

**AIR QUALITY SECTION  
TOXICS PROTECTION UNIT  
AIR TOXICS ANALYTICAL SUPPORT TEAM  
REPORT OF INVESTIGATION AT  
CAROLINA SOLITE CORPORATION, AQUADALE, NC, STANLY COUNTY  
Investigation #960009**

On June 13, 1996, the following Air Quality Section (AQS) staff conducted a renewal inspection at Carolina Solite Corporation: Jessica Bellas, Toxics Protection Unit; David Edgington, Stationary Source Compliance Unit; and Ronald Slack, Mooresville Regional Office. At the request of the Air Permits Branch, the renewal inspection was conducted to provide information to support renewal of Permit # 3225R10 for Carolina Solite. The specific goals of the inspection were to ensure that Solite was complying with the Continuous Emission Monitoring Systems (CEMS) requirements of Permit No. 3225R10 and whether the analytical laboratory at the Oldover facility had shown improvements since the ATAST Investigation Report #960007.

#### **CEM REQUIREMENTS**

Prior to this inspection, an outstanding issue concerned the CEMS was the accuracy of the daily calibrations. In February of 1996, it was noted by David Edgington that during the daily calibrations the CEMS was being adjusted prior to recording the drift. On March 18, 1996, a letter was sent to Walter Rippey of Carolina Solite from Michael Aldridge of the AQS (see attachment) stating that the drift must be recorded prior to making adjustments.

This inspection could not positively affirm that Carolina Solite has corrected this problem. Todd Callahan and Adrienne McLain, both employees of Carolina Solite, gave assurances that they are quantifying the drift prior to making adjustments but could not offer any documentation to support this claim.

The exceedances recorded by CEMS during the Second Quarter of 1995 were investigated further at this inspection. These exceedances were reported to the Stationary Source Compliance Unit (SSCU) by Carolina Solite but Carolina Solite claimed that they were due to analyzer malfunctions. A review of the operation and maintenance logs for the CEMS by David Edgington revealed that on the days in which the exceedances occurred there were no log entries made to document these malfunctions. The absence of a record documenting malfunctions for the exceedances in their logs shows that Carolina Solite can not demonstrate that the analyzer malfunctions actually occurred. Therefore, it was recommended by the Stationary Source Compliance Unit that enforcement be sought for nine (9) violations of the 2.3 lb SO<sub>2</sub>/mmBtu three-hour rolling average emission standard as stated in condition A.2. The inspection report completed by David Edgington is attached.

#### **Laboratory Issue**

The ATAST Investigation Report #960007 documented several deficiencies in the Oldover laboratory. Some of the primary problems documented in report #960007 were lack of an approved

analytical methodology for the detection of PCBs, no quality assurance or quality control practices and a lack of recordkeeping for the laboratory. It is important to note that any analytical methodology, quantitative or qualitative, must have Quality Assurance/Quality Control (QA/QC) to show that the results are valid. At this inspection, the Oldover laboratory had not yet instituted an approved analytical methodology for PCBs. They explained that the method being used is a "hybrid" of EPA Method 600/4-81-045 and another method that was developed by Carolina Solite in 1984. However, the method that was being implemented in the laboratory on 6/13/96 does not have many similarities to EPA Method 600/4-81-045.

Neither Oldover nor Carolina Solite have proven that the method they developed to detect PCBs in hazardous waste is scientifically valid. The "Method Validation" was not available at the time of this inspection as we were informed that the Corporate Chemist from Carolina Solite was still finishing calculations. However, after interviewing the chemists in the laboratory about the contents of the "Method Validation," they could not supply sufficient evidence that the method validation had experimentally analyzed sufficient laboratory variables. According to the chemists, the method validation performed at the Oldover laboratory consisted of both chemists running a total of 63 spiked samples (7 spiked samples at the same concentration performed for each of the 9 aroclors  $\rightarrow 9 \times 7 = 63$ ) using the "hybrid" method. However, since a similar "method validation" was not performed using an analytically reliable method, there are no means available to compare the "hybrid" method to a reliable method. The Oldover laboratory must perform a side by side comparison of the "hybrid" method with EPA Method 600/4-81-045. In an effort to obtain the "method validation," a letter was sent to Adrienne McLain from Jessica Bellas on July 3, 1996 to request this information. It has not been received by the AQS to date.

