

COUNTY OF



ALLEGHENY

RICH FITZGERALD
COUNTY EXECUTIVE

NOTICE OF VIOLATION

July 21, 2021

CERTIFIED MAIL - 9489 0090 0027 6047 4907 49

Mr. Scot Whyte, Plant Manager
INEOS Composites US, LLC
2650 Neville Road
Pittsburgh, PA 15225

RE: Notice of Violation #210702 – Violations of Operating Permit 0037-OP19b Conditions IV.8 and V.A.3.r; Article XXI (“Air Pollution Control”), §2108.01.c, §2103.12.a.2.B, §2103.12.h.1, and §2103.12.i; and 40 CFR 62.14630 at property 2650 Neville Road, Pittsburgh, PA 15225.

Dear Mr. Whyte:

This letter constitutes notification of ACHD’s determination that the following violations occurred at the INEOS Composites US, LLC (INEOS) plant on Neville Island.

1) Failure to timely report a breakdown (§2108.01.c)

On March 20, 2020, the ACHD received notice of a breakdown of the Polyester Resin Plant Thermal Oxidizer due to a power outage (Breakdown No. 21605). The time of the breakdown was 9:47 PM. The breakdown was initially reported at 11:10 PM, 83 minutes after occurrence. Such late reporting is a violation of OP No. 0037-OP19b (“OP19b”) Condition IV.8 (§2108.01.c), which states:

In the event that any air pollution control equipment, process equipment, or other source of air contaminants breaks down in such manner as to have a substantial likelihood of causing the emission of air contaminants in violation of this permit, or of causing the emission into the open air of potentially toxic or hazardous materials, the person responsible for such equipment or source shall immediately, *but in no event later than*



DEBRA BOGEN, MD, DIRECTOR
ALLEGHENY COUNTY HEALTH DEPARTMENT
AIR QUALITY PROGRAM
301 39TH STREET • CLACK HEALTH CENTER • BUILDING 7
PITTSBURGH, PA 15201-1811
PHONE (412) 578-8103 • FAX (412) 578-8144
24-HR (412) 687-ACHD (2243)
WWW.ALLEGHENYCOUNTY.US/HEALTHDEPARTMENT



sixty (60) minutes after the commencement of the breakdown, notify the Department of such breakdown and shall, as expeditiously as possible but in no event later than seven (7) days after the original notification, provide written notice to the Department.

2) Failure to report an exceedance of the limit for HCl from Table 1 of 40 CFR 62 Subpart III, Commercial and Industrial Solid Waste Incinerators (CISWI), 62 ppm measured at 7 percent oxygen, dry basis at standard conditions (62 ppmdv @ 7% O₂) within 30 days of receiving the sample result.

On January 14, 2021, the ACHD received notification from INEOS Composites US, LLC of a sampling event in which the Maximum Theoretical Concentration (MTC) calculation indicated an exception for hydrogen chloride (HCl). This sampling event took place on September 1, 2020 during a compliance test of the Polyester Resin Plant, Thermal Oxidizer stack. The notification indicated that the analytical report for the September 1, 2020 sampling program (Eurofins Test America) was dated 9/15/2020. The report was submitted to ACHD and EPA almost 4 months following this date. This is in violation of Condition V.A.3.r of Permit No. 0037-OP19b, which states:

At any time the permittee identifies a sample result from the liquid waste analyses that exceeds the MTC established as per condition V.A.3.q above for the sampling time frame, the permittee will have thirty (30) days from the date of receiving the sample result to notify the US EPA and the Department.

3) Failure to follow the Waste Analysis Plan, Table 5.2 Analytical Methods, Total Chlorine (OP19b Condition V.A.3.n)

Condition V.A.3.n of OP19b states, “Analytical methods use{d} for the liquid waste analyses shall be in accordance with Section 5 of the WAP. [§2103.12.a.2.B; §2103.12.i]” The Waste Analysis Plan (WAP) was submitted to US EPA via email December 10, 2004 and forwarded to the ACHD on December 7, 2005. Table 2 of the 2004 WAP lists the analytical method for total chlorine as “SW-846 Methods 5050 and 9056” (see Attachment 1 to this NOV). Method 5050 is a sample preparation method used in conjunction with Method 9056. Also, Method 9056 was renamed Method 9056a in 2007 without any substantive changes other than clarification (Method 9056a uses ion chromatography as does Method 9056). Method 9056a is approved by U.S. EPA and is an acceptable substitute for Method 9056.

