# Urethane Foams from Animal Fats: VIII. Properties of Foams from Epoxidized Tallow Trimethylolpropane Polyols[1]

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## ABSTRACT

Polyols made by reacting trimethylolpropane with epoxidized tallow were converted to urethane foams by reaction with a polymethylene polyphenylisocyanate in the presence of fluorotrichloromethane. Adjusted with triisopropanolamine or an oxypropylated triamine to hydroxyl equivalent of either 100 or 120, the polyols yielded rigid foams of density 1.5-2.0 lb/ft$^3$, open cell content 15-19%, and compressive strength 34-49 psi. These values were superior to those of similar foams from hydrated epoxidized tallow. Polyols made from epoxidized tallow-trimethylolpropane-HBr and adjusted to equivalent wt of 100 and 120 by triisopropanolamine gave foams whose small-scale flammability test samples burned less than 20% of their length. At hydroxyl equivalent 100, foams had density 1.6-1.8, open cell content 20-21%, and compressive strength 34-39 psi; in flammability tests burned <20% of length. The present foams were stronger than those made earlier from solvent-purified hydroxybrominated tallow. Formulation with half the normal amount of freon gave foams of higher compressive strength but lower flame resistance.

## INTRODUCTION

The preparation of polyols by reaction of epoxidized tallow with trimethylolpropane (TMP) and TMP-HBr has been described (1). The polyols are considered as belonging to two series, the toluenesulfonic (TA) and hydrobromic acid (TX) series, respectively. The present paper reports on foam formulations employed and foam properties obtained. As a first step, the polyols, in hydroxyl equivalent 257-165 and 532-243, were blended with either of two hydroxyl-rich low mol wt compounds to lower the hydroxyl equivalent to 100 or 120, as required for castor oil (2). The present polyols, being higher in hydroxyl content than some previous fat-based polyols (3-5) required less of such adjuvants. The adjusted polyols were converted to low density rigid urethane foams by reaction in a one step method with a polymethylene polyphenylisocyanate, following a standard procedure. The foams from bromine containing polyols were tested for fire retardance. The effect upon properties of employing less blowing agent than usual also was appraised in both series.

TABLE I

Adjustment of Equivalent Wt of Polyols

| | Polyol | | Percent adjusting agent for OH[a] equivalent of | | | |
|---|---|---|---|---|---|---|
| | | | 100 | 120 | 100 | 120 |
| No. | OH no. | OH equiv. | TIPA[a] | | NIAX LA-700 | |
| TA 1 | 218 | 257 | 55 | 41 | --- | 52 |
| TA 2 | 257 | 218 | 51 | 35 | 67 | 46 |
| TA 3 | 314 | 179 | 46 | 28 | 62 | 38 |
| TA 4 | 340 | 165 | 44 | 25 | 61 | --- |
| HET[b] | 142 | 395 | 59 | 46 | --- | --- |
| TX 1 | 99 | 567 | 61 | 48 | 75 | 60 |
| TX 2 | 125 | 449 | 60 | 47 | 74 | 58 |
| TX 3 | 231 | 243 | 53 | 38 | 69 | 45 |
| HBT[c] | 92 | 610 | --- | 51 | --- | --- |

[a]TIPA = triisopropanolamine and OH = hydroxyl.

[b]HET = hydrated epoxidized tallow (unpublished data of this laboratory).

[c]HBT = hydroxybrominated tallow, purified (Ref. 5).

## EXPERIMENTAL PROCEDURES

The materials listed were used as received: triisopropanolamine (TIPA) (Union Carbide Corp., New York, N.Y.) (OH no. found, 850); NIAX LA 700, an oxypropylated triamine (Union Carbide Corp.) (OH no. found, 710); dabco-33-LV, 33% solution of triethylene diamine in dipropylene glycol (Houdry Process and Chemical Co., Marcus Hook, Pa.); silicone L530 (Union Carbide Corp.); freon 11, fluorotrichloromethane (Matheson Gas Products, New York, N.Y.); polymethylene polyphenylisochanate (PAPI) (Upjohn Co., Kalamazoo, Mich.).

### Procedures

*Adjustment of equivalent wt:* The equivalent wt of both series of fat-based polyols were adjusted to 100 and 120 by incorporation of required amounts of TIPA or NIAX LA-700. The proportions used are given in Table I.

*Formulation and testing:* The adjusted polyols were converted into urethane foams by a one-step process described previously (3-5). A typical formulation is shown in Table II.

