North Atlantic

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The Northeast Utilities System

Ted C. Feigenbaum
Senior Vice President &
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NYN- 94066

June 16, 1994

United States Nuclear Regulatory Commission
Washington, DC 20558

Attention: Document Control Desk

Reference: Facility Operating License No. NPF-86, Docket No. 50-443

Subject: False Negative Blind Tests

Dear Mr. Bush:

North Atlantic Energy Service Corporation (North Atlantic) has enclosed two reports entitled "Investigation Report on Blind Performance Test Samples." The enclosed reports, prepared by Duo Research, Inc., address three false negative blind tests which were recently experienced. These tests do not constitute unsatisfactory performance on the part of SmithKline Beecham Clinical Laboratories under the HHS guidelines according to Dr. Robert E. Willette of Duo Research, Inc.

It is our understanding that you wish to be informed of all false negative tests, regardless of whether or not they constitute unsatisfactory performance.

If you have any questions, or wish any additional information, please feel free to contact Mr. Bruce Seymour, Security Manager at 603-474-9521, extension 4015.

Very truly yours,

Ted C. Feigenbaum

TCF:ALL/act

Enclosures

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cc: Mr. Thomas T. Martin
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United States Nuclear Regulatory Commission
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Mr. Albert W. De Agazio, Sr. Project Manager
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Mr. Loren L. Bush
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North Atlantic
June 16, 1994

ENCLOSURE TO NYN-94066

INVESTIGATION REPORT
on
Blind Performance Test Samples

Objective:

The licensee shall investigate any unsatisfactory performance testing result. A record shall be made of the investigative findings and the corrective action taken by the laboratory. The licensee shall send the document to the NRC as a report of the unsatisfactory performance testing incident within 30 days.

References:

1. 10 CFR 26 Appendix A, Subpart 2.8(e)(4)
2. FR 53, 11970 (1988), Subparts 3.19(b)(2) & (5)

Observation:

A blind quality control sample containing a combination of morphine, morphine-3-glucuronide, codeine and 6-acetylmorphine was reported negative by the SmithKline Beecham Clinical Laboratories - Norristown. An unannounced laboratory site visit was conducted by Dr. Robert E. Willette on May 25, 1994.

Findings:

The sample was prepared for submission to the laboratory by the Seabrook site on March 23, 1994. It was received by the laboratory on March 24, 1994, and subjected to the initial immunoassay test on March 25th. The sample gave a positive response in the Emit opiate assay, with a value that was 1.585 times the cutoff calibrator (300 ng/mL of morphine) value. The sample was then submitted to the GC/MS confirmation assay on March 26th. The sample was analyzed for codeine and morphine, obtaining concentrations of 129 and 293 ng/mL, respectively. Although there was a problem noted with one or more control samples by the analyst, the final reviewer determined that this did not constitute sufficient cause to repeat the analysis. Since the morphine result was below the cutoff of 300 ng/mL, the sample was reported as negative.

An identical sample was submitted by the Seabrook site, which was received by the laboratory on May 13th, and another identical sample was also received on May 11th by the laboratory, which was submitted by a different client under DOT testing rules. Both samples gave similar screening values, 1.585 and 1.549, respectively. The May 11th DOT sample gave analytical results of 101 ng/mL of codeine and 240 ng/mL of morphine, and was reported as negative. The May 13th sample submitted by Seabrook produced results of 109 and 331 ng/mL, respectively. As this result was above the cutoff, it was reported as a positive for morphine.

The average reference values obtained on this sample were 112 ng/mL for codeine, 522 ng/mL for total morphine (119 free morphine plus ca. 500 as the glucuronide), and 61 ng/mL of 6-acetylmorphine.

These results are in contrast to a similar sample (from a previous lot) that was analyzed by the laboratory on February 5, 1994, in which the GC/MS results were 109 ng/mL of codeine and 615 ng/mL of morphine. Average reference values for this sample were 109 ng/mL of codeine and 597 ng/mL total morphine.

Findings (continued):

The significant drop in total morphine from the earlier sample, together with the variable results obtained with the three latter samples, suggests incomplete hydrolysis and recovery of morphine from it natural glucuronide conjugate. However, the laboratory includes high and low control samples that contain a 50:50 mixture of free and conjugated morphine to monitor the hydrolysis step. In each of the GC/MS runs reviewed, the recovery of total morphine from these control samples was within the expected limits. This does not completely rule out the possibility of incomplete hydrolysis of the sample reported as negative, but the low recovery of morphine in three identical samples tested on three separate occasions suggests possible other variables.

