Commonwealth Edison Company
1400 Opus Place
Downers Grove, IL 60515

December 20, 1995

ComEd

U.S. Nuclear Regulatory Commission
Attn: Document Control Desk
Washington, D.C. 20555

Subject: LaSalle County Nuclear Power Station Units 1 and 2
Response to October 25, 1995 NRC Request For Additional Information On LaSalle Unit 1 RPV Surveillance Material Testing and Analysis, Reactor Vessel Material Surveillance Program - Appendix H
NRC Docket Nos. 50-373 and 50-374

References:

See Attached

In accordance with Appendix H of 10 CFR 50, ComEd provided the original LaSalle Unit 1 RPV Surveillance Materials Testing and Analysis, March 1995 submittal (Reference 1). Due to an error which was discovered in the surveillance specimen chemical analyses, ComEd provided the Revision 1 LaSalle Unit 1 RPV Surveillance Materials Testing and Analysis, June 1995 submittal (Reference 2). In Reference 3, the NRC Requested Additional Information (RAI) on this matter. The following is ComEd's response to the Staff's Request For Additional Information (RAI). The ComEd response retains the numbering sequence of the Staff's RAI, although Question 1. has been reformatted for clarity.

1. **Provide a chronology of events [see A. below] including a description of the following items:**

   Why was the surveillance material retested? [see B. below]

   What was the root cause of the error in the previously reported data? [see C. below]

   What corrective action has been implemented to assure the error is not repeated? [see D. below]

   A.

   A chronology of significant events follows:

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- Surveillance Specimen Capsule pulled from LaSalle County Station Unit 1, March 25, 1994.

- Prepared Specimen Capsule shipped from LaSalle to General Electric (GE) Vallecitos Nuclear Center (VNC) on July 29, 1994.

- Capsule received at VNC on or about August 5, 1994.

- Initial test results were received by ComEd from GE on March 9, 1995.

- Initial Surveillance Specimen Capsule report docketed with NRC on March 23, 1995 (Reference 1).

- On April 11, 1995 discussions were held with ABB/Combustion Engineering (ABB/CE), GE, Nuclear Energy Institute, and Wisconsin Public Service's Kewaunee Station lead ComEd to believe that a problem potentially exists with the initial GE chemistry results.

- ComEd verbally requests that GE review the initial chemistry results for possible errors on April 12, 1995.

- GE determines that a systematic but conservative error was made on initial chemistry analysis and verbally informs ComEd on or about April 20, 1995. GE subsequently documented this in a letter (Reference 4, attached) to ComEd on April 25, 1995, which also addressed root cause and corrective action.

- Problem Identification Form (PIF) 373-200-95-00877 initiated at LaSalle April 21, 1995.

- LaSalle Nuclear Licensing Administrator verbally informs LaSalle NRC Project Manager of the error on May 1, 1995.

- Portions of the specimens (five each of weld and plate Charpy halves) are shipped from VNC and received at Argonne National Laboratory (ANL), May 4, 1995

- On May 23, 1995 ComEd requests additional information from GE on corrective actions implemented by GE (Reference 5, attached).

- ANL check chemistry results received by ComEd on or about May 31, 1995.

- ComEd reviews results of ANL "check" chemistry, and the revised GE chemistry, with ABB/CE on or about June 2, 1995.
- Revised Surveillance Specimen Capsule report with corrected GE chemistry results docketed with NARC on June 21, 1995 (Reference 2).
- ComEd receives additional information on GE corrective actions on September 26, 1995 (Reference 6, attached).
- On September 28, 1995 ComEd verbally requests additional information from GE on selection of standard reference materials.
- PIF closed at LaSalle, October 27, 1995.
- ComEd receives additional information from GE on selection of standard reference materials for chemical analysis on October 30, 1995 (Reference 7, attached).
- RAI (Reference 3) received at LaSalle, November 1, 1995.

B.

The initial GE chemistry values were shared with ABB/Combustion Engineering, NEI, and Kewaunee Nuclear Power Plant. Discussion between ComEd and these parties lead ComEd to request that GE review the initial testing and verify that the results were accurate. In the process of this review, GE discovered that an error had indeed been made during the initial chemistry testing. This lead ComEd to request that the specimens be retested.

C.

The root cause of the error in the previously reported data was that incorrect dilution factors for the actual metal specimens being tested were entered into the plasma spectrometer. Since the dilution factors for the reference standards being tested were entered correctly, the error was not detected during the testing, or during review of the test results. It was therefore concluded that this error was caused by inadequate process controls in the form of lack of independent verifications on the part of GE personnel involved in the original testing and analysis of the LASalle Unit 1 RPV surveillance specimens.

It should also be noted that a predisposition existed on the part of both GE and ComEd subject matter experts to accept the variable chemistry results due to prior knowledge of industry experience with chemistry variability associated with the particular ABB/CE weld metal involved (1P3571).

D.

The following corrective actions were taken to verify that the chemistry results provided in the revised report are correct:

- The corrected GE chemistry results were independently confirmed by ANL.
- ABB/Combustion Engineering reviewed the corrected GE results and the ANL "check" results and agreed with the results for the ABB/CE 1P3571 weld metal.

The following corrective actions are in place to assure that the error is not repeated:

- In the future GE will independently verify dilution factor calculations.
- In the future GE will independently verify spectrometer inputs.
- In the future GE will independently verify final chemistry results with spectrometer output.
- In the future ComEd will pursue re-analysis when chemistry data is unusual in any respect, and will apply GE lessons learned to any future vendor supplied surveillance reports.

General Electric is currently testing a Surveillance Specimen Capsule removed from LaSalle Unit 2 on March 1, 1995. It is ComEd's belief that the aforementioned corrective actions taken by GE and ComEd will prevent recurrence of a similar error in this ongoing testing.

2. Provide a list of surveillance capsules and materials previously tested by General Electric. Are the test results from these surveillance capsules correct? Explain the basis for this conclusion.

See Reference 8 (attached) for the answer to this question. ComEd has reviewed and concurs with the bases and conclusions of Reference 8.

3. Describe the procedures and actions taken by the licensee and its vendor, General Electric, in meeting the requirement of 10 CFR Part 21, "Reporting of Defects and Noncompliance."

As detailed in the above chronology, once it had been confirmed that an error existed in the initial chemistry results, a Problem Identification Form (PIF) was initiated at LSCS in accordance with administrative procedure LAP-1500-8TB. This document served to track investigation of the root cause of the error, its resolution, and corrective actions. In this case, because the analysis of the RPV specimens was completed under GE's Part 21, Safety Related, Quality Assurance Program, ComEd requested that GE evaluate the Part 21 applicability of this error. The results of GE's evaluation (Reference 8, attached) indicate that Part 21 is not applicable in this instance, and that no concern exists with the possibility of past errors in chemistry results. ComEd concurs with GE's evaluation.

