February 7, 2003

DRAFT Study Plan: Impurity Analysis of Products

The following is a proposed study plan for the analysis of PFOA in 11 different telomer-based products (see below). Samples will be provided by TRP member companies for analysis. Analyses need to be completed by May 2003.

Product Types: acrylate and urethane polymers (11 product formulations)

The proposed study is designed to determine whether PFOA is present in each product, and if it is present, at what levels and finally the confidence of those reported levels. This study is a *screening study*. Matrix complexities may make these analyses particularly challenging.

PROPOSED APPROACH

-An LC/MS/MS methodology will be used for these analyses having lower limit of quantification of 1 ppm with a relative standard deviation of 20%.

-The study will be conducted as a GLP study.

-Assumes that extraction procedures submitted here will be successful for all 11 product formulations.

> Procedure I assumes that to a known amount of emulsion, methanol is added, shaken for a period of time, and centrifuged at 3000 rpm for 15 minutes. The supernate is diluted 1 to 100x with water, filtered and analyzed by LC/MS/MS.
> If necessary, methanol is added to the residue from the first extraction, sonicated, centrifuged and decanted. The supernate is diluted with water 1 to 10 x, filtered and analyzed by LC/MS/MS.
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> Procedure II:
> Method: Soxhlet
> -Add methanol to the emulsion and collect polymer as precipitate
> -After filtering the supernatant, analyze by LC/MS/MS
> -Dry the precipitate at low pressure, pack in the filter, and weigh
> -Using 140 mL of methanol, extract for 8 hours
> -Cool, and using methanol, bring extraction fluid to 200 mL and analyze by LC/MS/MS.
>
> Internal standard is used throughout. Procedure is time consuming.

-Extraction efficiencies should be evaluated on at least two or three product groups using both approaches. Prefer that heat not be added to sample.

-Can real blank/control be obtained/generated in laboratory? No.

-Extraction recoveries should be evaluated at 1 ppm, 10 ppm and 100 ppm. Analysis will use standard addition approach.

-Triplicate samples analyzed per formulation

-LC/MS/MS method developed for articles should be adapted for analyses of products - however, product composition may require method re-work.

-Final report will be requested; data summary will be acceptable for May 2003 submission.