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Component Analysis of Rigid Polyurethane Foams

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SUMMARY

A procedure is described for the analysis of the component parts of a rigid polyurethane foam. The technique can be used to identify the polyol, isocyanate, fire retardant, and blowing agent used in foam manufacture when less than 0.5 g of material is available. Techniques of pyrolysis/gas chromatography/mass spectrometry (Py–GC–MS), electron impact (EI) and chemical ionization/mass spectrometry (CI–MS), and high pressure liquid chromatography (HPLC) were essential for component characterization and identification. Foam samples were base-hydrolyzed and the resulting polyols and amines were identified by EI and CI–MS, and HPLC. A combination of CI–MS, Py–GC–MS, and halogen and phosphorus-sensitive chromatographic detectors were used for qualitative and quantitative analysis of the fire retardant. The blowing agent was determined by Py–GC–MS. The composition of the foam was confirmed by the synthesis and characterization of a chemically identical foam using known starting materials.

INTRODUCTION

For the past two decades, there has been a steady growth in the commercial use of polyurethane foams, many of which contain specialized or propriatory formulations. The analysis of component parts of these foams, including fire retardants or other additives that may have been added in manufacture, presents a challenge to the chemist who is concerned with characterizing urethane-containing materials. The analyst has turned to conventional spectroscopic techniques such as infrared (IR) and nuclear magnetic resonance (NMR) spectroscopy to "fingerprint" or classify formulations [1, 2]. Destructive techniques such as thermal analysis [3], evolved gas analysis [4], pyrolysis/gas chromatography [5], and pyrolysis/gas chromatography/mass spectrometry [6] have been applied with limited success to the classification of polyurethane foams. A more recent technique for identification involves degradation of the polyurethanes by hydrolysis and the identification of the polyamines and polyols that are formed [7, 8]. The latter technique has been successfully applied to a variety of polyurethane formulations in a recent publication [9].

Each of the above analytical techniques, when applied individually, provides limited information about the chemical composition of the polyurethane foam sample. Commercial formulations are often encountered for which structural information is not available, and, in these cases, it is necessary to integrate the resultant data from many analytical techniques to identify the components of the urethane foams. In this report, we describe a systematic approach that integrates information from many analytical techniques to chemically identify the polyol, isocyanate, fire retardant, and blowing agent of a rigid urethane foam. Less than 0.5 g of foam sample is needed even when prior structural information is not available.

EXPERIMENTAL

Infrared spectra were recorded using a Perkin-Elmer Model 237 grating infrared spectrometer. Solid samples were run as KBr pellets and liquids were run neat. Proton nuclear magnetic resonance (^{1}H-NMR) spectra were recorded using a Varian EM-390 spectrometer (Varian Associates, Palo Alto, California) with $CDCl_3$ solvent and tetramethylsilane as an internal standard.

The thermogravimetric studies were performed on a Mettler Model 1 Thermoanalyzer equipped with a corrosive gas inlet. Samples (10 ± 0.1 mg) were heated at 10 °C/min using either helium or air at a flow rate of 160 ± 10 ml/min. A Pyroprobe Model 100 (Chemical Data Systems, Oxford, Pennsylvania) was also used to heat the samples to a specified pyrolysis temperature. Sample pyrolysis using the Pyroprobe was carried out in the injection port of a Model 7620 gas chromatograph (Hewlett-Packard Co., Palo Alto, California) equipped with flame ionization (FID) and flame photometric (FPD) detectors. The effluents from the gas chromatograph were also analyzed in some experiments using a Model 5930A mass spectrometer (Hewlett-Packard) interfaced with a Model 5933A data system (Hewlett-Packard). The mass spectrometer was scanned from *m/e* 15 to 300 at 100 amu/s.

The elemental composition of samples was determined by energy dispersive X-ray analysis (Edax International, Inc., Prairie View, Illinois) coupled to a Cambridge Stereoscan electron microscope (Cambridge Instrument Co., Ossining, New York). Carbon, hydrogen, nitrogen, and oxygen content was measured using a Model 240 Elemental Analyzer (Perkin-Elmer Corp.).

Chemical ionization (CI) mass spectra were obtained using a Model 5982 gas chromatograph/mass spectrometer (GC–MS) system equipped with a Model 5933A data system (Hewlett-Packard). Samples (5 to 20 μg) were introduced into the ion source (150 °C) via a direct insertion probe that was heated slowly from ambient to 325 °C over a 10 minute interval. Mass spectra between *m/e* 60 and 700 were recorded and stored every 8 seconds. The CI experiments used methane (0.5 mmHg, UHP grade) that was bled into the ion source coaxially to the direct insertion probe.

Separation of the aromatic polyamines was carried out on a Model 601 liquid chromatograph (Perkin-Elemer Corp.) equipped with a variable-wavelength detector set at 240 nm. A 25 cm column (Sil-X-I, Perkin-Elmer Corp.) was used isocratically at 30 °C with an isopropanol/hexane ratio of 30:70.