Each formulation was mixed in a paper cup by an electric drill-driven impellor. The resulting foams were stored for 1 month, then cut into samples for measurement of density, open-cells, and compressive strength. Open-cell content was measured by an air comparison pycnometer (3). Relative flammability was measured by a small-scale modification of American Society for Testing and Materials Method D-1692 (4). To avoid misinterpretation of terms, flammability test results are reported in terms of percentage of sample length burned. Experiments also were made using half the normal proportion of fluorotrichloromethane. Test results are reported in Tables III, IV, and V.

TABLE II

Formulation of Adjusted Polyols Converted into Urethane Foams

| Polyol[a] | Wt (g) |
|---|---|
| Tallow-TMP polyol (OH no. 257) | 6.5 |
| TIPA (to adjust OH equiv. to 120) | 3.5 |
| Dabco-33-LV | 0.25 |
| Silicone L530 | 0.15 |
| Fluorotrichloromethane | 4.1 |
| PAPI | 12.3 |

[a]TMP = trimethylolpropane, TIPA = triisopropanolamine, and PAPI = polymethylene polyphenylisocyanate. PAPI was used in 5% excess.

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[1] Presented at the AOCS Meeting, New Orleans, May 1973.

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TABLE III

Foam Properties—Toluenesulfonic Acid Series

| | At OH equivalent 100 | | | At OH equivalent 120 | | |
|---|---|---|---|---|---|---|
| Polyol[a] | Density lb/ft$^3$ | Open-cells, % | Compressive strength, psi | Density lb/ft$^3$ | Open-cells, % | Compressive strength, psi |
| Equivalent wt adjusted with TIPA[b] | | | | | | |
| TA 1 | 1.7 | 17 | 41 | 1.6 | 17 | 36 |
| TA 2 | 1.7 | 17 | 39 | 1.6 | 19 | 34 |
| TA 3 | 1.6 | 16 | 38 | 1.6 | 19 | 37 |
| TA 4 | 1.7 | 17 | 40 | 1.6 | 17 | 38 |
| HET[a] | — | — | — | 1.5 | 100 | 13 |
| Equivalent wt adjusted with NIAX LA-700 | | | | | | |
| TA 1 | — | — | — | 1.7 | 19 | 36 |
| TA 2 | 1.8 | 14 | 49 | 1.7 | 15 | 44 |
| TA 3 | 1.8 | 16 | 42 | 1.6 | 16 | 35 |
| TA 4 | 2.0 | 15 | 60 | — | — | — |

[a] TA = toluenesulfonic acid. HET = hydrated epoxidized tallow, control.
[b] TIPA = triisopropanolamine.

## DISCUSSION

As is seen from Table I, the present two series of polyols have higher OH numbers (and lower OH equivalents) than hydrated epoxidized tallow and purified hydroxybrominated tallow, cited for comparison. Without enrichment, however, even the TA 4 and TX 3 polyols lacked hydroxyl contents high enough to produce rigid low density foams of acceptable open-cell content and compressive strength. Castor oil, with OH no. ca. 165 and OH equivalent 340, typically has been blended with hydroxy-rich polyols of lower mol wt to give OH equivalents of 100 or, less satisfactorily, 120 (2). These equivalents were chosen for the present formulations, and the adjuvants selected were the trifunctional TIPA and the pentafunctional NIAX LA-700. Table I lists the percentages of these materials incorporated in the polyol blends at the equivalents chosen. The present TA and TX polyols required considerably less adjuvant, respectively, than hydrated epoxidized tallow (HET) and hydroxy brominated tallow purified (HBT). Only 25% TIPA was required at the 120 level by polyol TA 4.

Test data on foams made in the normal way from the TA polyols are reported in Table II. Formulated with TIPA at OH equivalent 100, these polyols gave foams of low density, ca. 17% open-cells and compressive strength ca. 40 psi. Open-cells were measured by the full-atmosphere procedure. (The half-atmosphere procedure, used for comparison on a few samples, gave 25% lower values.) Formulated at an OH equivalent of 120, the foams all had slightly lower densities, open-cell content a little higher on the average, and good, but slightly lower, compressive strengths. These foams were clearly superior to a control, based upon hydrated epoxidized tallow. When NIAX LA-700 was the adjusting agent, foam properties were also quite satisfactory from polyols adjusted to either 100 or 120 equivalent wt. Compressive strengths suffered a little at the higher equivalent.

The test results on the foams made in the standard way from the TX or brominated polyols are reported in Table IV. Adjusted to equivalent wt of 100 by TIPA, these polyols gave foams with density and compressive strength approximating those from the unbrominated polyols at equivalent 120. However, in the brominated series, open-cell content was a little higher, at ca. 20%, than the unbrominated series. At equivalent 120 attained with TIPA, both open cells and compressive strength suffered substantially, except for the TX 3 polyol. Each of the three TX polyols gave foams burning less than 20% test sample length. The TX 3 polyol emerged as the one least impaired by the higher equivalent wt of 120. The last named gave foams superior in compressive strength and fire-resistance to those from purified hydroxybrominated tallow, described previously (4).