4. **Provide the NRC with sample surveillance weld material to confirm the licensees test results. Since this will include irradiated material, packaging and transportation of the material will probably require special handling. The licensee is requested to contact the NRC project manager to arrange for the time and place of delivery of the irradiated surveillance specimens to the NRC contractor.**

Although ComEd understands the concerns of the Staff in this matter, it is our belief that the shipment of the irradiated specimens to the NRC's contractor, Oak Ridge National Laboratory (ORNL), is unnecessary. This belief is based on the fact that ANL has already completed an independent chemical analysis of the LaSalle Unit 1 RPV specimens. See attached Reference 9 for a description of the testing performed and the results obtained. ComEd initiated this independent analysis as a part of the corrective actions described above. The results of the ANL analysis corroborate the revised GE chemical analyses, as shown in the following table:

SAMPLE #	GE CAPSULE REPORT		ARGONNE CHECK ANALYSIS	
PLATE SAMPLES	**% Cu**	**% Ni**	**% Cu**	**% Ni**
417	.14	.56	.14	.47
433	.12	.49	.14	.57
435	.11	.50	.18	.60
4JC	.13	.50	.14	.54
4J6	.15	.58	.15	.56
AVERAGE =	.13	.53	.15	.55
WELD SAMPLES				
443	.20	.75	.19	.69
44A	.20	.76	.18	.70
44F	.22	.83	.19	.71
44M	.22	.73	.18	.64
45E	.23	82	.18	.69
45E (ANL DUPLICATE)	N/A	N/A	.18	.64
AVERAGE =	.21	.78	.18	.68

The ANL check analysis was performed under a Quality Assurance Program which is structured in accordance with DOE 5700.6C, incorporates the requirements of ASME NQA-1, and satisfies 10CFR50 Appendix B. It is therefore highly unlikely that any further error, or significant inconsistency has gone undetected. ComEd further believes that the risk and costs associated with the handling and shipping to ORNL of these irradiated materials in order to verify the results of ANL is not compensated by an increase in the margin of safety to the public. It is also ComEd's desire to retain the tested ABB/CE 1P3571 weld metal specimens in long term storage for possible use at some future time (e.g., to be reconstituted for reannealing and reirradiation studies). This is in keeping with the guidance provided by the Staff in Information Notice 90-52: "Retention of Broken Charpy Specimens."

In conclusion, ComEd understands and appreciates the NRC's concern over the error in the chemistry results for the LaSalle Unit 1 RPV specimen capsule. ComEd also believes that, as the licensee, we have already taken all appropriate actions necessary to document, evaluate, correct, report, and prevent recurrence of this unfortunate error, including independent assessment of specimen chemistries by ANL.

Please direct any questions you may have concerning this matter to this office.

Very truly yours,

Gary G Benes

Gary G. Benes
Nuclear Licensing Administrator

Attachments

cc: H. J. Miller, Regional Administrator - RIII
P. G. Brochman, Senior Resident Inspector - LSCS
M. D. Lynch, Project Manager - NRR
Office of Nuclear Facility Safety - IDNS

REFERENCES

1. Letter from Mr. G. G. Benes (ComEd) to the USNRC, dated March 23, 1995.
2. Letter from Mr. G. G. Benes (ComEd) to the USNRC, dated June 21, 1995.
3. Letter from Mr. R. M. Latta (USNRC) to Mr. D. L. Farrar (ComEd) Request for Additional Information (RAI), dated October 25, 1995.
4. Letter from Mr. T. A. Caine (GE) to Mr. T. D. Spry (ComEd), dated April 25, 1995 (attached).
5. Letter from Mr. T. D. Spry (ComEd), to Mr. T. A. Caine (GE), dated May 23, 1995, Chron. # 214035 (attached).
6. Letter from Mr. T. A. Caine (GE), to Mr. T. D. Spry (ComEd), dated September 26, 1995 (attached).
7. Letter from Ms. E. W. Sleight (GE), to Mr. T. D. Spry (ComEd), dated October 30, 1995 (attached).
8. Letter from Mr. T. A. Caine (GE), to Mr. T. D. Spry (ComEd), dated December 15, 1995 (attached).
9. Letter from Mr. D. G. Graczyk (ANL), to Mr. J. M. Chynoweth (ComEd), dated November 22, 1995 (attached).

REFERENCE 4

GE Nuclear Energy

General Electric Company
175 Curtner Avenue, San Jose, CA 95125

April 25, 1995

Mr. Tom Spry
Commonwealth Edison Company
1400 Opus Place, Suite 300
Downers Grove, IL 60515

Subject: **Re-evaluation of LaSalle 1 Surveillance Specimen Chemistry Tests**

Reference: [1] Carey, RG, "LaSalle Unit 1 RPV Surveillance Materials Testing and Analysis," GE-NE-523-A166-1294, March 1995.

The NRC has recently been scrutinizing data on weld IP3571, the same weld as is in the LaSalle 1 surveillance program, so you asked us to double check the information we have on the surveillance weld to make sure there were no "surprises". At my suggestion, Vallecitos double-checked the chemistry test results, and found a systematic error which caused the chemistry values reported in Table 3-3 of [1] to be generally high, and therefore inaccurate and overly conservative. The cause of the error, its correction and the corrected results are provided below.

Root Cause

As described in Section 3.2.3 of the surveillance report, the chemistry test is done with a plasma spectrometer, which determines the concentration of selected elements in a solution. The solution for the test is made by dissolving about a one gram piece of a specimen in nitric and hydrochloric acid and then diluting in water so that a dilution factor of about 625 is achieved. This means that the concentration of a given element, like Cu, in the solution is about 1/625th what it would be in the metal specimen. This dilution factor range has been determined in the past to provide element concentrations in solution with optimum spectrometer detectability for low alloy steels. In addition to testing the surveillance specimens, a set of five NIST reference materials of known composition are tested using the same procedure.

In order for the spectrometer to print out the right metal weight percents, the dilution factor for each specimen is input to the computer that interfaces with the spectrometer. When the dilution factors for the surveillance specimens were input to the spectrometer for evaluation of Mn, Ni, Cu, Mo and Cr, all of which are done at one time, incorrect values were input. The same error was not made for the reference materials, so the error was not caught during the testing or during review of the test results. Furthermore, Vallecitos did not have any input on expected chemistry results from San Jose with which to compare their results. The P evaluation, which is done separately, was done correctly, so those values are not re-evaluated.

When the chemistry testing report was received in San Jose, it was reviewed during incorporation into the surveillance report, and the presence of some unusual values was noted. However, no detailed verification of the calculations which were part of the test was performed at either Vallecitos or San Jose.

Corrective Action

A meeting was held between the San Jose personnel responsible for the surveillance report and the Vallecitos personnel responsible for the chemistry testing. The purpose was to review the testing process to find steps where errors could occur and where detailed verification would be useful in catching operator errors. The Vallecitos personnel explained the process of identifying, weighing, dissolving and diluting the samples for the spectrometer evaluation. There is a good process in place, which is adequately checked by preparing the reference materials in the same way. On process improvements, the following conclusions were reached:

- The calculations involved in determining the dilution factors should be verified.
- The inputs to the spectrometer should be verified, namely specimen identification and dilution factor.
- The chemistry values in the final report should be verified against the outputs from the spectrometer.

Results

The corrective steps above were taken, using the raw data from the previous tests, such as sample weights and spectrometer readings. The results are shown in Table 1. The results for the six elements evaluated are reported for every specimen, except for P, which is shown for the same six specimens as in [1].

Comparison with CMTR data in [1] and a recently discovered letter from CE, attached, shows that the revised chemistries are consistent:

Source of Chemistry Data	%Mn	%Ni	%Cu	%Mo	%Cr	%P
Plate:						
Surveillance Specimen Sample Average	**1.29**	**0.53**	**0.13**	**0.50**	**0.256**	**0.015**
Vessel Plate CMTR [1]	1.24	0.49	0.15	0.46	-	0.011
CE Letter on Surveillance Welded Plate	1.26	0.55	0.14	0.55	0.27	0.011
Weld:						
Surveillance Specimen Sample Average	**1.50**	**0.77**	**0.21**	**0.51**	**0.078**	**0.016**
CE Letter on Surveillance Welded Plate	1.38	0.78	0.21	0.55	0.07	0.015

As we discussed, we will ship five each of the plate and weld Charpy halves to Argonne for confirmatory testing. The shipment, which is expected to be in a Type A cardboard box by Federal Express, will be ready to leave Vallecitos by May 2. We will need the shipping address for Argonne.

