mass occurred at temperatures of  $164^{\circ}\text{C}$  for standard foam to  $247^{\circ}\text{C}$  for EG4 foam. The first loss in mass was caused by diffusion of carbon dioxide, which plays a role of porophor in the foams studied. Carbon dioxide is formed in reaction to the excess of isocyanate groups ( $-\text{NCO}$ ) with water. At temperatures from  $85$  to  $110^{\circ}\text{C}$ , diffusion of triethylenediamine from foams can also occur. Triethylenediamine (DABCO) as a 33% solution in dipropylene glycol was applied as a catalyst in reaction to polyisocyanates with polyols. The extrapolated beginning of the main loss in mass occurred at temperatures from  $181^{\circ}\text{C}$  for standard foam to  $258^{\circ}\text{C}$  for EG4 foam. Extrapolated temperature of the main loss in mass of standard foam is connected, among others, with decomposition of urea formed in reaction of polyisocyanate with water and urethane groups. As the content of borates in the foam composition is increased, the increase of flexible segments occurs, which contributes to higher resistance to the raised temperature. However, the highest rate



**Figure 4** Thermogram of foam containing 0.4 of chemical equivalent of borate.



**Figure 5** Dependence between retention of foam and content of boroorganic compounds.

of loss in mass was observed at temperatures from  $289^{\circ}\text{C}$  for standard foam to  $345^{\circ}\text{C}$  for EG4 foam.

Thermal conductivity for all the foams prepared was from  $30.0$  to  $31.8$  ( $\text{mW}/\text{mK}$ ), and there was no strict correlation with the content of closed cells in foams.

Taking into consideration vertical tests for the determination of flammability of PUR-PIR foams, it was found that the residue after combustion of foams (retention) increased as the content of new polyol was increased in foam composition. Dependence between retention and amount of oligomerols containing boron is presented in Figure 5.

Retention value was within the range from  $67.3\%$  for standard foam to  $91.2\%$  for EG4 foam. Thus, the anti-flammability action of boron and nitrogen introduced into polyurethane plastic together with the new polyol was confirmed.

## SUMMARY

The rigid PUR-PIR foams containing derivative of  $N,N'$ -di(methyleneoxy-2-hydroxyethyl)urea and boric acid were prepared by a one-stage method at equilibrium ratio of isocyanate ( $\text{NCO}$ ) to hydroxide ( $\text{OH}$ ) groups equal to  $3 : 1$ . It was found that application of the compound prepared had an effect on prolongation of start time, time of expansion, and time of gelation.

It was found that the addition of boroorganic compounds to foam composition resulted in reduction of water absorptivity in foams from  $6\%$  for standard foam to  $0.61\%$  for foam containing the highest content of borate, whereas the distinct increase in content of closed cells was observed, i.e., to  $97.1\%$  for EG4 foam in comparison with standard foam ( $84.1\%$ ).

On the basis of results obtained, it was found that brittleness of foams prepared with the addition of boroorganic compounds was considerably reduced.

Brittleness of foams was reduced as the amount of the new compound was increased in the foam composition. The lowest brittleness was obtained for foams containing the highest amount of *N,N'*-di(methyleneoxy-2-hydroxyethyl)urea and boric acid (20.2%), whereas the brittleness of standard foam was 36.5%.

It was also observed that the softening point was reduced as the amount of the compound prepared was increased in foam composition.

Analysis of the flammability tests, i.e., the values of flame extinguishing and its range, allowed us to qualify the prepared foams to self-extinguishing ones. Maximum reduction in flammability of foams, i.e., the highest retention (91.2%), was obtained for foams containing maximum content of *N,N'*-di(methyleneoxy-2-hydroxyethyl)urea and boric acid derivative, whereas retention for standard foam was 67.3%. A favorable effect of the new polyol on reduction of flammability of foams was observed as the amount of this polyol was increased in foam composition.

The developed and discussed foam recipes guarantee preparation of PUR-PIR foam, characterized by compressive strength about 400 kPa, which con-

siderably reduced flammability as well as higher amount of closed cells and reduced brittleness.

It should be emphasized that the new compound, i.e., *N,N'*-di(methyleneoxy-2-hydroxyethyl)urea and boric acid derivative, performs two important tasks in PUR-PIR foams obtained. Namely, it reacts with polyisocyanate when applied in polyurethane pre-mix as polyol, which allows obtaining high-molecular polyurethane, whereas the presence of boron and chlorine in the structure of the new compound guarantees a considerable reduction of flammability of PUR-PIR foams prepared with its participation.

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