

All QC runs and samples associated with batches 11/21/01, and 12/19/01 gave typical results similar to all the previous batches analyzed on the new Agilent GCMS for method 525.2 with the following exception: the Endrin breakdown result was suddenly greater than 70%. Unfortunately this was not realized until 3 weeks after the injections were made. The instrument was removed from service and a thorough investigation was begun to attempt to determine and correct the causes of the high Endrin breakdown as well as low recoveries of several other less stable compounds. Involved in our investigation was Agilent staff, Finnigan staf., our 2 service consultants from MDL Systems, our gas suppliers - Air Gas Corporation, and members of our staff. Simultaneously, 4 other instruments in the laboratory suddenly experienced high Endrin breakdown also.

Around January 22, the problems were isolated and for the most part corrected for each instrument. The causes were due to a combination of various problems, some which may have been building over time, i.e. bad vial of Endrin standard, contaminated gas lines and gas purifying traps, much too high injection port temperatures, bad lot of GC septa, improper design of Finnigan Ion Trap injection port, and inherently poor design of Agilent GC injection ports. Though we can not be conclusive as to which causes contributed the most to the problem, and which ones affected each instrument exactly, all in all with many corrections made, we believe we are now able to control the Endrin breakdown levels. In addition, we observed improved recoveries thus far for the 18 compounds analyzed by our method 525.2.

In order for me to reaffirm the reliability of data obtained from the original injections for these 2 batches, we reinjected them and compared the new data against the original data. All results agree with improved responses for problem compounds and surrogates. The Endrin breakdown was 10.6%. Thus, I reported the originally obtained data but the newly obtained Endrin % breakdown result.

David Vinn
1/30/12