Once all issues are resolved (Argonne data, appropriate CF, etc.), and you give us final instructions for revision of the report [1], it should take about a week to revise and issue.

Weld Metal Chemistry Factor

The IP3571 weld metal chemistry factor (CF) reported in Appendix B of [1] is 261. There are several possible ways to recalculate the CF, as shown below. The CF documented in Revision 1 of [1] will be determined by GE and ComEd later.

Specimens Only	0.21% Cu, 0.77% Ni	CF = 192
Average of Specimen and Letter Data	0.21% Cu, 0.775% Ni	CF = 193
Average of Specimen, Letter and Weld Qual. Data (0.37% Cu, 0.75% Ni)	0.26% Cu, 0.77% Ni	CF = 207

As part of the report revision, an appropriate CF will be documented for the surveillance plate material as well.

This letter, and the supporting analyses, will be added to the surveillance capsule test design record file, DRF B13-01746. Let me know if there is anything else you need to resolve this issue with the NRC. I am disappointed that this happened, and I will be personally involved in the LaSalle 2 work to make sure, as best I can, that it is right the first time.

Regards,

Tom Caine

TA Caine, Principal Engineer
Reactor Internals Management Projects
(408) 925-4047, Fax -4175

Table 1. LaSalle 1 Surveillance Sample Chemical Analysis

Metal Sample ID	Metal Sample Type	Mn (wt%)	Ni (wt%)	Cu (wt%)	Mo (wt%)	Cr (wt%)	P (wt%)	
411	Base	1.41	0.56	0.15	0.53	0.278		
4J1	Base	1.24	0.51	0.13	0.48	0.253		
436	Base	1.36	0.57	0.15	0.54	0.287		
437	Base	1.40	0.57	0.15	0.55	0.278		
4J5	Base	1.20	0.50	0.13	0.45	0.228		
4JC	Base	1.13	0.50	0.13	0.44	0.224		
4J6	Base	1.42	0.58	0.15	0.55	0.282		
43B	Base	1.35	0.56	0.14	0.54	0.273		
413	Base	1.24	0.51	0.13	0.48	0.247		
417	Base	1.28	0.56	0.14	0.54	0.283	0.015	
433	Base	1.21	0.49	0.12	0.46	0.239	0.015	
435	Base	1.26	0.50	0.11	0.44	0.203	0.016	
		1.29	0.53	0.13	0.50	0.256	0.015	Data Avg.
		0.09	0.04	0.01	0.05	0.028	0.001	Std. Dev.
444	Weld	1.47	0.75	0.22	0.49	0.077		
44M	Weld	1.41	0.73	0.22	0.48	0.073		
45K	Weld	1.62	0.80	0.21	0.52	0.080		
45M	Weld	1.57	0.80	0.21	0.53	0.081		
45D	Weld	1.55	0.80	0.22	0.54	0.079		
447	Weld	1.47	0.79	0.22	0.53	0.082		
44F	Weld	1.60	0.83	0.22	0.55	0.082		
44U	Weld	1.44	0.73	0.20	0.48	0.072		
4LD	Weld	1.50	0.74	0.20	0.49	0.073		
443	Weld	1.47	0.75	0.20	0.47	0.081	0.017	
44A	Weld	1.41	0.76	0.20	0.52	0.079	0.016	
45E	Weld	1.47	0.82	0.23	0.55	0.082	0.014	
		1.50	0.77	0.21	0.51	0.078	0.016	Data Avg.
		0.07	0.04	0.01	0.03	0.004	0.002	Std. Dev.

911 W. MAIN CHATTANOOGA, TENN. 37401

COMBUSTION DIVISION

CHATTANOOGA WORKS

December 14, 1971

General Electric Company
Atomic Power Equipment Department
175 Curtner Avenue
San Jose, California 95125

Attention: Mr. J. Pricolo, Jr.
Subject: Surveillance Test Program
Your Ref: G.E. P.O. #205-H0401
Our Ref: C.E. Contract #2867

Gentlemen:

Attached is a copy of Table I which shows the chemistry of the plate material and deposited weld metal as required by Item 2 of C.E. Purchase Order #205-H0401, Revision No. 15, dated 11/11/71. Samples for analysis of both plates and the weld joining the plates were obtained at the 1/4T level.

Very truly yours,

COMBUSTION ENGINEERING, INC.

D. A. Howard

D. A. Howard
Nuclear Components Department

DAH/sw

Enc.

cc: Mr. J. Pyle
Mr. H. C. Reichard
Mr. J. N. Morgan

TABLE I

plt. Mat'l ↓

Lab No. Code No.	P-12858 G-5604-1	P-12859 G-5604-2	D-1134 Weld
Si	.18	.16	.19
Sul	.018	.019	.009
Phos	.011	.009	.015
Mn	1.26	1.28	1.38
Car	.16	.20	.12
Cr	.27	.13	.07
Ni	.55	.53	.78
Mo	.55	.55	.55
Cu	.14	.12	.21
(T) Al	.022	.029	.004
N2	.006	.007	.012
V	.005	.005	.007
Co	.013	.011	.016
Fe	96.46	96.61	96.28

Commonwealth Edison
1400 Opus Place
Downers Grove, Illinois 60515

REFERENCE 5

214035

May 23, 1995

Thomas A. Caine
Reactor Internals Management Projects
General Electric Company
175 Curtner Avenue
San Jose CA 95125

Subject: T. A. Caine to T.D. Spry, "Re-evaluation of LaSalle 1 Surveillance Specimen Chemistry Tests," 4-25-95

Jim Chynoweth and I have reviewed your letter, which was intended to explain the cause of the LaSalle 1 surveillance capsule specimen chemistry error and the corrective actions implemented by GE to prevent recurrence.

We understand your explanation of the chemical analysis process and the way in which the error occurred. But for corrective action and process improvement, we ask that you respond in the following areas:

- ☐ Describe the corrective action options considered before arriving at the final conclusions. Are the corrective actions identified truly the best solutions to the problem?
- ☐ Explain the degree of independence which the additional verifications referred to in <u>Corrective Action</u> will have from the process itself.
- ☐ Was benchmarking the GE chemical analysis process to the practices of independent laboratories considered? If so, was it pursued? If not, why not?

Additional layers of review may have the effect of "correcting" a process problem which should be designed out of the process in the first place. Benchmarking may be a way to identify an alternative to adding more reviews to the existing process. For example, Com Ed analytical chemistry personnel pointed out that simply having the raw data and dilution calculations for the test specimens as well as the NIST control specimens on the same sheet, instead of separate sheets, could be enough to flag a systematic error or inconsistency. However, analytical chemistry personnel from Argonne National Laboratory stated that their standard practice for reactor programs work is to have an independent verification of all calculations performed by a second analyst.

We now recognize that requesting a chemical reanalysis should be an integral part of the approach to dealing with chemistry data which is unusual in any respect. In retrospect, rather than take the conservative, bounding approach to data which we believed to represent weld metal variability, we should have obtained a deadline extension and had the chemical analyses redone. Reactor safety would not have been compromised with either approach, but verification by reanalysis would have eliminated any question of the identity of the surveillance specimens, and would have prevented subsequent damage to our credibility with the NRC in being forced to docket a revised version of the report.

Also, we understand that a review of the LaSalle 1 surveillance chemistry error for Part 21 applicability will be documented by GE, and that at this time it is your conclusion that the error is not Part 21 applicable.