Stephanie Wheeler, the Oldover Chemist, said during the renewal inspection on 6/13/96 that it takes about 30 minutes to run the "fingerprint parameters" tests. Mike Deyo (Carolina Solite Corporate Chemist) said that the Oldover Chemist performs the following tests in "30 minutes, sometimes 40 minutes." The "fingerprint parameters" tests are as follows: ash, water content, chlorine, BTU, specific gravity and PCBs. It is disconcerting that the Oldover chemist is only given 20-40 minutes to run these six tests. Although it may be possible to run these tests in this short period of time, the tests would certainly be rushed which could result in sloppiness. It is understood that the laboratory results must be prompt so there are no interferes with operations of the plant but promptness can not be used as justification to not perform the analytical tests with sufficient care.

Since the inspections at the laboratory in February 1996 (ATAST Investigation Report #960007), the following improvements were observed at the Oldover laboratory as follows:

- 1.) A "back-up chemist" has been hired for the laboratory and the AQS recommended that the "back-up chemist" continue employment.
- 2.) New equipment has been purchased for a Florisil clean-up procedure on "dirty" samples. It is important to note that as of this inspection the equipment has not yet been used for a Florisil clean-up.
- 3.) The 9 aroclor standards are injected once a month. However, these standards should be injected at concentrations of 5 ppm or lower to ensure the 5 ppm detection limit as reported to AQS by Carolina Solite in a letter from E.E. Martin dated 3/1/84.

4.) There is one daily standard injected. This daily standard is a mixture of only 3 of the 9 aroclors. It is recommended that all 9 aroclors be used in the daily standard. However, if problems arise and all nine aroclors can not be used, the AQS will accept a proper QC check in lieu of using 9 aroclors for the standard providing there is a previously approved method validation comparing the aroclors. It was observed during the inspection that the Oldover laboratory is currently recording the retention time and areas for the chromatograms for the standards. However, they are not calculating the percent difference and have not established an acceptability level for the percent difference. In other words, the data that is being generated by the Oldover laboratory is not being utilized. For example, on the standard that was run on 5/30/96 in the Oldover laboratory, there is 26.7 percent difference between peak 15 of the monthly standard and peak 15 of the daily standard. This amount of difference could be unacceptable. When the percent difference becomes unacceptable, the monthly standards MUST be reinjected before the samples are run.

5.) A QC log book is being maintained for the PCB method. This log book includes chromatograms of the standard, certificate of analysis for the aroclor standards, an ECD maintenance check list and retention times and areas for the standards. However, as previously mentioned, the percent difference is not calculated and there is no "cutoff" limit for acceptability. Again, the data generated is not being utilized.

It was noted in the inspection that the chromatograms for the actual samples are stored separately since they must be kept with the manifest for the RCRA permit. A preventative maintenance log is also maintained.

6.) Spiked samples are being injected every 20 samples and recorded. The purpose of a spiked sample is to determine relative percent difference and recovery. Again, the relative percent difference and the recovery is not calculated so it would seem that this data is not being utilized. When the percent difference becomes unacceptable, the monthly standards MUST be reinjected before the samples are run.

7.) The aliquot of sample used by Solite in their analyses has been increased from 20 microliters to 0.5 milliliters. However, the EPA method calls for an aliquot of 1 milliliter. While this is an improvement, there is still no method performance to show that this modification is valid.

8.) There has been a "water back extraction" clean up implemented to clean "dirty" matrices. This clean-up procedure is listed in the "Carolina Solite Oldover Aquadale Laboratory Quality Assurance Plan" dated May 28, 1996 which has not been critically reviewed by the Division of Air Quality. It has been used about 3 times in during May and the first two weeks of June.