RESULTS AND DISCUSSION

An outline of the direct and indirect methods of analysis that were performed on the unknown foam sample is shown in Fig. 1. Direct methods are those analytical techniques which were applied to the foam sample without any sample derivatization or pretreatment. The direct methods used in this work were infrared (IR) spectroscopy, elemental analysis, thermal analysis, and pyrolysis/gas chromatography/mass spectrometry (Py–GC–MS). Indirect methods are those that used a derivatization or pretreatment step prior to analysis. High pressure liquid chromatography (HPLC), CI/mass spectrometry, and gas chromatography are the reported indirect methods. The chemical and structural information obtained by these techniques was combined to identify the foam formulation. As shown in Fig. 1, the chemical composition of the unknown foam was verified by synthesizing a formulation from known polyols, isocyanates, fire retardant, and blowing agent. The synthesized material was subjected to the same analytical procedures and the data were compared to results from the unknown formulation. When all data from the two foams correspond, identification of the foam was complete.

The following paragraphs describe the procedures and the results from the analysis of an unknown rigid foam sample. Although the results are described for only one foam sample, this procedure is applicable, in principle, to the analysis of any material containing polyester, polyether, polyurethane, polyurea, or other similar hydrolyzable linkages.

Direct methods of analysis

Infrared spectrum

The IR spectrum of a 1 mg sample of foam is shown in Fig. 2. The classification of the foam as a urethane is determined by the absorption band at 6.5 μm (amide, N–H deformation), 5.8 μm (amide, C=O stretch), and 3.0 μm (N–H and O–H stretches). The presence of polyether linkages, arising from the polyol portion of the urethane, is suspected because of the strong absorption at 9.4 μm (C–O–C stretch). Aromatic groups, which are indicated by the bands at 6.25 μm (C=C stretch) and 12 μm (aromatic C–H bend), are characteristic of methylene diphenyldiisocyanate (MDI)-based foams. The absence of

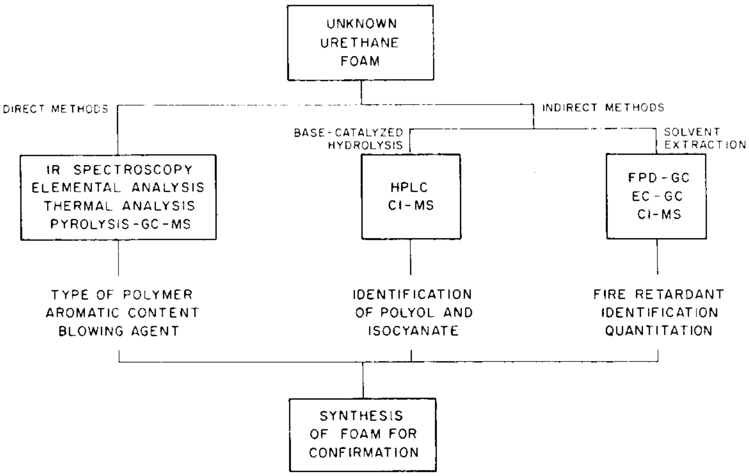


Fig. 1. Analytical methods used for the identification of component parts of a rigid urethane foam.

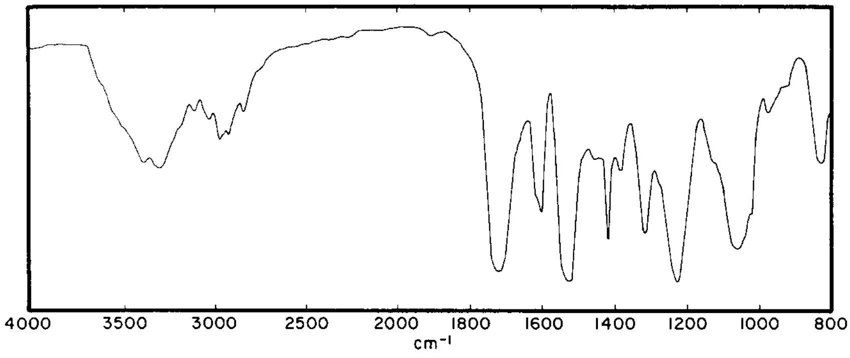


Fig. 2. Infrared spectrum of unknown.

an absorption band at 4.4 μm shows that any residual isocyanate groups, which are usually present for long periods of time after the manufacture of a foam, have reacted with moisture in the environment. It was noted that the IR spectrum of this unknown foam matched the IR spectra of many other MDI-based foams that were formulated using different polyols [10]. Thus we tentatively identified the unknown as an MDI-based foam but further work was needed for a more specific identification.

Elemental composition

Quantitative analysis of the sample was determined without prior drying. The elemental composition of the foam was 59.60% C, 6.09% H, 8.93% N, and 25.38% O. These values are within the range of other rigid urethane foams which we have examined.

Results from the X-ray elemental analysis of this foam are shown in Fig. 3. The elements detected were aluminum (1.5 keV), silicon (1.7 keV), phosphorus (2.0 keV), chlorine (2.6 keV), and iron (6.4 and 7.1 keV). The silicon content was assigned to a surfactant, and the phosphorus could be from a fire retardant. The chlorine might be from a fluorocarbon blowing agent or it could have been incorporated in the fire retardant. The aluminum and iron may have originated from reaction or storage vessels that came in contact with the raw materials used to make the foam.

