

Telomer Research Program



Clariant



Analysis of Telomer-Based Products and Telomer-Treated Articles

Scope and Rationale

The Telomer Research Program (TRP)¹ has studies underway to investigate the potential formation and/or release of perfluorooctanoic acid (PFOA) from telomer-based products and consumer articles treated with such products. The purpose of this work is to determine whether articles treated with representative telomer-based products contribute to significant human or environmental exposure to PFOA. The full scope of the program is outlined in the TRP Letter of Intent.²

Project Overview

The TRP has contracted with an outside research laboratory to analyze for the presence of PFOA in the major telomer-based products in commerce and articles treated with those products. This *screening study* will investigate levels of PFOA in the three major end-use categories: carpets, textiles and paper. The primary products that the TRP members market for these applications are *polymers*, which represent approximately 85% of the telomer-based products that the TRP members manufacture and sell. The product classes to be tested are those that the respective TRP member companies manufacture as actual items of commerce.

While there are many potential telomer-based polymeric products, a relatively small number represent the primary product classes in commerce (12 products). The results of this screening study will provide a comprehensive initial picture of exposure potential by focusing on these major end-use polymeric products and the corresponding articles of commerce. The treated articles to be tested are actual carpet, textile, and paper substrates treated under representative or actual mill conditions with the subject telomer-based polymeric products.

Approach and Methodology

Method Development and Validation

A significant amount of work was required to develop and coordinate the appropriate sampling, shipment and analysis plans as well as to synthesize and characterize primary and [1,2-¹³C] perfluorooctanoic acid analytical standards. This initial work was conducted by analytical experts from the TRP member companies in parallel with efforts to solicit bids from potential contract labs. The initial work by the contract lab will require method development for quantitative determination of PFOA in the various article matrices and telomer-based products as well as

¹ The companies participating in the TRP include Asahi Glass, Clariant GmbH, Daikin Industries and E.I. duPont de Nemours & Company.

² Letter of Intent for the Telomer Research Program submitted to Stephen L. Johnson (US EPA) on March 14, 2003. Ref. AR226-_____.

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validation of the methodology for determining PFOA in treated articles. More on method development and method validation is discussed below.

Article Analysis

Samples of the 12 telomer-treated articles will be collected from pilot plant facilities or from commercial manufacturing facilities and analyzed for PFOA using the developed and validated analytical method. The study is designed as a *screening study* to determine whether PFOA is present in each treated article collected, and if it is present, at what levels and the confidence of those reported levels.

Types of Articles: representative textile, carpet and paper articles (12 total)

An LC/MS/MS methodology using a [1,2-¹³C] perfluorooctanoic acid internal standard will be used for these analyses having the objective of attaining a lower limit of quantification of 1 part per billion (ppb) with a relative standard deviation of less than 20%.

Method development will be conducted with the goal of having extraction efficiencies fall within 70-110% for three matrices -- carpet (nylon), textile (polyester) and paper -- at the three PFOA fortification levels of 1 ppb, 10 ppb and 100 ppb.

One hundred sixty two (162) untreated shaved-carpet, textile and paper samples were generated for the method development and method validation portion of this study. If scouting experiments show acceptable recoveries at the levels noted, the contract lab will proceed with method validation.

Method Validation for PFOA in Articles:

The method for the determination of PFOA in treated articles will be validated at 1 ppb, 10 ppb and 100 ppb in each of carpet (nylon), paper (cellulose) and fabric (polyester) under Good Laboratory Practices (GLP). Method validation will comprise of demonstrating the accuracy and precision of the method by analyzing six samples spiked at each fortification level on two separate days. Hence, 36 samples for each article -- 6 each at 1 ppb, 10 ppb and 100 ppb on each day -- will be analyzed for a total of 108 analyses over the two-day period of the method validation. Analysis of articles will not proceed until method validation and protocol for analysis of articles has been approved.

Samples: Each of the 12 articles tested will be treated with a single telomer-based polymeric product. Six (6) sub-samples will be analyzed for each article/product pair, selected using a block design approach. Seventy-two (72) sub-samples for articles (12 treated articles x 6 sub-samples) will be generated and twelve (12) products will be collected for analysis resulting in four unique carpet/product pairs, six unique textile/product pairs and two unique paper/product pairs. Included for each of the 12 articles will be 4 untreated samples to be used as control and for extraction efficiency verification.

Product Analysis

This screening study is designed to determine the level of PFOA in each product class using an LC/MS/MS method. Matrix complexities may make these analyses particularly challenging.

Product Types: acrylate and urethane polymers (12 product formulations)

Samples of the 12 primary telomer-based polymeric products will be provided by TRP member companies to the contract lab for analysis. A standard addition approach using LC/MS/MS methodology is being used for these analyses of telomer-based products, having lower limit of quantification for PFOA of 1 part per million (ppm) with a relative standard deviation of 20%. A [1,2-¹³C] perfluorooctanoic acid internal standard will be used throughout the study. Before the product samples are analyzed, extraction efficiencies will be evaluated on at least two or three product groups using modified methodologies supplied by the TRP. Product analyses are being conducted under GLP.

Results

The results of this screening study will be prepared as a report, including the following details:

Summary of Method and Results

Materials used

Equipment used

Reagents and standards used

Analytical Procedures

Preparation of stock standard solution, preparation of fortification of articles and preparation of calibration standards

Pre-processing of samples, sample fortification procedures, analyte extraction and final sample preparation procedures

Instrumentation

LC/MS conditions

LC/MS Calibration and Samples Analysis

Calculation Methods

Results and Discussion

Detector response

Control and/or Blank

Recoveries

Extraction Efficiency

Limit of Quantification and Limit of Detection

Conclusions

Schedule

Work by the TRP analytical experts began in January 2003 to develop the appropriate sampling, shipment and analysis procedures as well as primary and [1,2-¹³C] perfluorooctanoic acid analytical standards for the proposed work. In concert with this effort, potential contract labs were identified and bids were solicited. The selected laboratory received the final study plans and sample requirements in February 2003. The internal standard was prepared by one of the TRP companies and delivered to the lab in March 2003.

The initial work to develop and validate analytical and sample methods will be conducted in March–April 2003. The analysis of telomer-treated articles and telomer-based polymeric products will be initiated in April 2003. The final report is expected by June / July 2003. The results will be provided to EPA as soon as they become available.