If there are any questions, please call me at 708.663.7268 (FAX.7171)

Sincerely,

Thomas D Spry

Thomas D. Spry
Steam Generator and Reactor Vessel Projects

cc: J.C. Blomgren J.M. Chynoweth A.L. Kochis G.G. Benes
P.J. Zurawski

ALK

REFERENCE 6

GE Nuclear Energy

General Electric Company
175 Curtner Avenue San Jose, CA 95125

September 26, 1995

Mr. Tom Spry
Commonwealth Edison Company
1400 Opus Place, Suite 300
Downers Grove, IL 60515

Subject: **Closure of Issues Related to Surveillance Specimen Chemistry Testing**

References: [1] Letter dated 4/25/95, TA Caine to TD Spry, "Re-evaluation of LaSalle 1 Surveillance Specimen Chemistry Tests."

[2] Letter dated 5/23/95, TD Spry to TA Caine, same subject.

In [1], I described the cause of chemistry testing errors on the LaSalle 1 surveillance capsule specimens, and the corrective action GE has established to prevent similar occurrences in the future. In [2], you requested responses to several issues, in order to close the subject. The issues and our responses are below.

"Describe the corrective action options considered before arriving at the final conclusions. Are the corrective actions identified truly the best solutions to the problem?"

The root cause of the problem was human error in performing calculations with the raw data. Since standard reference materials (SRMs) are tested with each set of specimens, which checks both the method of obtaining raw data and performing calculations, it was agreed that the best process improvement was independent calculations from the raw data and independent review of the spectrometer results. The option of verification at Vallecitos (by test personnel) vs. at San Jose (by personnel responsible for the surveillance report) was discussed, and it was concluded that San Jose verification was preferred for several reasons:

- It would improve the surveillance report author's understanding of the chemistry testing process,
- The surveillance report author knows better the chemistry values that are expected, and
- Being less knowledgeable of the process, San Jose personnel are likely to do a more meticulous verification.

While more elaborate means of verification could be considered, they could become cost prohibitive, so overall I think the corrective action in [1] is the best solution. It is also consistent with our QA process for verification to comply with 10CFR50 Appendix B requirements for safety-related work. One enhancement to the independent nature of the testing and verification, not mentioned in [1], is our intent to retain in San Jose any advance information on the expected chemistries of the specimens, to assure that the Vallecitos testing is unbiased.

"Explain the degree of independence which the additional verifications referred to in Corrective Action will have from the process itself."

The results reported by Vallecitos, weight percent of each element, is calculated by the spectrometer, based on raw data of μg/g concentration and the input value of dilution factor. The dilution factor is calculated from several raw data values related to sample weight, sample dissolution and dilution. In the corrective process, every calculation made with raw data at Vallecitos is independently duplicated at San Jose. Thus, once Vallecitos generates the raw data, using methods verified by SRM results, the calculation steps are performed twice by independent sources and discrepancies, if any, are resolved between the two sources.

"Was benchmarking the GE chemical analysis process to the practices of independent laboratories considered? If so, was it pursued? If not, why not?"

The GE chemical analysis process has been benchmarked, in a sense, by comparing results of numerous tests of surveillance specimens with past analyses done by test labs of vessel fabricators like CE and CB&I. In addition, the SRM specimens allow the GE process to be benchmarked against an absolute chemistry. Since the error on the LaSalle 1 specimens related to calculations and not to testing processes per se, benchmarking the practices of other laboratories was not considered. We are, however, always interested in cost effective improvements, and appreciate the information provided in [2].

The 10CFR21 review of this issue is complete. GE has concluded that there is no safety concern connected with the possibility of a past error in chemistry test results. I was contacted by Barry Elliot of the NRC on this subject, and provided him an informal explanation by phone of the issue, and why it is not a safety concern. He inquired about receiving documentation on the issue, so I referred him to our person with administrative responsibility for Part 21 issues. We have received no formal request from the NRC on the subject, so I believe the informal discussions with Barry were sufficient.

If you have any questions on the responses above, please call me.

Regards,

Tom Caine

TA Caine, Principal Engineer
Reactor Internals Management Projects
(408) 925-4047, Fax -4175

cc: R Willems
BJ Branlund

REFERENCE 7

GE Nuclear Energy

Structural Mechanics Projects
175 Curtner Avenue M/C 747
San Jose, CA 95125
(408) 925-1472

EWS-954

cc: **B.J. Branlund**
T. A. Caine
P. S. Wall

October 30, 1995

Tom Spry
Commonwealth Edison
1400 Opus Place, Suite 400
Downers Grove, IL 60515

SUBJECT: Response to inquiry regarding selection of SRMs for spectrometer calibration

Reference: Conversation with Tom Caine on 9/28/95

Dear Tom,

In response to your inquiry regarding the selection of Standard Reference Materials (SRMs) I am including brief descriptions of the basis for calibration and system control. In summary, it has been determined that NIST standard solutions are used for spectrometer calibration and SRMs are used for system control.

CALIBRATION:

The DCP Spectrometer is calibrated with National Institute of Standards and Technology (NIST) standards meeting the criteria of INPO. When the spectrometer is calibrated for determination of multiple elements, standards of each element under consideration (Fe, Mn, Cu, Ni, Mo, etc.) must be used in the calibration procedure. In addition, the chemical compositions of the standards are selected to be similar to the composition expected in the test specimens such that the spectrometer results are valid for the expected concentration of a typical surveillance sample.

SYSTEM CONTROL:

The use of an appropriate SRM provides sufficient periodic monitoring of the system's measurement and performance capabilities. SRMs are issued by the NIST and are selected on the basis of the closest possible simulation of the actual test sample material. The degree of compatibility is based upon the similarity of both the matrix and level of analyte in the material. It is recommended that several SRMs be used to ensure the absence of bias and/or to identify the nature of any existing bias throughout the measurement range. In the procedure used at the Vallecitos Nuclear Center, five different SRMs are utilized.

We hope that the descriptions of calibration and system control answer your questions regarding our monitoring and calibration procedures. If you should have any further questions or concerns, please feel free to call us.

Sincerely,

E.W. S.

Ericka W. Sleight
Engineer
RPV Surveillance Program

REFERENCE 8

GE Nuclear Energy

General Electric Company
175 Curtner Avenue, San Jose, CA 95125

December 15, 1995

Mr. TD Spry
Commonwealth Edison Company
1400 Opus Place, Suite 300
Downers Grove, IL 60515

Subject: **NRC RAI on LaSalle 1 Surveillance Chemistry Testing**

Dear Tom,

Here are the responses on requests 2 and 3 of the subject RAI.

2. GE has performed testing on numerous BWR surveillance capsules since its first in 1984. As part of most testing scopes, the surveillance plate and weld chemistries of the irradiated surveillance specimens have been determined, typically by testing 2-3 pieces from different specimens of each material.

   The results of the gamma spectrometry testing by which copper and nickel content were determined are provided in Table 1. For most of those tests, CMTR (certified material test report) chemistry data are available as a check on the validity of the surveillance chemistry tests. Comparison of the GE test results and the CMTR results show agreement within expected values when considering the uncertainties:

   - The GE method has ±5% relative uncertainty,
   - The CMTR method had some uncertainty,
   - The materials themselves have some chemistry variability

   In a few cases, there is no baseline chemistry data for surveillance weld materials, either because chemistries for the heat in the as-welded condition were unknown, or because the heat number used in the weld was unknown. In these cases, there is still good confidence in the validity of the chemistry results because the surveillance plate specimens were tested at the same time by the same process and performer. These plate specimen results agree well with the plate CMTR data, which are available for comparison.