Thermal degradation

The thermogravimetric (TGA) and the derivative thermogravimetric (DIG) curves for

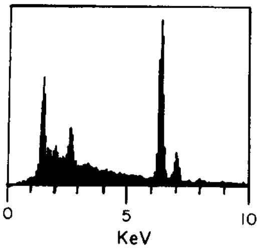


Fig. 3. EDAX elemental analysis of unknown foam.

the unknown foam are shown in Fig. 4. The bimodal weight loss in the TGA curve with maxima at 240 and 400 °C is similar to curves from other fire-retarded urethane foams examined in this laboratory [11].

Pyrolysis studies

A 3.5 mg sample of the foam was heated to 1000 °C in using the Pyroprobe 100 and analyzed by GC–MS. The sample degradation products were separated by temperature-programming a 2 m column of Chromosorb 101 support (Johns-Manville Co., Denver, Colorado) from −20 to 220 °C at 10 °C/min. The reconstructed mass chromatogram, (the summed intensities of all ions from *m/e* 15 to 450 amu) is shown in Fig. 5. The compounds that were identified from these chromatograms by relative retention times and mass spectrometry [12] are listed in Table 1. The GC–MS data from pyrolysis establish the identity of the blowing agent used in foam manufacture as $CFCl_3$ (peak 11, Table 1). The presence of the aromatic amines in the mass chromatograms supports the hypothesis that the unknown foam is an MDI-based polyurethane. However, additional data were necessary to identify the specific isocyanate that was present.

TABLE 1

Volatile products from the pyrolysis of unknown foam

Peak No.	Compound	TC integration[a]
1	Carbon dioxide	154.7
2	Ethylene	3.5
3	Ethane	4.9
4	Propene	36.6
5	Chloromethane	4.7
6	Chloroethylene	1.0
7	Isobutene	n.i.
8	Butadiene	n.i.
9	Chloroethane	2.4
10	Ethanol	1.6
11	Trichlorofluoromethane	n.i.
12	Not identified	3.2
13	Chloropropene	1.4
14	1,2-Dichloroethane	15.2
15	Benzene	18.4
16	Not identified	3.3
17	Toluene	4.1
18	Phenol	n.i.
19	Ethylbenzene	n.i.
20	Xylene	n.i.
21	Aromatic amine	n.i.
22	Aromatic amine	n.i.

[a] Not integrated.

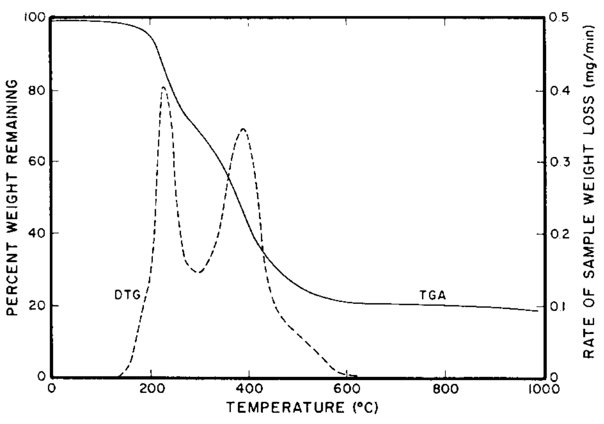


Fig. 4. TGA and DTG curves for unknown foam in helium.

Indirect methods of analysis

Fire retardant

The X-ray elemental analysis of the foam (Fig. 3) indicated that phosphorus and chlorine were present in the sample. The sample was analyzed for a phosphorus-containing fire retardant by the following procedure: about 3 mg of the foam were crushed, extracted with 300 μl warm CH_2Cl_2, and filtered. The

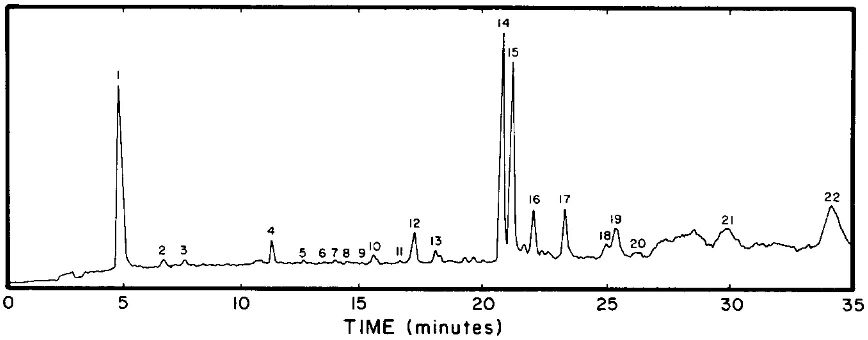


Fig. 5. Pyrolysis–GC–mass chromatogram of unknown foam.

remaining foam was rinsed with 200 μl CH_2Cl_2, and combined filtrates were reduced in volume to 300 μl. A 3 μl sample of the colorless solution was injected onto a 2 m long GC column containing a UCW-98 liquid phase. A single response of the FPD (wavelength specific for phosphorus) was observed for this sample that was not detected in the solvent blank. Thus, one phosphorus-containing compound had been extracted from the foam.

Additional information from this extract was obtained from CI–MS data. A 60 mg sample of foam was extracted (as described above) and the solvent was removed by passing dry nitrogen over the liquid surface. The residue was heated to 250 °C on a direct insertion probe inside the CI–MS ion source resulting in a mass spectrum containing ions at *m/e* 285, 287, 289, and 291 in a ratio of 100:98:29:4, respectively. Ions appearing in 2 amu intervals with this ratio of intensities are characteristic of a compound with a molecular weight of 284 containing 3 chlorine atoms [13]. Other ions in the mass spectrum appeared at *m/e* 249, 251, and 253 with relative intensities of 100:68:10. These ions represent the loss of HCl from the *m/e* 285 ion cluster.

