Analytical Report

AURIGA POLYMERS INC.
1550 Dewberry Road
Spartanburg, SC 29307
U.S.A.
[864] 579-5008 Tel
[864] 579-5972 Fax
Brad.Smith@US.Indorama.net

To: **Joe Drbohlav**

From: **Brad L. Smith**
Analyst:

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Subject: **Ethylene Glycol (EG) Migration from Half-Liter PET Bottles**

SUMMARY

The migration of ethylene glycol from 1/2-liter PET bottles into water which is held at 40 °C and room temperature over a four week period is measured. The results show that at 40 °C, after four weeks the bottle contents contain 57 ppb EG. Likewise, at room temperature after four weeks the bottle contents contain 23 ppb EG.

INTRODUCTION

The California EPA office of Environmental Health Hazard Assessment (OEHHA) intends to list ethylene glycol (EG) as being known to cause reproductive toxicity. The request has been given to the Auriga R&D Analytical lab to provide information on EG migration from PET bottles. This report documents the work performed to quantify the free EG which migrates from PET half-liter bottles into water.

RESULTS AND DISCUSSION

Experimental

Twenty-four gram, ½-liter PET bottles were prepared at the Auriga bottle lab from 3301 PET resin (a water grade resin produced by Auriga Polymers). Twelve of these bottles were rinsed and then filled (470 g) with DI water. The bottle openings were covered with aluminum foil and then capped. Six of these bottles were placed into a 40 °C oven and six were placed in a cabinet at room temperature (temperature in the cabinet was not controlled). After four weeks, three of the bottles at 40 °C and three of the bottles at room temperature were taken and the contents poured into twelve glass containers respectively to end the migration portion of the study. The PET bottles were rinsed with DI water and the rinse was added to the glass container until a total of 500 grams of

water were in the glass bottles. The remaining six bottles (three at 40 °C and three at room temperature) are held in the oven and in the room temperature cabinet for an additional two weeks (total of six weeks). The results of the six week study will be reported separately.

Evaporation Procedure. All liquid samples obtained from the PET bottles as well as standards prepared for validation of the method follow the same evaporation procedure.

Each liquid sample (500 mL) was poured into a 600 mL beaker. A Teflon stir bar was added and each beaker was placed into an 85-95 °C water bath on a hot plate. The water contents were allowed to stir and evaporate until about 50 ml of water remained at which time the liquid was transferred to a 100 mL beaker and the beaker was returned to the water bath for continued evaporation. Once approximately 5 mL of liquid remained, the contents were transferred to a 20 mL vial. The beaker was rinsed with DI water and the rinse was added to the vial until the vial contained 10 grams of liquid. Each sample from the PET bottles was analyzed in triplicate for EG according to the following GC parameters:

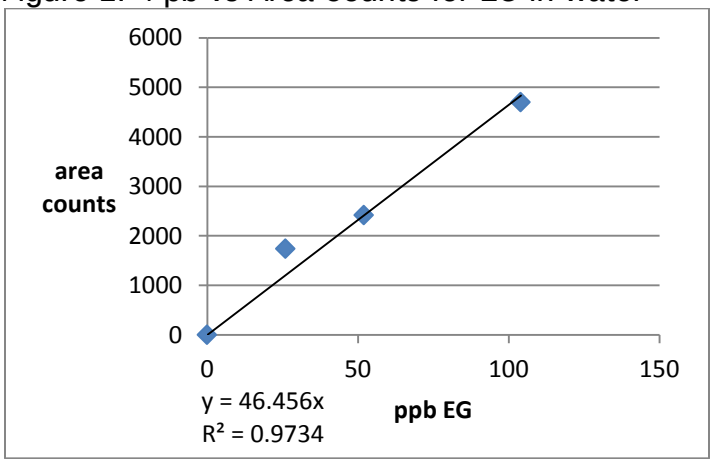
Instrument: HP 5890-plus
Column: RESTEK Stabilwax®-DA, 30 meter, 0.53 mmID, 1 μm DF
Injection: 2 μ
Temperature program:
- Injector Temperature: 240 °C
- Detector Temperature: 250 °C
- Initial temperature: 120 °C for 2 minutes
- Ramp: 10 °C/minute to 220 °C
- Hold: 5 minutes

Water, (2μ) was injected onto the column twice between each analysis to ensure any EG carryover was minimized.