3. When the issue of chemistry testing errors on the LaSalle 1 specimens was identified, an internal process was started at GE to determine if the error created a Potential Safety Concern (PSC). Under the GE process, a PSC is evaluated to determine if a 10CFR21 reportable condition exists.

   In accordance with the GE process, an evaluation of the PSC was made. The focus of the evaluation was on the potential use of past chemistry test results for safety-related purposes. For most past analyses, the plant beltline analyses continued to be based on CMTR data alone, even after surveillance test results were available, so the test results were not used for safety-related purposes. Two instances were identified where surveillance material results were used to supplement existing beltline CMTR data.

   - In one case, Hope Creek, the chemistry test results were averaged with existing beltline weld data to provide a best estimate chemistry for one of the beltline heats. The test results had been independently verified and found to be correctly evaluated, using the new process established after the LaSalle 1 error.

   - In the other case, Hatch 1, the test results were used to establish the copper value for a beltline weld for which no value was available. The test results for elements that could be compared with the existing element values from CMTR data agreed well, providing a basis that the surveillance copper value would be representative of the beltline copper.

   Since there was no evidence of incorrect test results having been generated in either of the two cases above, GE determined that the PSC was not a reportable condition under 10CFR21.

   One aspect of the evaluation of the PSC that was not originally considered is that surveillance chemistry test results issued in GE reports might have been used by PWRs to develop best estimate chemistries for their beltline materials. Based on the validity of the past chemistry test results, as demonstrated in Table 1, and the fact that all plants with 1P3571 weld material are aware of the correct test results from LaSalle 1, GE has determined that a 10CFR21 reportable condition related to surveillance material chemistry testing does not exist.

If you have any questions or comments on the responses, please call me at the number below.

Regards,

Tom Caine

TA Caine, Principal Engineer
Reactor Internals Management
(408) 925-4047

cc: BJ Branlund, GE
D Kelly, GE

Table 1. Previous GE Surveillance Chemistry Test Results

Plant	Test Date	Plate or Weld	Material Heat No.	Chemistry Data Source	Cu Wt %	Ni Wt %	Mn Wt %	Mo Wt %	Comments
Cofrentes	1993	P	E12761	GE	0.03	0.60	1.48	0.58	
				CMTR	0.03	0.62	1.46	0.57	
		W	D60468	GE	0.07	0.76	1.70	0.50	
				CMTR	0.06	0.72	1.63	0.49	
Cooper	1993	P	C2307-2	GE	0.22	0.76	1.28	0.50	
	1987			CMTR	0.21	0.73	1.25	0.47	
		W	20291	GE	0.22	0.74	1.33	0.54	
				CMTR	-	-	-	-	CMTR data not available
Duane Arnold	1986	P	B0673-1	GE	0.15	0.70	1.35	0.62	
				CMTR	0.15	0.61	1.37	0.55	
		W	(SMAW)	GE	0.02	1.95	1.25	0.49	
				CMTR	-	-	-	-	CMTR data not available
FitzPatrick	1987	P	C3278-2	GE	0.12	0.62	1.35	0.49	
				CMTR	0.13	0.60	1.26	0.48	
		W		GE	0.31	0.72	1.45	0.50	
				CMTR	-	-	-	-	Weld heat # unknown
Hatch 1	1985	P	C4114-2	GE	0.11	0.65	1.40	0.56	
				CMTR	0.13	0.70	1.43	0.54	
		W	1P3571	GE	0.28	0.76	1.40	0.50	Heat # determined during 9201,S1 work
				Ind. avg.	0.31	0.77	1.31	0.51	Mn,Mo values from one CMTR
Hatch 2	1991	P	C8554	GE	0.08	0.63	1.39	0.60	
				CMTR	0.08	0.58	1.32	0.52	
		W	51912	GE	0.12	0.10	1.18	0.56	
				CMTR	0.16	-	1.13	0.53	
Hope Creek	1995	P	5K3238/1	GE	0.09	0.66	1.32	0.56	
				CMTR	0.09	0.62	1.42	0.54	
		W	D53040	GE	0.06	0.46	1.28	0.32	
				CMTR	0.09	0.68	1.69	0.51	
Millstone 1	1993	P	C1079-1	GE	0.22	0.49	1.32	0.47	
	1984			CMTR	0.19	0.51	1.31	0.49	
		W	34B009	GE	0.20	.59-1.09	1.29	0.55	Separate Ni wire added during welding
				CMTR	0.18	1.03	1.34	0.49	

GE = Surveillance chemistry test by GE
CMTR = Fabrication test report
N/A = Not available

ESW = Electroslag weld
SAW = Sub-arc weld

SMAW = Shielded metal arc weld

Table 1. Previous GE Surveillance Chemistry Test Results

Plant	Test Date	Plate or Weld	Material Heat No.	Chemistry Data Source	Cu Wt %	Ni Wt %	Mn Wt %	Mo Wt %	Comments
Peach Bottom 2	1991	P	C2761-2	GE	0.10	0.54	1.26	0.48	
				CMTR	0.11	0.54	1.30	0.47	
		W	(ESW)	GE	0.10	0.32	1.44	0.49	
				CMTR	-	-	-	-	Weld heat # unknown
Peach Bottom 3	1990	P	C3103-1	GE	0.13	0.63	1.46	0.51	
				CMTR	0.14	0.60	1.35	0.47	
		W	(ESW)	GE	0.11	0.40	1.56	0.50	
				CMTR	0.11	0.41	1.56	0.51	
Santa Maria De Garoña	1995	P	524720-4487	GE	0.08	0.73	0.59	0.60	
				CMTR	0.10	0.72	0.58	0.61	
		W	(SAW)	GE	0.33	0.09	1.43	0.50	
				Surv. Spec.	0.30	0.09	1.49	0.51	Data from a report provided by Nuclenor
Susquehanna 1	1993	P	C2433-1	GE	0.09	0.60	1.32	0.56	
				CMTR	0.10	0.63	1.30	0.57	
		W	411L3071, or 402K9171	GE	0.02	0.94	1.19	0.55	
				CMTR Avg.	0.03	0.96	1.18	0.52	
Susquehanna 2	1994	P	C2929-1	GE	0.12	0.63	1.32	0.58	
				CMTR	0.13	0.64	1.27	0.56	
		W	411L3071 or 401S0371	GE	0.02	0.94	1.16	0.54	
				CMTR Avg.	0.03	0.98	1.19	0.53	

GE = Surveillance chemistry test by GE
CMTR = Fabrication test report
N/A = Not available

ESW = Electroslag weld
SAW = Sub-arc weld

SMAW = Shielded metal arc weld

ARGONNE NATIONAL LABORATORY
Chemical Technology Division, Analytical Chemistry Laboratory
9700 South Cass Avenue, Argonne, Illinois 60439-4831

Telephone: (708)252-3489
Fax: (708)252-3146
E-mail: Graczyk@cmt.anl.gov

REFERENCE 9

November 22, 1995

Mr. James Chynoweth
Commonwealth Edison Co.
Systems Materials Analysis Department
555 South Joliet Road
Bolingbrook, IL 60440

Dear Mr. Chynoweth:

This letter is in response to your request for information regarding chemical analyses performed during May of 1995 by Argonne's Analytical Chemistry Laboratory (ACL) on a set of reactor surveillance capsule specimens submitted by Commonwealth Edison Co. Areas addressed here include (1) chronology of analysis; (2) description of the analytical methodology applied; and (3) summary of the ACL's Quality Assurance program. We trust this response will satisfy your needs for information related to these aspects of the analytical work performed on Commonwealth Edison's behalf. If, however, you have questions on any of the items presented, please feel free to contact me at the above phone number.