The GC and CI–MS data showed that the fire retardant in the foam contained phosphorus, 3 chlorine atoms per molecule, and a molecular weight of 284. Tables of commercially-used fire retardants [14] listed only one compound meeting the above criteria: tris(β-chloroethyl) phosphate, $O{=}P(OCH_2CH_2Cl)_3$. Tests on authentic samples of this phosphate ester produced identical GC retention times on two separate liquid phases (UCW-98 and 3% OV-1) using FPD and ECD modes of detection. The CI mass spectrum of the foam sample extract was identical to the spectrum of the authentic sample.

Quantitative analysis of the tris-(β-chlorethyl) phosphate was carried out on a 10 mg sample. This sample was finely ground and transferred to a continuous extraction unit charged with 75 ml ethanol. After a 15 hour extraction period the methanolic solution was reduced in volume to <10 ml by careful distillation through a 100 mm Vigreaux column. This concentrate was diluted to 10 ml in a volumetric flask. The concentration of tris-(β-chloroethyl) phosphate was determined by comparing solutions of known concentration to the unknown.

The extent to which the fire retardant had been removed from the foam by the continuous methanol extraction procedure was examined using a Py–GC technique. Accurately weighed samples (0.1 - 1 mg) of the unknown foam from before and from after the methanol extraction were pyrolyzed directly into the injection port of the FPD equipped GC using a Pyroprobe Model 100. The fire-retardant was volatile when the samples were heated to 400 °C for 40 s so that the compound vaporized from the foam and was swept by the carrier gas into the GC column for analysis. The results from two experiments were that 95 and 97% of the tris-(β-chloroethyl) phosphate had been removed from the foam by the methanol extraction. The fire retardant level in the foam, as calculated from the concentration of the methanol extract and corrected for 95% extraction efficiency, was 3.3%.

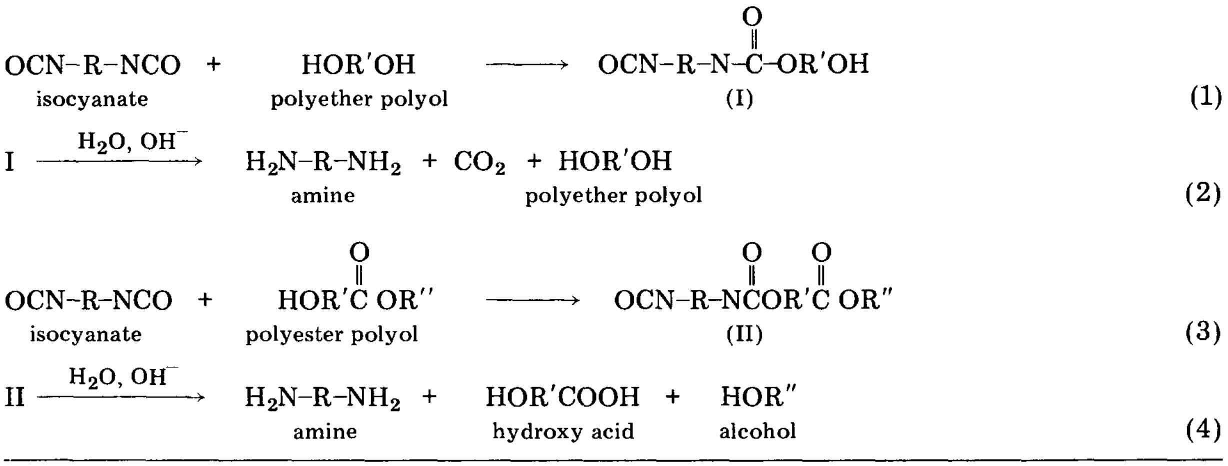
Analysis of foam hydrolysates

To identify the polyol and isocyanate components, the unknown foam was hydrolyzed by aqueous base [9]. Although information about the configuration of constituents in the polymer is lost upon hydrolysis, degradation of the urethane polymer in this manner produces compounds which are easily related to the original foam components. Ester and amide bonds are hydrolyzed under base catalyzed conditions while ether linkages remain intact. Thus, when hydrolyzed, a urethane (eqn. 1) produces a polyamine corresponding to the original isocyanate and the original polyol (eqn. 2). Polyester polyols (eqn. 3) are further hydrolyzed to hydroxy acids and alcohols (eqn. 4).

(See eqns. (1) - (4) on page 112.)

After the polymer has been hydrolyzed, characterization of the resulting products becomes a problem of separation and identification of the amine and polyol components. The amine fraction has typically been examined by thin-layer chromatography (TLC) and IR spectrometry [7, 8]. The polyol fraction has been more difficult to characterize and a combination of TLC and GC separation methods have been employed upon derivatized samples.

