Northern States Power Company

414 Nicollet Mall
Minneapolis, Minnesota 55401-1927
Telephone (612) 330-5500

February 10, 1994

10 CFR part 26
Appendix A
Section 2.8.e.4

U S Nuclear Regulatory Commission
Attn: Document Control Desk
Washington, DC 20555

MONTICELLO NUCLEAR GENERATING PLANT
Docket No. 50-263 License No. DPR-22

PRAIRIE ISLAND NUCLEAR GENERATING PLANT
Docket No. 50-282 License DPR-42
50-306 DPR-60

Unsatisfactory Blind Performance Test

On January 11, 1994 a blind performance test discrepancy was discovered by an NSP FFD collector and subsequently reported to the NSP Medical Review Officer (MRO). According to the blind specimen supplier , El Sohly Laboratories, the blind specimen in question (Chain of Custody # 996202) was spiked positive for opiates , with a known value of 596 ng/ml (Codeine) and 617 ng/ml (morphine). Initial testing of the sample by MEDTOX confirmed the presence of opiates, however concentration levels were detected and recorded at approximately double the known values. NSP's MRO uses a deviation rate of 15% for acceptance.

In accordance with 10 CFR part 26 , Appendix A, Section 2.8.e.4, NSP completed an investigation into the unsatisfactory blind test result and identified corrective actions to prevent reoccurrence. (See attached Nuclear Quality Department (NQD) report of investigation dated February 2, 1994)

Results of NSP's investigation indicate that a procedural error in the dilution process was the cause for the testing discrepancy. MEDTOX failed to dilute the sample by a factor of (2) as was annotated on the GC/MS confirmation cover sheet . A MEDTOX analyst believing that the sample had been diluted by two , calculated the test results by doubling them , thus accounting for the 100% discrepancy.

A027
1/1

As further required by 10 CFR 26 Appendix A, Section 2.8.e.4, attached is a record, signed and dated by the individuals responsible for day-to-day management and operation of MEDTOX, identifying investigation findings and corrective actions to prevent recurrence.

This letter contains no NRC commitments.

Please contact Mel Opstad (612-337-2038) if you have any questions related to this matter.

Mal

Roger O. Anderson
Director, Licensing & Management Issues

C: Regional Administrator, Region III
NRR Monticello Project Manager, NRC
Monticello Sr Resident Inspector, NRC
NRR Prairie Island Project Manager, NRC
Prairie Island Sr Resident Inspector, NRC
Jay E. Silberg

Attachments: MEDTOX Investigation of Specimen # E2037, Signed & Dated 01-31-94
NSP Nuclear Quality Department Investigative Report, Signed & Dated 02-03-94

NSP Distrbution :

Doug Antony
George Miserindino
Randy Cleveland
Craig Johnson
Brian Anderson
Frank Evitch
Dr. James Woodburn

MEDTOX LABORATORIES

402 West County Road D • Saint Paul, Minnesota 55112 • 612-636-7466

MEDTOX INVESTIGATION OF SPECIMEN

(#E203704)

This document relates to the investigation of a blind specimen submitted by Northern States Power to MEDTOX Laboratories on 1/10/94. The specimen was identified with the social security number 530-30-8445 and chain of custody number 996202. This specimen was assigned the MEDTOX accession number E203704. The sample was positive for opiates by immunoassay and confirmed by GC/MS with codeine = 1244 ng/ml and morphine = 1322 ng/ml. The results were reported on 1/12/94. NSP requested that the specimen be sent to Smith Kline Laboratories in Chicago for an opiate retest. NSP requested MEDTOX to retest the specimen on 1/27/94. The retest was still positive for codeine and morphine. The codeine = 656 ng/ml and morphine = 638 ng/ml were approximately one-half of the original values reported.

The calibrators and controls were evaluated for both GC/MS assays and found to be acceptable. All three controls on both assays were well within their reference ranges. In both assays the specimens were diluted by 2 due to the high immunoassay value (greater than 125). The dilution is performed in order to accommodate the high codeine and morphine specimens encountered so that less specimens need to be repeated and turn around time decreased.

The first specimen set up on 1/11/94 by the analyst was not diluted in half before the assay was run but was multiplied by two thus accounting for the two-fold difference in results.