Chronology: Attachment 1 to this letter contains a tabular presentation of events that took place during the processing of the surveillance specimens at Argonne. The samples were received by our Special Materials Group (SPM) on May 4, 1995 and were transferred the same day to Argonne's Energy Technology Division where, under the direction of Dwight R. Diercks, each specimen was machined to produce chips of the steel. Portions of the chips from each specimen were subsequently analyzed to determine carbon, sulfur, and selected metal concentrations (including Cr, Cu, Fe, Mn, Mo, Ni, and Si). Results of the individual determinations were reported by the ACL to D. Diercks, who transmitted the reports to Commonwealth Edison. Results for carbon and sulfur were transmitted on May 18, 1995, and the report for metals was sent on May 31. One sample (Specimen No. 417) was subsequently reanalyzed to follow up on a high recovery for iron observed in the first measurements. Results of this reanalysis were reported on October 27, 1995. Copies of the Reports of Analytical results for the carbon and sulfur determinations and a summary report of the final results for metals are provided in Attachment 2.

Methodology: Carbon in each steel specimen was determined with a LECO Corporation Model WR-12 Carbon Determinator, which conforms to the test method described in ASTM Designation E 1019-94 (Total Carbon by the Combustion Instrumental Measurement Method). For these analyses, the instrument was calibrated and calibration verified with Carbon-in-Steel standards obtained from either LECO Corporation or Alpha Resources, Inc.; both of these suppliers reference traceability of their standards to NIST Reference Materials, including NIST 8j, 12h, 15g, 15h, and 335.

Sulfur was determined with a LECO Corporation Model 518 Determinator, which conforms to the test method described in ASTM Designation E 1473-94a (Sulfur by the Combustion-Iodate Titration Method). The instrument was calibrated with ACS Reagent Grade Potassium Sulfate according to manufacturer's instructions and the calibration was verified by analysis of the NBS Reference Material SRM 32d.

Metallic elements were determined by Inductively Coupled Plasma-Atomic Emission Spectrometry (ICP-AES) analysis of solutions prepared by dissolving weighed portions of the chips from each specimen in hydrochloric acid containing a small amount (few drops) of hydrofluoric acid. Although not yet an ASTM designated method, plasma spectrometry is a well-recognized and widely used technique for the determination of metallic elements in solution. The system used for the Commonwealth Edison surveillance specimens is an ARL Model 3520 Spectrometer equipped with glovebox containment for analysis of radioactive samples (a brief description of the ICP-AES capabilities in the ACL is provided in Attachment 3). The ICP-AES system was calibrated immediately before the samples were analyzed, using spectrometric standard solutions procured from either SPEX Industries Inc. of Edison, NJ, or Fisher Scientific of Fair Lawn, NJ. The SPEX industries standard is a multielement calibration standard that includes all the elements that were measured, except Mo and Si. For these latter two elements, Reference Standard Solutions from Fisher were used. Copies of the certification provided by the suppliers for both the SPEX and Fisher solutions are enclosed in Attachment 4. The certificate for the SPEX solution includes references that establish traceability of individual components of the standard to NIST. Certificates for these components are available for review if needed.

Both the dissolution procedure and ICP-AES measurements applied for the surveillance specimens were verified by carrying a sample of NBS 32d Steel through the analysis process along with the samples.

Quality Assurance: Activities in the ACL are carried out under an organizational Quality Assurance Plan (Analytical Chemistry Laboratory Quality Assurance Plan, Document No. C0030-0221, Revision 06, June 1994) structured in accordance with U.S. Department of Energy (DOE) Order 5700.6C, Quality Assurance. This plan incorporates the Quality Assurance (QA) requirements of ASME NQA-1 (Quality Assurance Program Requirements for Nuclear Facilities) as well as the DOE required elements of Training, Quality Improvement, and Independent and Management Assessments. By virtue of our QA program's conformance with the requirements of ASME NQA-1, ACL activities satisfy 10CFR50, Appendix B (Quality Assurance Criteria for Nuclear Power Plants and Fuel Reprocessing Plants). A copy of the Cover Page and Table of Contents of the ACL QA Plan is provided as Attachment 4. If you should want a copy of the entire document, please contact Fred Martino, ACL QA/QC Coordinator, at (708) 252-4474.

Sincerely,

Donald G Graczyk

Donald G. Graczyk
Group Leader, Chemical Analysis
Analytical Chemistry Laboratory
Chemical Technology Division

DGG:amb
Attachments

cc: w/attach.
J. Laidler (2)
D. Green
D. Bowers
F. Martino
D. Diercks (ET)
Subject: Information on Analysis of Steels

List of Attachments
to
Letter: J. Chynoweth, Commonwealth Edison Co.

Attachment 1: Table, Chronology of Analysis for Commonwealth Edison Steel Samples
(1 page)

Attachment 2: Reports of Analytical Results for Carbon, Sulfur, and Metals in Commonwealth Edison Steel Samples.
(3 pages)

Attachment 3: Summaries of ACL Capabilities for ICP-AES
(2 pages)

Attachment 4: Certificates of Analysis for ICP Reference Standard Solutions
(3 pages)

Attachment 5: Cover Page and Table of Contents, Analytical Chemistry Laboratory Quality Assurance Plan
(3 pages)

Attachment 1

Chronology of Analysis for Commonwealth Edison Steel Samples Analyzed by Argonne National Laboratory

Analytical Chemistry Laboratory
Chemical Technology Division

Event	Date	Remarks
Specimens Received at ANL (Special Materials Group)	May 4, 1995	Delivery to Special Materials Group
Specimens Transferred to Building 212 for Preparation (Machining)	May 4, 1995	
Samples Transferred to Analytical Chemistry Laboratory (Building 200) for Carbon/Sulfur Determinations	May 9, 1995	SPM Doc. No. E3259
Analyzed for Carbon	May 16, 1995	ACL Logbook 769, p. 51
Analyzed for Sulfur	May 18, 1995	ACL Logbook 769, pp. 52,53
Sulfur and Carbon Results Reported to D. Diercks	May 18, 1995	ACL Job No. 95-0240
Sulfur and Carbon Data Transmitted by D. Diercks to ComEd.	May 18, 1995	Date of transmittal is not certain; on-or-about.
Samples Transferred to Analytical Chemistry Laboratory (Building 205) for Metals Determinations	May 22, 1995	SPM Doc. No. C7002
Portions Dissolved in Mineral Acids	May 22, 1995	ACL Logbook 1174, pp. 67-70
Solutions Analyzed by ICP-AES (ARL 3520 Instrument)	May 23, 1995	ACL Logbook 1174, pp. 67-70
Metals Data Reported to D. Diercks	May 25, 1995	ACL Job No. 95-2062
Revised Metals Report Issued (Missing Decimal Point in One Fe Value)	May 31, 1995	ACL Job No. 95-2062
Metals Data Transmitted by D. Diercks to ComEd.	May 31, 1995	Date of transmittal is not certain; on-or-about.
Specimen 417 Reanalyzed	Oct. 22-23, 1995	ACL Logbook 1174, p. 170
Reanalysis Results Reported to D. Diercks	Oct. 27, 1995	ACL Job No. 95-2062, continued.
Metals Data Compiled into Summary Reportand Transmitted to ComEd	Nov. 22, 1995	See Attachment 2.

Attachment 2

Reports of Analytical Results for Carbon, Sulfur, and Metals in Commonwealth Edison Steel Samples.