When a limited amount of sample is available, HPLC and CI–MS techniques are the best methods presently available to identify



$$\underset{\text{isocyanate}}{\mathrm{OCN{-}R{-}NCO}} + \underset{\text{polyether polyol}}{\mathrm{HOR'OH}} \longrightarrow \underset{\text{(I)}}{\mathrm{OCN{-}R{-}N{-}\overset{\overset{O}{\|}}{C}{-}OR'OH}} \qquad (1)$$

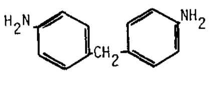
$$\mathrm{I} \xrightarrow{\mathrm{H_2O,\ OH^-}} \underset{\text{amine}}{\mathrm{H_2N{-}R{-}NH_2}} + \mathrm{CO_2} + \underset{\text{polyether polyol}}{\mathrm{HOR'OH}} \qquad (2)$$

$$\underset{\text{isocyanate}}{\mathrm{OCN{-}R{-}NCO}} + \underset{\text{polyester polyol}}{\mathrm{HOR'\overset{\overset{O}{\|}}{C}\,OR''}} \longrightarrow \underset{\text{(II)}}{\mathrm{OCN{-}R{-}N\overset{\overset{O}{\|}}{C}OR'\overset{\overset{O}{\|}}{C}\,OR''}} \qquad (3)$$

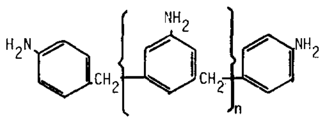
$$\mathrm{II} \xrightarrow{\mathrm{H_2O,\ OH^-}} \underset{\text{amine}}{\mathrm{H_2N{-}R{-}NH_2}} + \underset{\text{hydroxy acid}}{\mathrm{HOR'COOH}} + \underset{\text{alcohol}}{\mathrm{HOR''}} \qquad (4)$$

the compounds present in the hydrolysates. Thus, 50 mg samples of the unknown foam were hydrolyzed in 50% potassium hydroxide at 150 °C as described in a previous publication [9]. CI mass spectra of the hydrolysates were recorded as the sample was heated from ambient to 320 °C over a period of approximately 10 minutes. Figure 6 is a time-averaged mass spectrum of the unknown foam hydrolysate. For comparison, Figs. 7, 8 and 9 are the CI mass spectra of the hydrolysates of three urethane foams of known but different composition. These standard foams were formulated from polymethylene polyphenylisocyanate and the following polyols: (1) propoxylated trimethylol propane (Wyandotte, TP-340); (2) propoxylated sucrose-glycerine (Olin, Poly-G 531S); and (3) a propoxylated sucrose diethanolamine (Olin, Poly-G 530SA). It is evident that distinct mass spectra arise from hydrolysates of foams which differ in polyol composition.

The ions in the four CI mass spectra that can be assigned to the amine derivatives of polymethylene polyphenylisocyanate are listed in Table 2. These assignments were made on the basis of the methane-CI mass spectra of amine standards III and IV:



(III)



(IV)

The base hydrolysis of a standard sample of 4,4′-methylenediphenyl diisocyanate was used to synthesize III. A mixture of the homologs of IV was obtained from the hydrolysis of a sample of polymethylene polyphenylisocyanate in aqueous base. The components of IV were separated by HPLC, and the CI mass spectra were used to identify the following compounds: IV, $n = 0$ *ortho* isomer; IV, $n = 0$ *para* isomer; IV, $n = 1$; and IV, $n = 2$. Figure 10 displays the mass spectrum of compound IV. The nearly identical mass spectra of the unknown foam hydrolysate and the standard confirms the identity of the isocyanate in the foam formulation.

The hydrolysates were analyzed by HPLC to provide additional information about the composition of the isocyanate was used in foam manufacture. The HPLC chromatogram of the hydrolysate is shown in Fig. 11(a) and Fig. 11(b) is a chromatogram of the hydrolysis products of polymethylene polyphenylisocyanate (containing ~50% MDI and the balance of higher polymerics). CI–MS was used to identify the compounds that eluted in each chromatographic peak. The corresponding chromatographic peaks, shown in Figs. 11(a) and 11(b), contain the same compounds in approximately equal ratios. These data add support to the conclusions that were previously drawn concerning the structure of the isocyanate.

Several ions observed in the CI spectrum of the foam hydrolysate could not be assigned to polyamines III or IV. In other urethane foam hydrolysates these ions have been shown to arise from the polyol [9]. The mass spectrum of the polyol, obtained by subtracting the ion intensities of IV (Fig. 10) from the mass spectrum of the hydrolysate of the unknown foam is shown in Fig. 12. This mass spectrum, in

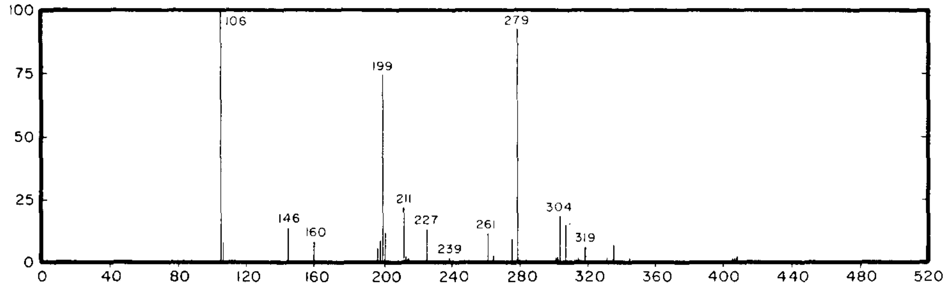


Fig. 6. Composite CI–MS spectrum of unknown foam hydrolysate.