Validation of Method

Standards containing 104, 52 and 26 ppb EG are prepared in 500 mL of water respectively. The water was evaporated according to the procedure as outlined above. Each standard once concentrated to 10 grams was analyzed by GC to determine the area counts for the EG peak. The results are shown in Figure 1.

Figure 1. Ppb vs Area Counts for EG in water



ppb EG	area counts
52	2416
0	0
104	4698
26	1741

Sample Analysis

Four Week Study – 40 °C and Room Temperature. The results from the 4-week trial at 40 °C are shown in Table 1. The EG migration data is obtained in triplicate (three bottles tested) with each PET bottle sample analyzed in triplicate. Good reproducibility is observed between each bottle analyzed and each replicate injection, Table 1.

Table 1. 40 °C, 4 weeks

	Injection 1	Injection 2	Injection 3	Average		
Bottle#	area counts	area counts	area counts	area counts	std dev	%variance from average
1	2739	2611	2640	2663	67	0.09
2	2660	2693	2697	2683	20	0.84
3	2619	2618	2671	2636	30	-0.94
			average	2661	24	

Ave Area Cts of all pts	std dev	% error
2661	43	1.6

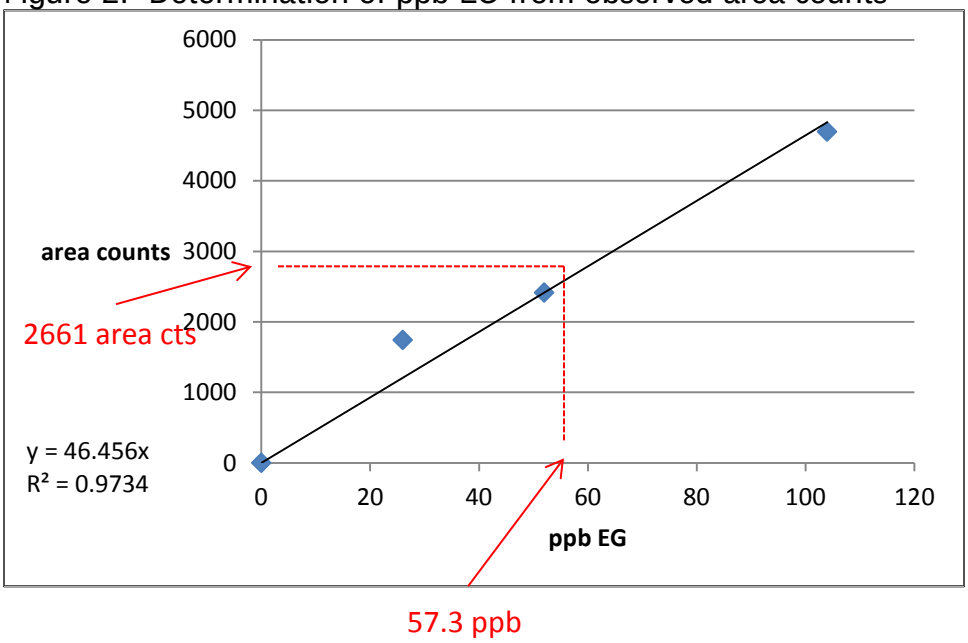
The same procedure was followed for the three sample bottles at room temperature for 4 weeks. Table 2 shows the results for the 4 week, room temperature trial.

