AR226-0562

Study Title

Analytical Laboratory Report on Presence and Concentration of Potassium Perfluorooctanesulfonate in Serum and Liver of Sprague-Dawley® Rats Exposed to PFOS via Gavage

REPORT AMENDMENT NO. 1

Amendment Date:

19 April 2000

Performing Laboratory

3M Environmental Technology & Safety Services
3M Environmental Laboratory
935 Bush Avenue
St. Paul, MN 55106

Laboratory Project Identification

ET&SS LRN-U2006
ET&SS FACT Tox-012

This amendment modifies the following portion(s) of the final report:

1. ***FINAL REPORT:***
Make the following addition to the final report text.
ADD:
After the final report was issued (signed and archived), it was discovered that the purity value stated for the analytical reference substance was inaccurate. This value was based only on NMR analyses. Subsequent chemical characterization is occurring to determine the actual purity of theses substances (PFOS Lot 193). If necessary, the final report will be amended to detail the analytical results using the purity value from the certificate of analysis when it is issued for PFOS Lot 193.
REASON:
To describe the changes that will be made to the final report when the certificate of analysis is issued for PFOS Lot 193.

2. ***FINAL REPORT:***
Make the following addition to the exceptions list in the Statement of Compliance section.
ADD:
   - The purity and stability of the analytical reference substance is unknown and was not determined prior to the initiation of this study.

REASON:
The Statement of Compliance section was incomplete.

3. ***FINAL REPORT:***
The purity of the analytical reference substance is 99.28% (Lot 193), as listed in Table 2.
AMEND TO READ:
The purity of the analytical reference substance (PFOS Lot 193) listed in Table 2 is unknown.
REASON:
The purity value stated for the analytical reference substance is incorrect.

4. ***FINAL REPORT:***
Make the following addition to the final report text under circumstances that may affect the quality of the data.
ADD:
   - According to the Dilution Summary Sheet, the extracts for F0 liver samples 8243M, 8244M, 8249M, 8250M, and 8255M were diluted 0.001mL into 1.0mL (1:1000), but were calculated as 1:2000. On the same sheet, the extracts for F0 liver sample 8181M and F1 liver sample 8937 Pup were diluted 0.001mL into 1.0mL (1:1000), but were calculated as 1:500.
   - The result for 8249M liver sample was quantitated based on peak response that exceeds the highest calibration standard by 20%.

REASON:
To include the circumstances that were found after the final report for this study was issued.

5. *FINAL REPORT:*

On page 4 of 16, a print error occurred that replaced several lines of the Exceptions to GLP compliance text with an "X" character, without any change in the spacing between the subsequent bullets on the rest of the page. The incorrect page will be replaced in the final report and it will archived in the study folder.

AMEND TO READ:

The correct page 4 of 16 is attached to this amendment. The following list is the complete text lost in the print error:

- The identity, strength, purity, and composition defining the test or control article was not determined at the time of the study; however, these analyses were performed on the lot of PFOS used subsequent to the present study.
- Two separate study directors were assigned to the *in vivo* and the analytical portions of this study.
- From 15 September 1998 until 1 October 1998, the protocol was not sponsor approved and was during that time period not in compliance.
- Deviations were not consistently approved by the study director as required by GLP regulations.
- Data corrections were not always recorded as required by GLP regulations.
- Data were not always attributed to the individual responsible for recording the data.

REASON:

The final printed report with the incorrect page does not reflect the complete report document as reviewed by the study director and sponsor representative.

Amendment Approval

Marvin T Case — 19 April 2000

Marvin T. Case, D.V.M., Ph.D., Sponsor Representative — Date

Kristen Hansen — 04/19/00

Kristen J. Hansen, Ph.D., Study Director — Date

STATEMENT OF COMPLIANCE

Study Title: Analytical Laboratory Report on Presence and Concentration of Potassium Perfluoro-octanesulfonate in Serum and Liver of Sprague-Dawley® Rats Exposed to PFOS via Gavage

Study Identification Number: FACT Tox-012

This study was conducted in compliance with Food and Drug Administration Good Laboratory Practice (GLP) Regulations for Nonclinical Laboratory Studies [Data Requirement(s): 21 CFR (Part 58)], with the exceptions in the bulleted list below. In addition, the present study has been audited retrospectively by an independent quality assurance unit. Audit procedures and findings for audits performed at the 3M Environmental Laboratory and at participating contract laboratories are housed with documentation pertinent to this study in archives at the 3M laboratory and will be retained for at least 10 years. The analytical portion completed at the 3M Environmental Lab was performed in accordance with 3M Environmental Technology and Safety Services Standard Operating Procedures.

Exceptions to GLP compliance:

- Storage containers for the reference substance, PFOS, were not labeled with name, CAS number, batch number, expiration date, or storage conditions.
- Details of the preparation, maximum storage time, and stability properties of the reference substance, PFOS, are unknown.
- The identity, strength, purity, and composition defining the test or control article was not determined at the time of the study; however, these analyses were performed on the lot of PFOS used subsequent to the present study.
- Two separate study directors were assigned to the *in vivo* and the analytical portions of this study.
- From 15 September 1998 until 1 October 1998, the protocol was not sponsor approved and was during that time period not in compliance.
- Deviations were not consistently approved by the study director as required by GLP regulations.
- Data corrections were not always recorded as required by GLP regulations.
- Data were not always attributed to the individual responsible for recording the data.
- Records in electronic form do not meet the criteria set forth under 21 CFR(11).
- A finalized sample log-in /tracking system was not in place at the time of the study; however, draft documents did exist and were used to log in and track samples.