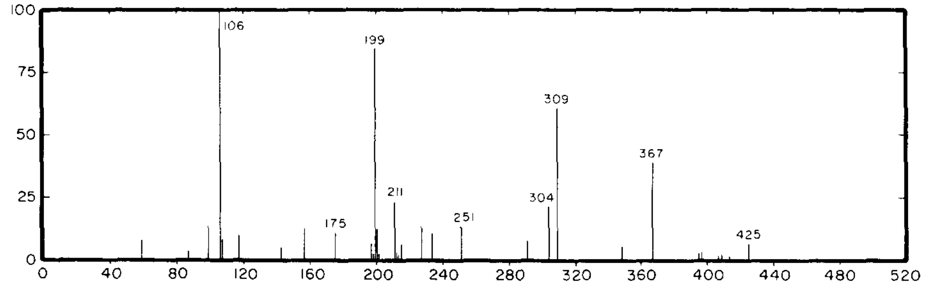


Fig. 7. Composite CI–MS spectrum of foam standard 1 hydrolysate.

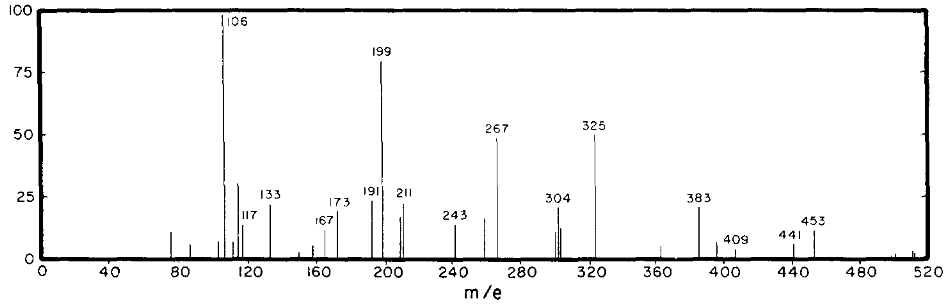


Fig. 8. Composite CI–MS spectrum of foam standard 2 hydrolysate.

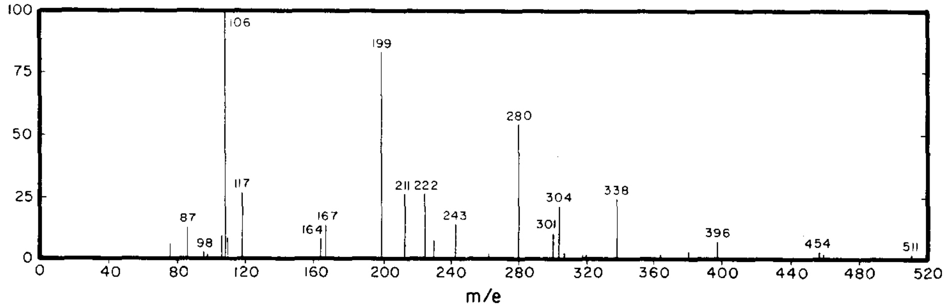


Fig. 9. Composite CI–MS spectrum of foam standard 3 hydrolysate.

itself, did not provide enough information to identify the polyol, but it did indicate that the molecular weight of the major compound is 278. The mass spectra of polyols on hand in this laboratory were compared with the spectrum shown in Fig. 12. The spectrum of the unknown polyol matched the spectrum of only one standard, a propriatory polyol manufactured by Dow Chemical Company. The spectra of other standard polyols were sufficiently dissimilar as to provide a positive identification of the unknown polyol.

TABLE 2

CI–MS spectrum of amine derivatives of polymethylene polyphenylisocyanate

m/e	Relative intensity	Tentative assignment
106	100	$C_7H_8N^+$
107	7	
198	8	m^+, IV, $n = 0$
199	72	$(m + 1)^+$, IV, $n = 0$
200	11	
211	20	$C_{14}H_{15}N_2^+$ fragment of 304 and 409
212	2	
227	11	$(m + 29)^+$, IV, $n = 0$
239	1	$(m + 41)^+$, IV, $n = 0$
302	1	
303	1	m^+, IV, $n = 0$
304	18	$(m + 1)^+$, IV, $n = 1$
316	1	$C_{21}H_{22}N_3^+$ fragment of 409
332	2	$(m + 29)^+$, IV, $n = 1$
344	1	$(m + 41)^+$, IV, $n = 2$
407	1	
408	1	m^+, IV, $n = 2$
409	2	$(m + 1)^+$, IV, $n = 2$
511	1	$(m + 1)^+$, IV, $n = 3$