The following corrective action has been instituted. The GC/MS confirmation cover sheet has been modified to more effectively distinguish which specimens are to be diluted. This problem has been discussed with the analyst and the proper procedure for insuring that the diluted samples are handled correctly reinforced. The Forensic Supervisor, observed the analyst perform a GC/MS confirmation set-up with dilution protocol to insure that proper procedures are being followed. Documentation will be put into the individuals personnel file. In addition, the proper dilution handling procedure has been discussed with the GC/MS staff and the importance of the proper handling reinforced.

1-31-94

Gary Blomfield PhD

Internal Correspondence

Date **February 2, 1994**

From **LE Brehm, Sr QA Engineer** Location **RSQ 08**

To **RD Cleveland, Personnel Secur Team Leader** Location **RSQ 06**

Subject **Review of Blind Sample, chain of custody #996202**

On January 27, 1994, you requested from the Supervisor Quality Assurance Audits that an investigation be conducted and documented with regards to the error of a blind sample sent by NSP to MEDTOX for analysis on January 10, 1994. The purpose of this memo is to document the results of the review of the MEDTOX investigation by NQD of that blind sample.

The sample was provided to NSP by El Sohly Laboratories, Inc., Batch 0-0319, control number 530-30-8445, and submitted under the chain of custody number 996202. The known value for the specimen was codeine = 596 ng/ml and morphine = 617 ng/ml. The sample was confirmed positive for opiates at MEDTOX on January 11, 1994 by GC/MS with codeine = 1244 ng/ml and morphine = 1322 ng/ml. This is an approximate error of 100%. The MRO at NSP uses an error of less than 15% for acceptance.

Upon notification of the discrepancy, the MRO requested on January 18, 1994 that the sample be sen to SmithKline Beecham Laboratories in Chicago. These results were codeine = 624 ng/ml and morphine = 720 ng/ml, an approximate error of 5% for codeine and 15% for morphine. The MRO then requested that MEDTOX retest the sample. These results were codeine = 656 ng/ml and morphine = 638 ng/ml, an approximate error of 10% and 3%, respectively.

Laboratory	Codeine (ng/ml)	Morphine (ng/ml)
El Sohly	596	617
MEDTOX (initial)	1244	1322
SmithKline	624	720
MEDTOX (retest)	656	638

On January 31, 1994, I met with Dr. Gary Hemphill and Ms. Jennifer Collins of MEDTOX to discuss the cause of the above discrepancy. MEDTOX found upon investigation that this was a dilution error. Typically, when the initial immunoassay indicates a level greater than 125 ng/ml, the GC/MS Confirmation Cover Sheet is annotated to dilute the sample by two. This reduces turn around time due to reducing the need to rerun samples because of instrument limitations. In this case, the sample was not diluted by two as was annotated on the GC/MS Confirmation Cover Sheet, but when calculating the result, analyst assumed that the sample had been diluted as indicated and multiplied the results by two, thus accounting for the 100% discrepancy. It appears that the sample was not diluted due to an error by the analyst, who is a relatively new employee (3-4 months). The GC/MS Confirmation Cover Sheet used to direct the dilutions is

confusing. The need for dilution, altnough identified, is not readily distinguishable. This type of error has not occurred prior to this at MEDTOX. It should be noted that NSP had no other specimens in the original batch of ten specimens (011194A). It should also be noted that the possibility for a false positive to have occurred due to this type of error is very small. The initial immunoassay screens the specimen with no dilutions such that those below the cutoff level are not confirmed using GC/MS.

In order to prevent recurrence, MEDTOX revised the GC/MS Confirmation Cover Sheet to better identify those specimens that are to be diluted. In addition, the Forensic Supervisor observed the analyst responsible for this error perform a GC/MS setup with dilution to verify proper performance. No performance problems were found. During the setup process, the analyst physically moves the sample in the rack after the dilution. The analyst was counseled regarding the procedure for insuring that the diluted samples are properly handled and a letter describing this incident was placed in his file. The proper dilution handling procedure was discussed with the staff and the importance reinforced.

I conclude that no false positives should have resulted due to this error and that MEDTOX has implemented corrective action to prevent recurrence. This error does not reduce the effectiveness of NSP's Fitness for Duty Program.

Any questions, I can be reached at 337-2204.

Sincerely,

Lau Brehm

LE Brehm
Sr QA Engineer

cc: Dr. J Woodburn
Dr. DG Hemphill (MEDTOX)
TE Amundson