ANALYTICAL CHEMISTRY LABORATORY
Argonne National Laboratory
Argonne, IL 60439

REPORT OF ANALYTICAL RESULTS

Sample Material: Steel Samples

Submitted by: D. Diercks

Date Received: 5/9/95

Date Reported: 5/18/95

Submitter's Sample No.	ACL Sample No.	Carbon, Wt. %	Sample Wt., g (for Carbon)	Sulfur, Wt. %	Sample Wt., g (for Sulfur)
417	95-0240-01	0.182	0.4822	0.024	0.5406
	-01 Duplicate	--	–	0.016	0.9597
433	-02	0.185	0.3402	0.015	0.9468
435	-03	0.192	0.2955	0.014	1.1772
443	-04	0.150	0.6596	0.012	0.9407
	-04 Duplicate	0.146	0.4027	–	–
44A	-05	0.174	0.2611	0.011	0.9893
44F	-06	0.193	0.2789	0.013	1.1380
44M	-07	0.170	0.3822	0.010	1.2739
45E	-08	0.166	0.3804	0.012	1.2740
4JC	-09	0.192	0.3303	0.017	0.9624
4J6	-10	0.199	0.2393	0.016	0.9839
	-10 Duplicate	0.188	0.3486	0.016	1.0231
	K_2SO_4	Known: 18.40% S	Found: 18.28% S		
	NBS Standard: SRM 32d	Known: 0.027% S	Found: 0.028% S		

OTE: Unused sample material will be returned to the Customer. Prepared samples will be discarded one (1) month fter the date of this report unless other arrangements are made. When making future inquiries regarding this report, lease reference the ACL sample number(s) above. For further information about the results reported here, please call . Ross at 2- 3492

eference(s): L. Ross Notebook No. 769, pp. 51-53.

opies To: D. Diercks
D. Green
D. Graczyk
L. Ross
ACL 200 File

Analyst(s): L. Ross

for L. Ross.

mb
19/95
84 (4-94)

ANALYTICAL CHEMISTRY LABORATORY
Argonne National Laboratory
Argonne, IL 60439

REPORT OF ANALYTICAL RESULTS

Sample Material: Steel Samples
Submitted by: D. Diercks
Date Received: 5/9/95
Date Reported: 5/18/95

Submitter's Sample No.	ACL Sample No.	Carbon, Wt. %	Sample Wt., g (for Carbon)	Sulfur, Wt. %	Sample Wt., g (for Sulfur)
	95-0240	Carbon Quality Control Standards			

Standard, Wt. % C	% Recovery Before Samples Run	% Recovery After Samples Run
0.0072	94.4	102.8
0.021	99.0	101.4
0.059	95.6	98.3
0.172	--	99.5

Carbon determined using the LECO WR-12 Analyzer.
Combustion is at ~1500°C in pure oxygen.
Instrument detection limit (IDL) is 25 µg C.
Carbon results: ±5%.

NOTES: Sulfur determined using the LECO Model 518 Determinator.
Sulfur results on samples: ±10%.

NOTE: Unused sample material will be returned to the Customer. Prepared samples will be discarded one (1) month after the date of this report unless other arrangements are made. When making future inquiries regarding this report, please reference the ACL sample number(s) above. For further information about the results reported here, please call L. Ross at 2- 3492

Reference(s): L. Ross Notebook No. 769, pp. 51-53.

Copies To: D. Diercks
D. Green
D. Graczyk
L. Ross
ACL 200 File

Analyst(s): L. Ross

/amb
5/19/95
MT-84 (4-94)

ANALYTICAL CHEMISTRY LABORATORY
Argonne National Laboratory
Argonne, IL 60439

REPORT OF ANALYTICAL RESULTS

Sample Material:	Machined Steel Chips	Date Received:	5/23/95
Submitted by:	D. Diercks	Date Reported:	5/25/95, 10/27/95

Submitter's Sample No.	ACL Sample No.	Wt. %						
		Cr	Cu	Fe	Mn	Mo	Ni	Si
417	95-2062-01	0.22	0.14	94.9	1.13	0.62	0.47	0.24
433	-02	0.24	0.14	97.4	1.27	0.51	0.57	0.21
435	-03	0.18	0.18	93.7	1.27	0.48	0.60	0.20
443	-04	0.091	0.19	94.8	1.37	0.50	0.69	0.42
44A	-05	0.086	0.18	92.0	1.33	0.46	0.70	0.39
44F	-06	0.089	0.19	93.1	1.35	0.47	0.71	0.39
44M	-07	0.097	0.18	91.3	1.32	0.49	0.64	0.39
45E	-08A	0.094	0.18	91.7	1.37	0.48	0.69	0.26
45E Dup	-08B	0.095	0.18	87.9	1.31	0.47	0.64	0.20
4JC	-09	0.24	0.14	90.1	1.21	0.49	0.54	0.26
4J6	-10	0.24	0.15	93.4	1.27	0.51	0.56	0.24
					QA			
	NBS 32d Steel							
	Found	0.64	0.090	92.8	0.76	0.026	1.20	0.31
	(Known Value)	(0.71)	(0.096)	(96.4)	(0.80)	(0.038)	(1.19)	(0.30)
	% Recovery	90.1	93.8	96.3	95.0	68.4	101	103

Estimated uncertainties: ±10%

Summary report compiled from original report of 5/25/95 and followup analysis on Specimen 417 (ACL No. 95-2062-01), performed on 10/23/95.

NOTE: Unused sample material will be returned to the Customer. Prepared samples will be discarded one (1) month after the date of this report unless other arrangements are made. When making future inquiries regarding this report, please reference the ACL sample number(s) above. For further information about the results reported here, please call D. Bowers at 2- 4354.

Reference(s): Data recorded in CMT Book No. 1174, pp. 67-70, 170.

Copies To:	D. Diercks	ACL 200 File	Analyst(s):	D. Bowers
	D. Green			C. Sabau for DLB
	D. Graczyk			
/lll/amb	F. Martino			
11/22/95	D. Bower			
	C. Sabau (2)			

CMT-84 (4-94)

Attachment 3

Summaries of ICP-AES Capabilities

1. Inductively Coupled Plasma-Atomic Emission Spectrometry
2. "Hot" Inductively Coupled Plasma-Atomic Emission Spectrometry

Inductively Coupled Plasma-Atomic Emission Spectrometry (ICP-AES)

Application: The technique of ICP-AES is used to characterize diverse analytical samples by the simultaneous measurement of multiple cations in solution. The sample solution is introduced into an argon plasma in which the cations are excited and emit light at discreet wavelengths; the intensity of the emission is related to the concentration of ions in the sample.

Any elemental constituent that, upon excitation, emits light in the 180-800 nm spectral region can be quantitatively determined by ICP-AES. Most metallic elements and common non-metals such as boron, silicon, and phosphorus can be measured.

Instrumentation: Measurements are performed by using an Instruments SA JY86 Spectrometer System, which incorporates a 48-channel polychromator (JY-48), capable of the simultaneous determination of 42 elements, and a scanning monochromator (JY-38) for the determination of additional analytes. Both instruments are focused on a single plasma excitation source. The polychromator system is controlled by, and data are stored on, a DEC PDP-11/23 computer. A Gateway 2000 PC is used with the monochromator system for operation and data storage.

Sample Requirements: Samples for analysis must be in solution, and the concentration of total dissolved solids cannot exceed 20 mg/mL. Any mineral acid can be used for sample dissolution, but the acidity should be in the range 0.1 to 0.5N. Separation techniques (e.g., ion exchange, extraction) can be applied to improve detectability.

Detection Limits: Elements at concentrations from 1 to 500 ng/mL in aqueous solution can be detected, depending on the wavelength and sensitivity. Generally, results are reported if the measured concentration exceeds three times the detection limit; otherwise a "less-than" value is provided.