Table 2. Room Temperature, 4 weeks						
	Injection 1	Injection 2	Injection 3	Average		
Bottle#	area counts	area counts	area counts	area counts	std dev	%variance from average
1	1175	1123	1071	1123	52	5.63
2	1132	1048	1038	1073	52	0.90
3	916	1023	1042	994	68	-6.53
			average	1063	65	

Ave Area Cts of all pts	std dev	% error
1063	75	7.1

The concentration of EG that migrates into the water in the PET bottles can be determined by comparing the GC area counts for the samples as shown in Tables 1 and 2 with the validation curve of standards, Figure 2. As shown in Figure 2, the average GC area counts for EG in the PET bottle water which is held at 40 °C for 4 weeks is 2661 and corresponds to **57.3 ppb**. In like manner, the average GC area counts for EG in the PET bottle water at room temperature for 4 weeks is 1063 which corresponds to **22.9 ppb**.

Figure 2. Determination of ppb EG from observed area counts



EG Migration as a Function of PET Surface Area

The total number of grams of EG which migrate from the ½-liter PET bottles held at 40 °C for 4 weeks is:

57.3 parts/1000000000 * 500 grams = 0.00002865 grams

The total number of grams of EG which migrate from the ½-liter PET bottles at room temperature for 4 weeks is:

22.9 parts/1000000000 * 500 grams = 0.00001145 grams

The total surface area calculated for the ½-liter bottle is 55.65 sq inches (see Appendix).

Therefore the EG migration at 40 °C and room temperature over 4 weeks time is as follows:

40°C, 4 weeks

EG migration = 0.00002865 grams EG / 55.65 in^2 = 5.15 x 10^{-7} g EG / in^2

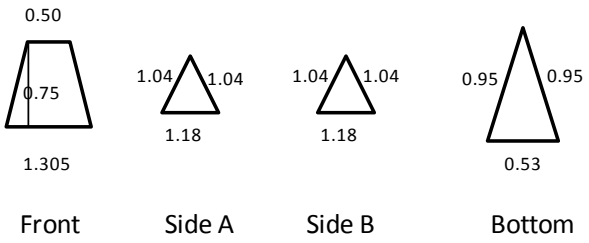
Room Temperature, 4 weeks

EG migration = 0.00001145 grams EG / 55.65 in^2 = 2.06 x 10^{-07} g EG / in^2

APPENDIX - Calculation of Bottle Surface Area

The ½-liter bottle is cylindrical with a pentaloid base. The area of the base is calculated and added to the area of the cylindrical part of the bottle. The neck of the bottle is not in contact with the water and therefore is not included in the area

Each pentaloid section consists of 4 sides:



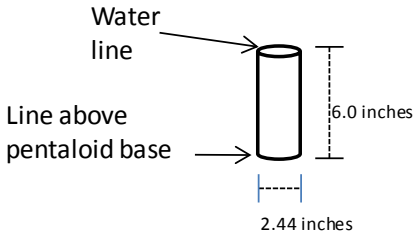
Front: $A = (b1+b2)/2 + h$; $A = (0.50+1.305)/2 \times 0.75 = 0.677$ sq in;
There are five fronts, total area = 5 x 0.677 sq in = 3.385 sq in

Side: $A = b * \text{sqrt}(4a^2 - b^2)/4$; $A = 1.18 * \text{sqrt}(4(1.04)^2 - (1.18)^2)/4 = 0.505$ sq in
There are 10 sides, total area = 10 x 0.505 = 5.06 sq in

Bottom: $A = b * \text{sqrt}(4a^2 - b^2)/4$; $A = 0.53 * \text{sqrt}(4(0.95)^2 - (0.53)^2)/4 = 0.24$ sq in
There are 5 bottoms, total area = 5 x 0.24 = 1.21 sq in

Total Pentaloid surface area: 3.385 + 5.06 + 1.21 = 9.655 sq inches

Surface Area from remainder of PET bottle:



$A = 2\pi rh$; $A = 2 \times 3.1414 \times (2.44/2) \times 6$;
$A = 45.99$ sq in

Total bottle surface area = 9.655 + 45.99 = 55.65 sq inches