Deviation from Documented Procedures Form

Date of Deviation: March 14, 2022 – May 3, 2022

Type of Deviation: GC/MS Method for late-eluting drugs

Describe the Deviation:
Exhibits 2 and 3 in case BCCL-21-1917 were requested to be worked per laboratory practices. An analyst ran exhibits 2 and 3 on Thursday, March 10, 2022, on GC/MS 7890B/5977A (Instrument #3) using the usual method, DrugTest35. The data was not reviewed until the following Monday, March 14, 2022. Sample 2.1 and 3.1 both did not appear until after the second blank after the run. The analyst then ran the same samples on Monday, March 14, on GC/MS 6890/5973 (Instrument #1) with a different method, DrugTest_LM, assuming the method would be longer. A different instrument was used due to the columns being different between Instrument #1 and #3. The DrugTest_LM method is also a hotter method than DrugTest35. This allowed for the identification of sample 2.1, Tadalafil, but sample 3.1 still carried over into the blank. However, this method was not used for confirmation due to the finding that the method was not verified. On Wednesday, March 16, both samples 2.1 and 3.1 were ran again on Instrument #1 under a different method, DrugTest50-FHTI. This altered method comprise a longer oven time, from 6.75 minutes in DrugTest50 to 21.75 minutes in DrugTest50-FHTI, and was able to generate usable data. This Category A identification using mass spectroscopy, in addition to the pharmaceutical identifiers on the blister packs, allowed the analyst to confirm the identity of the samples. Sample 2.1 was identified as Tadalafil and sample 3.1 was identified as Sildenafil.

The only exhibit, Exhibit #1, in BCCL-20-1408 was originally run March 21, 2022, on GC/MS 7890B/5977A (Instrument #3). There was only one blank between each sample. Upon review, there was no carryover in the blank; however, there was carryover into the next sample after the blank. Carryover indicated the sample contained Sildenafil. Due to no pharmaceutical identifiers or any other Category B identification, a standard was ordered. The standard was run on March 25, 2022, on GC/MS 6890/5973 (Instrument #1) using the DrugTest50-FHTI method. On March 27, 2022, the sample was run on Instrument #1 using the DrugTest50-FHTI method. This longer method was able to produce usable data for the sample. Having the mass spectroscopy and retention time of the sample and comparing it to the standard allowed for the identification of Sildenafil.

CS-19-05
"5.3 Analysts shall inject blanks / negative controls to verify that the system is clean prior to the analysis of samples or reference materials.
7.1.1 Check the negative or blank controls for the presence of drugs, drug metabolites, or interfering chromatographic peaks. If any are present, determine or evaluate the effect on the batch results."

Reason for Deviation: Dangerous drugs were identified in the blanks after the samples. An alternative method extending the hold time, DrugTest50-FHTI, was developed and used to identify the late-eluting drugs.

Laboratory Number(s) (if applicable): BCCL-21-1917, BCCL-20-1408

Applicable Analyst _____ Date 05/18/22

Applicable Analyst _____ Date 5/18/22

Quality Assurance Manager _____ Date 18 May 2022

Laboratory Director _____ Date 18 May 2022