Accuracy: Typically, measurements can be made with an estimated accuracy of 10% at analyte concentrations of 10 to 100 times the detection limit, and 3-5% if the concentration exceeds 100 times the detection limit.

Interferences: The method is reasonably free from chemical and ionization interferences. Spectral overlaps have to be evaluated on an individual basis and may require the use of alternative wavelengths and/or spectral interference corrections.

Time Required for Analysis: The following factors enter into time assessment:

1. Sample preparation (dissolutions, separation)
2. Number of samples of similar composition
3. Number of elements determined
4. Matrix complexity
5. Instrument calibration requirements
6. Analytical quality control

Based on these considerations, analytical time may range from 0.5 to 8 hours per sample. Usually multiple samples can be analyzed with less time per sample.

*****

Some information on this page is very general. Most analytical problems have unique characteristics that are not easily generalized. Please contact the ACL Office (2-4473) to obtain specific information about the application of this technique to your analytical problem.

Note: This summary is an updated version of one issued 3/28/84, and should replace it.

September 8, 1995

ANALYTICAL CHEMISTRY LABORATORY

"Hot" Inductively Coupled Plasma/Atomic Emission Spectrometry (ICP/AES)

Applicability: The ICP/AES system is used to determine the concentration of elements that, upon excitation, emit light in the 180 to 800 nm spectral region. This instrument, with the excitation source (plasma torch) interfaced with a glove box, is primarily used for elemental analyses of materials that are radioactive. Solutions with high levels of alpha-emitting isotopes and moderate levels (<100 mR/hr) of β, γ radionuclides can be safely analyzed with this instrument. This instrument can also be used to determine radioactive elements such as ^{99}Tc and heavy elements such as Th, U, Pu, Am, Np, and Cm.

Instrument: The ACL has an Applied Research Laboratories (ARL) Model 3520 scanning spectrometer that is interfaced to a glove box facility. The spectrometer is controlled with a DEC PDP 11/23+ computer and associated equipment.

Sample Preparation: Samples that are not received as solutions must be dissolved and also may have to be chemically separated in special cases by such techniques as ion exchange.

Detection Limits: The detection limit varies from 1 to 500 ppb in solution.

Accuracy and Precision: Accuracy and precision are dependent on sample signal response, which depends on the amount of element in the sample. Uncertainties of 5 to 10% are typical.

Interferences: Interference levels are dependent on the sample matrix, which could cause background shifts and/or spectral overlap. Background or baseline shift corrections can be made and spectral interference curves can be generated. Also, alternative wavelengths may be chosen for the particular element of interest.

Analysis Time: Analysis time varies with the sample preparation required and with the number of elements to be determined.

*****

Some information on this page is very general. Most analytical problems have unique characteristics that are not easily generalized. Please contact the ACL Office (2-4473) to obtain specific information about the application of this technique to your analytical problem.

December 20, 1988

Attachment 4

Certificates of Analysis for ICP Reference Standard Solutions

Rec'd. 10-25-94 AE

SPEX

Bottle #1 to E. Huff 200-K Wing 500mL
Bottle #2 to D. Bowers 205 500mL
(Each has a set of the MSDS sheets) AE

CERTIFICATE OF ANALYSIS

SPEX PART #: XANL-2

DESCRIPTION: 100 ug/ml each of Al, Ba, Be, Ca, Cd, Co, Cr, Cu, Fe, Mg, Mn, Ni, Pb, Sn, Sr, Tl, Zn, Zr and V

MATRIX: 5% HCl in H_2O

LOT NO: 4-244MM

The enclosed certificates represent analyses of the starting material of all elements contained in your Custom Multi-Element Solution Standard.

Each individual element in the solution has been referenced and an appropriate certificate is included.

Custom Multi-Element solutions are prepared by pipetting and diluting stock single-element solutions and a final ICP check is performed. Each solution is guaranteed stable and accurate to within +/-0.5% of labeled concentration for one year from date of shipment.

Please keep these certificate on file.

cd

F-115

FISHER SCIENTIFIC CHEMICAL DIVISION
One Reagent Lane, Fair Lawn, NJ 07410

ANALYTICAL CONTROL LABORATORY ANALYSIS

Name & Grade:
SILICON REFERENCE STANDARD SOLUTION 1ML = 1MG Si
Catalog Number: SS465
Lot Number: 947038
Date of Testing/Mfg: 12/14/94
P.O./ Other Customer ID:

This is to certify that units of the above mentioned lot number were tested and found to comply with the specifications of the grade listed. The following are the actual analytical results obtained:

Test	Unit	Result
APPEARANCE	PASS/FAIL	PASS-CLEAR COLORLESS LIQUID
CON-GRAVIMET.MTH-PPM	PPM	1002.00

Approved by:

Frederick H. Turk, FL Liquid Supv.	Robert Dowd FL Dry Supv.	Edgar E. Hess, BPF Lab Supv.

Date: 12/19/94 (Signed and dated original is on file)

NOTE: The data listed is valid for all package sizes of this lot of product, expressed as a extension of the catalog number listed above. If there are any questions with this certificate, please call Chemical Services (option 5) at (800) 388-8355.

Ref. No. SS465.947038.B1.

Location: FL

FISHER SCIENTIFIC CHEMICAL DIVISION
One Reagent Lane, Fair Lawn, NJ 07410

ANALYTICAL CONTROL LABORATORY ANALYSIS

Name & Grade:
MOLYBDENUM REFERENCE STANDARD SOLUTION, 1ML = 1MG Mo
Catalog Number: SM113
Lot Number: 936313
Date of Testing/Mfg: 11/04/93
P.O./ Other Customer ID:

This is to certify that units of the above mentioned lot number were tested and found to comply with the specifications of the grade listed. The following are the actual analytical results obtained:

Test	Unit	Result
APPEARANCE	PASS/FAIL	PASS-CLEAR COLORLESS LIQUID
CONCENTRATION IN PPM	PPM	999.000
CONCENTRATION_AA-P/F	PASS/FAIL	PASS

Approved by: Frederick H. Turk, FL Analytical QA Supv. or Edgar E. Hess, BPF Analytical QA Supv.

Date: 11/05/93 (Signed and dated original is on file)

NOTE: The data listed is valid for all package sizes of this lot of product, expressed as a extension of the catalog number listed above. If there are any questions with this certificate, please call Steven P. Davis, Analytical QA Manager, at (201) 703-3149.

Ref. No. SM113.936313.B1.

Location: FL

Attachment 5

Cover Page and Table of Contents, Analytical Chemistry Laboratory Quality Assurance Plan.

CONTROLLED COPY NO. 12

ARGONNE NATIONAL LABORATORY
9700 SOUTH CASS AVENUE
ARGONNE, ILLINOIS 60439

ANALYTICAL CHEMISTRY LABORATORY
CHEMICAL TECHNOLOGY DIVISION

QUALITY ASSURANCE PLAN

DOCUMENT NO. C0030-0221
REVISION 06

Reissued: 6/28/94

(Date of Original Issue: February 26, 1982)
(See Page 1 for Revision Record)

		For	Date
Prepared by:	Frederic J. Martino ACL QA/QC Coordinator	CMT/ACL	6/27/94
Reviewed by:	Richard L. Malecha CMT QA Representative	CMT/ACL	6-28-94
Approved by:	David W. Green ACL Manager	CMT/ACL	6/28/94

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Distribution of ACL QA Plan

ACL Staff
J. E. Battles (CMT)
L. D. Chipman (OQA)
R. F. Malecha (CMT)
D. C. Parzyck (EQO)
J. R. Phillips (LANL)