Recommendation:

Although this is an isolated false negative report for morphine, it should be investigated further as two subsequent samples also were found to have quantitative results significantly below the average reference value.

Because these samples screened positive and were submitted to confirmation, the laboratory has retained them in frozen storage, which is standard practice for the laboratory. Therefore, it is recommended that the laboratory reanalyze the March 25th and May 13th samples and submit an appropriate aliquot to an outside reference laboratory for independent analysis. It is suggested that the aliquots be sent to ElSohly Laboratories, Inc., an HHS-certified laboratory that served as one of the reference laboratories for this particular sample lot.

The two samples are identified by the SPCL accession numbers 829182A and 878555A, received by the laboratory on 3/24/94 and 5/13/94, respectively.

Prepared for: North Atlantic Energy Service Corporation

by: Robert E. Willette Date: May 27, 1994

Duo Research Inc.

INVESTIGATION REPORT
on
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References:

1. 10 CFR 26 Appendix A, Subpart 2.8(e)(4)
2. FR 53, 11970 (1988), Subparts 3.19(b)(2) & (5)

Observation:

Two blind quality control samples containing a combination of THC-9-carboxylic acid (reference value 27 ng/mL) and another cannabinoid, in sufficient concentration to produce a positive screening result, were reported negative by the SmithKline Beecham Clinical Laboratories - Norristown. An unannounced laboratory site visit was conducted by Dr. Robert E. Willette on May 25, 1994.

Findings:

These two samples, which are designed to challenge the laboratory near the confirmation cutoff of 15 ng/mL, were submitted by the Seabrook site and received by the laboratory on April 28 and May 13, 1994. Both produced positive initial test results, giving values in the Emit cannabinoid assay that were 1.163 and 1.176 times the 100 ng/mL cutoff calibrator, respectively, and 3.519 and 3.180 times the 50 ng/mL cutoff calibrator, respectively. Both were submitted to GC/MS analysis. Although both samples gave quantitative values that were close to the reference value, the analyses were repeated with diluted aliquots because an interfering peak was observed in one of the ion scans, causing it to fail the ion ratio qualifying criteria. Similar results were obtained in the repeat analyses, so both samples were reported as negative, which is in accordance with certification requirements.

It is noted that the cannabinoid confirmation procedure utilized by the laboratory is a standard procedure used in all SBCL certified laboratories. This investigator has observed similar results with retests conducted in another SBCL facility, that is, interference in the 488 ion. Ms. Susan Mills, the laboratory responsible person, indicated that the laboratory had missed a cannabinoid in the most recent National laboratory Certification Program (the HHS-sponsored program) maintenance PT survey. It appears that the assay procedure does not provide either sufficient "clean up" during the liquid-liquid extraction step or separation during the chromatography.

Is also noted that a nearly identical sample containing the combination described above has been submitted to the laboratory on several occasions during 1993 and has never been reported as negative. In fact, the samples from the earlier lot had a slightly lower concentration of THC-9-carboxylic acid (reference average 22 ng/mL). Also, the laboratory has correctly identified samples containing the THC metabolite at concentrations just above the 100 ng/mL cutoff level.

Findings (continued):

It should also be noted that identical samples as discussed above have been reported correctly by several other laboratories to which they have been submitted as blind QC samples.

It appears that there may have been some change in the assay procedure that does not provide sufficient elimination of interferences at concentrations below 30 ng/mL.

Recommendation:

These two results, in and of themselves, do not constitute unsatisfactory experience under the prevailing regulations. However, the consecutive misses on these blind QC samples, together with reported miss(es) on NLCP PT surveys, support the observation that the current confirmation assay may require modification. It is recommended that the laboratory be requested to re-evaluate its assay procedure for possible changes to improve it performance at lower concentrations of the THC metabolite. Also, the laboratory should be requested to provide information about the NLCP maintenance PT survey results mentioned above.

Prepared for: North Atlantic Energy Service Corporation

by: Robert E. Willette Date: May 27, 1994

Duo Research Inc.