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2nd Draft Trip Report

Visit to National Medical Services Laboratory (NMS)

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Summary

At the request of Barbara Dawson (DuPont DCSE), on January 29, 2001, Daryl Cobranchi and I visited National Medical Services Laboratory in Willow Grove, Pennsylvania, to review their data and procedures for the determination of perfluorooctanoic acid (PFOA) in blood samples taken in 1999 and 2000. After a discussion of the data and procedures with two of the scientists involved in recent data collection, we concluded that the concentrations reported for samples 386340-386389 and 452284-452329 were below the level of quantitation. The reported numbers are also not meaningful since the calculated values were significantly below the level of the lowest standard that the laboratory had run. We requested that NMS run lower concentration standards in the future and that they look at some common interferences to ensure that there was not a problem with coeluting peaks.

Detailed Report

National Medical Services (NMS) is a full-service, independent laboratory featuring a rather comprehensive schedule of analytical and consultative services in therapeutic drug monitoring, environmental / occupational toxicology, forensic toxicology, criminalistics and research &development. They work almost entirely with biological samples such as blood, urine, hair, serum, and plasma. They are accredited or licensed by 16 agencies such as the DEA, Pennsylvania Department of Health, and the College of American Pathologists. NMS is involved in ongoing proficiency testing with a number of these agencies. In May, 1999, Sue Mileti (DCSE), Barbara Larsen (CRD, CCAS), Barbara Dawson, and I visited NMS to see if they had the resources to do some additional development work for DuPont as well as to review the status of the PFOA in blood measurements they had done in the past. We concluded then that they had the resources in place to continue to measure PFOA in blood at the levels that they had measured in the past, but they did not have the personnel to do additional method development.

The NMS method for PFOA in blood is based on a method developed by DuPont in the 1980s. DuPont Polymer Products analytical group (now CCAS) used this method at the Experimental Station to measure PFOA levels in blood of DuPont workers from Washington Works, West Virginia. It involves adding an internal standard of perfluorodecanoic acid and sodium hydroxide to the whole blood, which is then freeze-dried. The acids are then derivitized to their methyl esters with methanolic hydrochloric acid, which is then extracted into hexane. Analysis is done by high-resolution capillary gas chromatography with electron capture detection (ECD) on a Hewlitt-Packard (now Agilent) 5890 gas chromatograph.

On January 29, 2001, Dr. Daryl Cobranchi (Sr. Res. Chem., CRD, CCAS) and I visited NMS to review recent data and procedures for the determination of PFOA in blood samples taken form workers at the DuPont Chambers Work site in Deepwater, New Jersey. Mr. Matthew McMullin, Director of Therapeutic Drug Monitoring and Clinical Toxicology, met us and introduced us to Dr. Laura Madrid, Group Leader for the Environmental Testing Section and Mr. Harry Garcia Flores, Chemist, in Dr. Madrid's group. Dr. Madrid has been head of the section since August 2000. She has a Ph.D. in toxicology. Mr. Flores is a bachelor's level analyst who has been with NMS since May 2000. Due to the short period of time between our request to review the data and our visit to the facility, NMS was not able to produce the raw data and reports on the 1999 samples. Unfortunately the chemist and the supervisor responsible for producing and approving the data and report were no longer with NMS so we were unable to interview them. We decided to concentrate then on a review of the 2000 data and procedures. Mr. Flores noted that he had a problem with linearity of the calibration curve. He also noted that he was uncomfortable reporting numbers lower than the lowest level quality control standard (100 parts per billion, (ppb)). Since all the levels that they reported were well below the 100-ppb level and since the calibration curve was not linear, we concluded that it was inappropriate to report specific numbers for these samples (386340-386389 and 452284-452329). Mr. Flores also pointed out that many of the samples showed high levels of background, making it difficult to ascertain if the peak was really due to PFOA. Since the measurement relies only on retention time and response by the ECD, other materials that come off the chromatographic column at the same time (coeluting peaks) might give a positive response, even if no PFOA were present and would be incorrectly reported as PFOA. We asked if NMS has looked for possible coeluting peaks. They had no recollection of having checked this system for coeluting peaks, so we asked them to check common species they generally found in blood such as nicotine, caffeine, and fatty acids which also might give a positive response to the ECD. We also asked them to give more information on reporting the results so that it would be clearer to us what they might have found.

Recommendations/Path Forward

In addition to looking for coeluting interferences, they will run a 50 ppb standard as part of their usual quality control process. If they see no peaks at the retention time of the methyl ester of PFOA, they will report "not detected". If they see a peak at the right retention time at less than the 50 ppb level (detectable, but not quantifiable), they will report it as "detected but below the level of quantitation" (LOQ). They will estimate the LOQ for each sample set.