Finally, it was explained to Jessica Bellas and Ron Slack by Mike Deyo, Adrienne McLain and Stephanie Wheeler that the PCB method is "qualitative" as opposed to "quantitative." They feel that this is only a screening test and does not have to follow an approved "quantitative" methodology since it is only a "qualitative" test. It is important to note that any analytical methodology, quantitative or qualitative, must have Quality Assurance/Quality Control (QA/QC) to show that the results are valid. Although the AQS understands that this method is only a screening method, a "qualitative" test still requires proper QA/QC. It appears that the use of this "qualitative" test argument is being employed only so that the percent difference for the standards and spiked samples



State of North Carolina  
Department of Environment,  
Health and Natural Resources  
Division of Environmental Management

James B. Hunt, Jr., Governor  
Jonathan B. Howes, Secretary  
A. Preston Howard, Jr., P.E., Director



June 21, 1996

**MEMORANDUM**

TO: Mark Hawes  
Regional Air Quality Supervisor  
Mooresville Regional Office

FROM: David Edgington - Environmental Engineer I *DE*

THROUGH: Dennis Igboko - Environmental Engineer II *DI*

SUBJECT: June 13, 1996, Inspection of the Continuous Emissions Monitoring System at  
Carolina Solite Corporation  
Aquadale, North Carolina  
Stanly County

On June 13, 1996, at the request of the Permits Branch, I conducted an inspection of the Continuous Emission Monitoring System (CEMS) at Carolina Solite. The purpose of the inspection was to ensure that Solite was complying with the CEM requirements of their current permit (Permit No. 3225R10).

Prior to the inspection, the only outstanding issue concerning the CEMS was the daily calibrations. In February of 1996, it was noted that during the daily calibrations the CEMS was being adjusted prior to recording the drift. This was brought to Solite's attention in our March 18, 1996, letter informing them that the drift must be recorded prior to making adjustments.

The inspection could not positively confirm nor refute that Solite has corrected this problem. However, both Todd Callahan and Adrienne McLain gave assurances that they are quantifying the drift prior to making adjustments.

During the inspection, another potential problem with the CEMS was discovered. This problem involves exceedances recorded by CEMS during the Second Quarter of 1995. Solite reported these exceedances, but claimed that they were due to analyzer malfunctions. Table 1 lists the exceedance and the reasons given by Solite for the exceedances.

Table 1: Exceedances at Carolina Solite

Date	Time	Exceedance (lb SO <sub>2</sub> /mmBtu)	Reason given for Exceedance
April 8, 1995	12:00 pm	2.4	Analyzer Pre-Amp Gain Required Adjustment.
April 23 & 24, 1995	22:00 - 01:00	2.7, 2.9, & 2.7	Solenoid Valve Sticking
May 3, 1995	12:00 - 13:00	2.5 & 2.4	Analyzer Pre-Amp Gain Required Adjustment.
May 20, 1995	15:00	2.5	Analyzer Pre-Amp Gain Required Adjustment.
May 23, 1995	04:00 & 20:00	2.7 & 2.7	Analyzer Pre-Amp Gain Required Adjustment.

A review of the operation and maintenance logs for the CEMS revealed no log entries on the days in which the above (Table 1) exceedances occurred. When this was brought to Mr. Callahan's attention he reviewed his reports to Adrienne McLain and could not find any reference that these conditions existed during the exceedances. In addition, during our closing meeting this issue was brought to Ms. McLain's attention at which time she stated that she thought the analyzer manufacturer was on-site at those times and that she would look into trying to find records for those problems. It is unlikely that the manufacturer was on-site for more than one of those incidences since they occurred on different days over a two month period and that some of the exceedances occurred either very late at night or very early in the morning. Also, there should be calibration records indicating the inaccuracy of the analyzer during these time periods, as well as a calibration demonstrating the problem had been corrected.

Since there is no record of the reasons given for the exceedances in their logs, Solite cannot demonstrate that the analyzer malfunctions actually occurred. Therefore, it is our recommendation that enforcement be sought for nine (9) violations of the 2.3 lb SO<sub>2</sub>/mmBtu emission standard as stated in condition A.3.c.ii. An enforcement package will be prepared by Headquarters for these violations.

Other than the above mentioned issues, the CEMS appear to be adequately recording the emissions from Kilns 5 & 6.