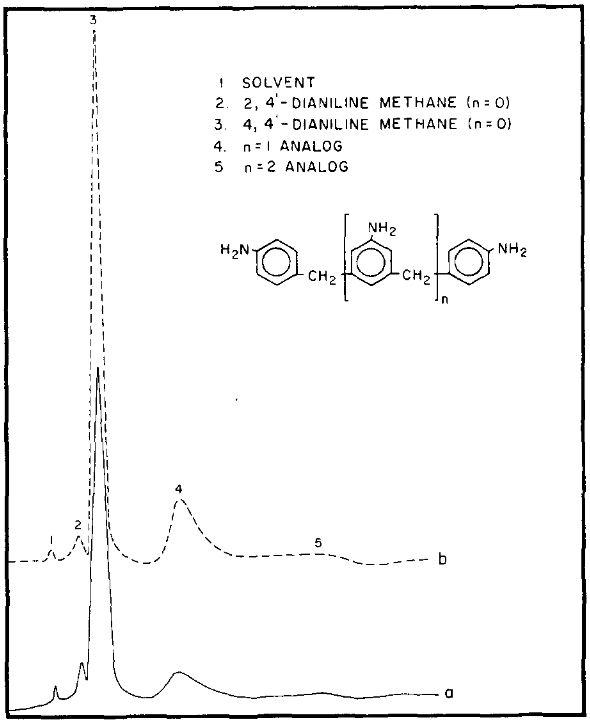


Fig. 11. HPLC traces of (a) unknown and (b) standard polyisocyanate hydrolysis products.

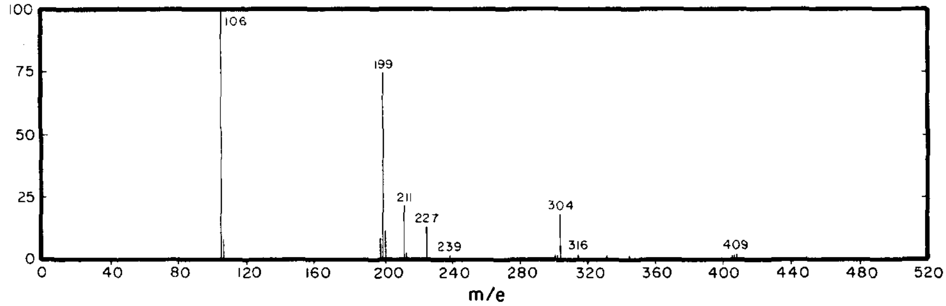


Fig. 10. CI–MS spectrum of polyamines IV.

Additional experiments were carried out to characterize the Dow polyol formulation. Most polyether polyols currently used in foam formulations contain a series of homologs incremented by one propoxy unit [1]. The polyol ions in the mass spectrum of the foam formulation (Fig. 12) are spaced 58 amu apart with relative intensities of 3:100:6. Since the only fragmentation observed in the CI–MS of polyols has been the loss of water molecules, we may infer that the ions in Fig. 12 represent $(M + 1)^+$ species [9]. Therefore, the polyol is composed of one major and two minor components with an average molecular weight of approximately 278. Vacuum distillation of the polyol produced one fraction boiling at 200 - 204 °C (0.045 mmHg) that accounted for greater than 95% of the sample. The elemental composition of the polyol was determined to be 56.0% C, 11.14% H, and 10.07% N. Assuming that oxygen was the remaining element, results in an empirical formula of $C_{13}H_{30}N_2O_4$ (calculated as 56.08% C, 10.86% H, 10.07% N, and 22.99% O).

The infrared spectrum of the polyol, shown in Fig. 13, contained the following absorption bands: O–H stretch (2.7 to 3.2 μm), C–N stretch (8.8 μm), O–H bend (7.8 μm), and C–O stretch (9.4 μm). The sample had a pH of 11.3 in a water–isopropanol mixture, suggesting the presence of an amine functionality.

The ^{1}H-NMR spectrum, shown in Fig. 14, contained a doublet at δ 1.18 and a broad singlet at δ 4.85 with relative areas of 9 protons and 4 protons, respectively. The doublet was

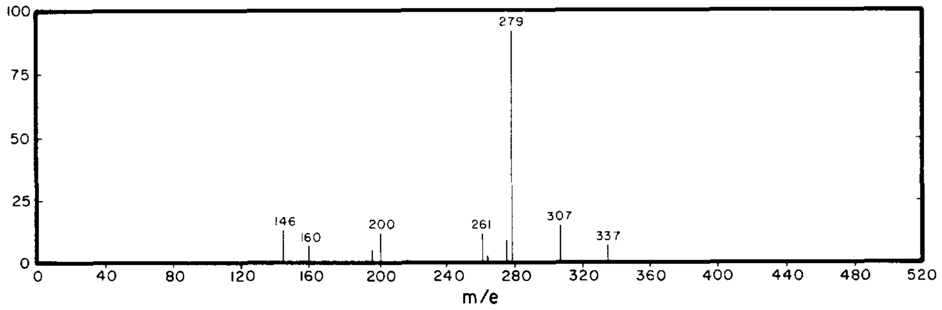


Fig. 12. CI–MS spectrum of unknown foam hydrolysate less the ions from IV.

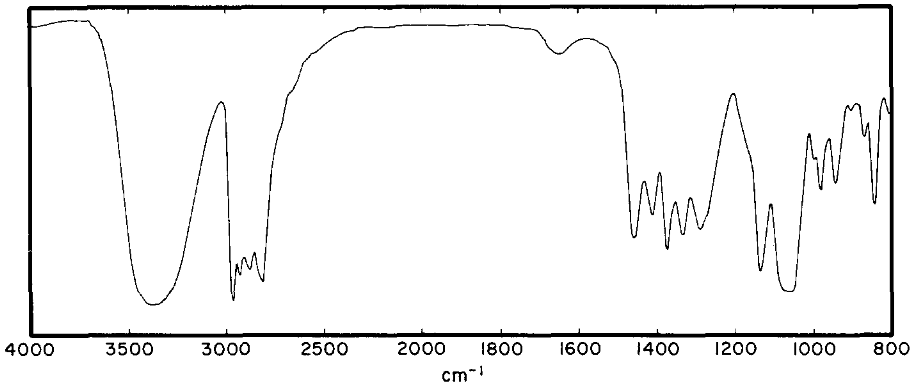


Fig. 13. Infrared spectrum of the Dow polyol.