The brominated polyols also were adjusted with NIAX LA-700. At OH equivalent 100, the compressive strengths were particularly good from each TX polyol, while open-cell content was variable. With NIAX LA-700, the flamma-

TABLE IV

Foam Properties—Hydrobromic Acid Series

| Polyol | | | | | | |
|---|---|---|---|---|---|---|
| No. | OH no. | Percent br. | Density, lb/ft$^3$ | Open-cells, % | Compressive strength, psi | Flammability, percent sample length |
| Adjusted with TIPA[a] to OH equivalent of 100 | | | | | | |
| TX 1 | 99 | 42 | 1.6 | 21 | 34 | < 20 |
| TX 2 | 125 | 30 | 1.7 | 20 | 39 | < 20 |
| TX 3 | 231 | 25 | 1.8 | 20 | 36 | < 20 |
| Adjusted with TIPA to OH equivalent of 120 | | | | | | |
| TX 1 | | | 1.7 | 30-50 | 16 | < 20 |
| TX 2 as above | | | 1.8 | 38 | 29 | < 20 |
| TX 3 | | | 1.8 | 21 | 35 | < 20 |
| HBT | 92 | 14 | 1.9 | 19 | 19 | 20-80 |
| Adjusted with NIAX, LA-700 to OH equivalent of 100 | | | | | | |
| TX 1 | 99 | 42 | 1.8 | 19 | 44 | < 20 |
| TX 2 | 125 | 30 | 1.9 | 23 | 44 | 20-80 |
| TX 3 | 231 | 25 | 1.9 | 16 | 43 | > 80 |
| Adjusted with NIAX, LA-700 to OH equivalent of 120 | | | | | | |
| TX 1 | | | 1.8 | 19 | 41 | < 20 |
| TX 2 as above | | | 1.7 | 20 | 38 | 20-80 |
| TX 3 | | | 1.7 | 16 | 34 | > 80 |

[a] TIPA = triisopropanolamine.

TABLE V

Effect of Decrease in Blowing Agent upon Foam Properties— Use of Half the Standard Proportion of Fluorotrichloromethane

| Polyol[a] | Density, lb/ft$^3$ | Open-cells, % | Compressive strength, psi | Relation r to density[b] | Flammability, percent sample length |
|---|---|---|---|---|---|
| TA 1 | 2.9 | 14 | 80 | 1.3 | > 80 |
| TA 2 | 2.6 | 15 | 71 | 1.4 | > 80 |
| TA 4 | 2.8 | 15 | 75 | 1.3 | > 80 |
| TX 1 | 2.5 | 17 | 64 | 1.4 | 20-80 |
| TX 3 | 2.7 | 16 | 70 | 1.6 | 20-80 |

[a] Adjusted with triisopropanolamine to OH equivalent of 100.

[b] In equation $s/s_0 = (d/d_0)^r$ (ref. 6). Foams made with the standard proportion of fluorotrichloromethane had compressive strength $s_0$ and density $d_0$, reported in Tables III and IV.

bility rating deteriorated to 20-80% and to >80% test sample length for the TX 2 and the TX 3 foams; the high bromine content of the TX 1 foams assumed greater importance. At an OH equivalent of 120, test results were analogous.

In general, the present fat-based polyols, both unbrominated and brominated, gave foams of better quality than those from HET and HBT, and on a par with those made from alkoxylated dihydroxystearic acids (7,8). The polyols responded well to blending with adjuvants, providing foam properties at equivalent wt 120 only a little inferior to those at 100. Properties were comparable to those of foams based upon vegetable oils (2,9).

### Decreased Blowing Agent

Because of an interest in foams of higher density than those considered up to now, a number of experiments were carried out using only half as much blowing agent as normally. Table V shows test values on such foams from polyols adjusted with TIPA to OH equivalent 100; these are to be compared with corresponding entries in Tables III and IV. Halving the freon used increased densities by 50-70%. It decreased open-cell content by 12-20% and doubled or slightly less than doubled compressive strength. Through the equation given in footnote b, Table V, compressive strengths at different densities were related by the exponent r. Values of r of 1.3-1.6 were encountered in these data, compared to 1.6 in low density rigid foams, based upon castor polyols (6). The flame tests were surprising for the brominated polyols. The denser foams burned 20-80% test sample length, i.e. they were not quite as fire-resistant as their counterparts of low density, which had burned less than 20% length. Apparently, the freon itself has a little fire retardance, or the greater density more effectively maintains heat at the flame front.

## ACKNOWLEDGMENT

E. Beatty cut foam samples.

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[Received September 20, 1973]