If you have any questions concerning the enforcement action or any other issue, please call me at (919)715-5872.

cc: Lee Daniel  
 Mike Aldridge  
 Jerry Clayton - Permits Branch  
 Ronald Slack - MRO  
 Central Files - Stanly County

State of North Carolina  
Department of Environment,  
Health and Natural Resources  
Division of Environmental Management

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CERTIFIED MAIL  
P 281 676 873

March 18, 1996

Mr. Walter A. Rippey  
Plant Manager  
Carolina Solite Corporation  
P.O. Box 987  
Albemarle, NC 28001

**SUBJECT:** Notice to Repeat Relative Accuracy Test Audit for 1996  
Warning - Daily Calibration Drift (CD) Test Procedures  
Air Permit No. 3225R10

Dear Mr. Rippey:

This letter is to inform Carolina Solite that the relative accuracy test audit (RATA) performed on February 20, 1996 will not be accepted as the annual certification for the SO<sub>2</sub> monitoring system. The test is rejected on the basis that the plant CEM system was not in the time shared mode as it normally operates. By not operating the CEM in the time shared mode the RATA does not demonstrate that the CEM can accurately measure the SO<sub>2</sub> emissions in the time shared mode. This requirement was made known to you in our February 1, 1996 letter, yet Solite refused to operate the system in the time shared mode and did not respond until February 26, 1996, nearly a week after testing was completed.

Carolina Solite is therefore requested to repeat the RATA for the SO<sub>2</sub> monitoring system no later than June 31, 1996. Failure to perform the RATA by this date may result in a Notice of Violation (NOV) for failure to perform the annual testing on this system as required by Specific Condition and Limitation 3.c.i of your Air Permit. Solite must notify this office at least fifteen (15) days prior to testing so that arrangements can be made to observe the test. In addition, it is recommended that a test protocol be submitted at least thirty (30) days prior to testing to ensure that all testing will meet with the DEM's approval.

On February 19 and 20, 1996, David Edgington of this office conducted a RATA observation on Kiln No. 6. During the test observation, he noticed a problem with the manner in which the daily

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Mr. Walter A. Rippey  
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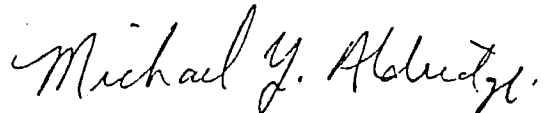
calibration drift (CD) check was being performed. Specifically, the technician was adjusting the analyzer prior to recording the CD contrary to the requirements in 40 CFR 60, Appendix B, Performance Specification 2, Section 6. According to the technician it is normal procedure to adjust the analyzer if the drift is greater than 25 ppm (2.5% of Span). However, by adjusting the analyzer prior to recording the calibration value the CD can not be determined. In order to meet the requirements of the applicable regulations cited above, the monitoring system must be allowed to complete its calibration without any adjustments to the system. If the system fails the calibration, you can then make the necessary adjustments to the system, and perform the calibration test again recording the new calibration value. Please note, records for all calibrations must be kept on file for at least five (5) years as required by Specific Condition and Limitation 13 of your Air Permit.

Please regard this as a warning and note that failure to perform the calibration drift checks in accordance with the applicable regulations may result in a Notice of Violation (NOV) with possible enforcement action.

Because of the above mentioned calibration problems, the Division of Environmental Management (DEM) is requiring Solite to submit the daily CD reports with your quarterly reports until further notification. These calibration reports will be reviewed with the emissions reports to ensure compliance with the regulations.

If you have any questions, please contact me at (919) 733-1470 or David Edgington at (919) 715-5872 .

Sincerely,



Michael Y. Aldridge  
Supervisor, Stationary Source Compliance

cc: Lee Daniel  
David Edgington  
Keith Overcash - MRO  
Mike Landis - MRO  
Adrienne McLain - Carolina Solite Corporation  
Central File - Stanly County

DATE: 5/30

RANGE:

ATTENUATION:

SAMPLE NUMBER	RETENTION TIME PREVIOUS	AREA PREVIOUS	RETENTION TIME TODAY	AREA TODAY
1	1.339	88147	1.366	71650
2	1.466	57701	1.495	45133
3	1.697/1.805	466075	1.729/1.839	371977
4	2.078	220358	3.7 = 2.10	176501
5	2.310	174600	2.350	136428
6	2.457	170783	2.506	137529
7	2.937	468654	2.985	371602
8	3.419	512332	3.473	405429
9	4.188	426303	4.253	345363
10	4.946	789950	5.018	622026
11	5.778	751780	5.862	583594
12	6.780	777913	6.874	600406
13	7.806	592433	7.907	454653
14	8.924	576399	9.053	428744
15	10.667	855526	10.807	625080
16	12.572	320816	12.743	240414