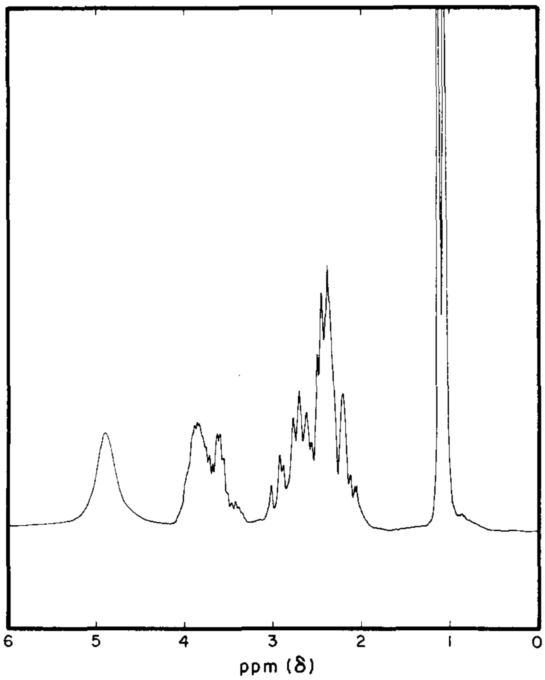
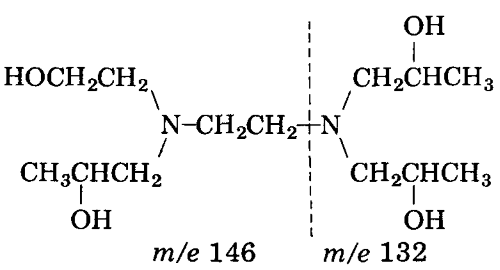


Fig. 14. Proton magnetic resonance spectrum ($CDCl_3$) of the Dow polyol.

assigned to the methyl groups of the propoxy units. The integration corresponds to three propoxy units per molecule. The broad singlet was assigned to hydroxyl protons and, from the relative peak areas, it can be estimated that there is an average of 4 hydroxyl groups per molecule. A compound containing 4 hydroxyl groups with a molecular weight of 278 has a theoretical hydroxyl number of 806.

The configuration of the propoxyl and the hydroxyl groups in the molecule was established from the electron impact mass spectrum of the polyol. Two major fragment ions were observed in the spectrum at *m/e* 132 and 146, and these ions are consistent with the following structure:



Structure V is in agreement with all data that we obtained for this polyol. The narrow molecular weight distribution of the polyol, which is unique among the formulations that were examined in this laboratory, is indicative of a non-catalyzed exhaustive propoxylation of the amine subunit.

Formulation of a comparable foam

The identification of the unknown foam was completed by synthesizing a chemically identical formulation from known components. A foam sample was prepared in our laboratory by the "one-shot" method using the formulation listed in Table 3. Since there were signs of slight charring on the edges of the unknown sample, it was assumed that the unknown foam had been exposed to heat. Our experience with tris-(β-chloroethyl) phosphate fire retardant has shown that it slowly volatilizes from MDI-based urethane foams at 150 °C, despite a boiling point of 330 °C. To compensate for the exposure to heat, the concentration of fire retardant in the laboratory-prepared foam was raised to a level of 1.5% phosphorus loading. A sample of this laboratory foam contained a fire retardant concentration of 10.2% as determined by the previously described extraction procedure.

The laboratory-prepared foam sample was subjected to the analytical testing procedures described in this paper, and the results were compared with data from the unknown foam. The synthesized foam performed identically to the unknown foam sample. However, there were predictable differences in the IR spectrum and the pyrolysis–GC data. The IR spectrum of the two foams were identical except for the presence of a $N=C=O$ stretch (4.38 μm) and a $N=C=N$ stretch (4.7 μm) in the synthesized foam. These absorptions are known to disappear upon sample aging. Pyrolysis–GC analysis of the freshly prepared foam formulation also produced higher concentrations of the blowing agent. With exception of these two data and the fire retardant level, the foam formulations were indistinguishable.

CONCLUSION

Using classical analytical techniques for organic analysis, chemical ionization mass spectrometry and high pressure liquid chromatography, it was possible to characterize and identify the composition of an unknown polymeric material. The chemical information was sufficiently complete that a laboratory-prepared foam was synthesized and shown to be identical to the unknown formulation. The techniques and procedure demonstrated in this paper should be applicable to the identification of many polyester, polyurea, and polyurethane materials.

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TABLE 3

Foam formulation

Weight (g)	Component	Source
34.0	polymethylene polyphenylisocyanate	Mondur, MR, Mobay
16.4	polyol	RA-800, Dow
1.0	silicone surfactant	L-520 silicone, Union Carbide
8.6	tris-(β-chloroethyl) phosphate	Fyrol CEF, Stauffer
8.0	trichlorofluoromethane	R-11, Racon

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