INEOS has not followed the analytical method specified in the WAP, Method 9056 (or 9056a), since it first began performing analyses required under CISWI (40 CFR Part 62 Subpart III). All liquid sample analyses since 2005 were performed using SW-846 Method 9251 (email from Eric Hunsberger to James Topsale June 16, 2011, see Attachment 2). Method 9251 is a colorimetric method using mercuric thiocyanate and ferric nitrate solution.

In addition, since at least March 2011, the normal contract laboratory for INEOS, Test America, has been sending split samples of what INEOS sent them to a subcontracted laboratory for analysis by XRF. INEOS contacted US EPA Region 3 regarding their XRF analyses run using Bruker AXS Spectraplus software for XRF Standardless Analysis. EPA Region 3 did not agree

that XRF was an “equivalent or better method” compared to SW-846 Method 9056. EPA’s concerns involved QC, calibration, and sensitivity. Subsequently, INEOS asked if EPA had the same concerns regarding SW-846 Method 9251. EPA determined that Method 9056A was a better method “because it contains more complete and rigorous QC requirements” and recommended that the WAP be amended to include Method 9056A (instead of 9056). See Attachment 2 for the email thread showing this conversation.

The ACHD has not received a complete, revised Waste Analysis Plan since the original Title V Operating Permit was issued in 2007. INEOS’s 2017 Operating Permit renewal application included Section 5 of the WAP with Table 2 showing SW-846 Method 9251 as the analytical method for total chlorine and with a request to revise the section to allow the use of XRF rather than Method 9251. Again, this makes the presumption that SW-846 Method 9251 was the approved method for total chlorine analysis, which is incorrect. SW-846 Method 9056 was the approved method and has been updated to Method 9056A. The use of SW-846 Method 9251, XRF, or an average of the two methods is not part of the Waste Analysis Plan that was approved for the Title V Operating Permit and is not acceptable.

The Department requests that INEOS contact the Department to discuss the alleged violations and corrective actions.

This Notice of Violation is neither an order nor any other final action of the Allegheny County Health Department. It neither imposes nor waives any enforcement action available to the Department under any of its statutes. If the Department determines that an enforcement action is appropriate, you will be notified of the action. Please be aware that any violation of the Article XXI regulations subjects a person to a variety of enforcement actions, including a civil penalty of up to \$25,000 per violation per day.

The Allegheny County Health Department is currently reviewing this violation to determine whether corrective actions are required and if a civil penalty is appropriate. Should you have any questions or require additional information, please contact Shannon Sandberg at (412) 578-7969 or email at shannon.sandberg@alleghenycounty.us.

Sincerely,



Shannon Sandberg
Enforcement Section Chief
Air Quality Program

enclosure

ATTACHMENT 1

Waste Analysis Plan, Section 5 – Analytical Methods

Ashland Specialty Chemical Company

December 10, 2004

Section 5

Analytical Methods

This section presents a description of the analytical methods for each parameter. In order to ensure the waste analysis program is capable of providing reliable data, Ashland will utilize standard testing methods from sources accepted by the USEPA. The methods referenced in this plan are described in:

- *Test Methods for Evaluating Solid Waste, Third Edition*, November 1986, and Updates (SW-846);
- ASTM Standard Methods; and/or
- Alternate equivalent methods approved in advance in writing by the Administrator.

5.1 Laboratories

Analyses will be performed by off-site contract laboratories. When a new contract laboratory is used to perform analyses, Ashland will review the laboratory quality assurance and quality control (QA/QC) plan. A copy of the QA/QC plan for each contract laboratory will be maintained on file. Only laboratories with acceptable QA/QC practices will be used to perform the analyses.

5.2 Characterization Parameters

The analytical methods that will be used for the characterization parameters for the liquid waste are provided in Table 5-1. Ashland will evaluate the parameters annually.

Table 5-1
Analytical Methods for Characterization Parameters

Parameter	Analytical Method ¹
Higher heating value	ASTM Method D-240
Specific gravity or density	ASTM Method D-1298 or ASTM Method D-1963
Viscosity	ASTM Method D-445

¹ The referenced analytical method, or an equivalent method, will be used in determining each parameter.

5.3 Compliance Parameters

The analytical methods that will be used for the compliance parameters are provided in Table 5-2. For calendar year 2005, Ashland will analyze for these parameters twice per month, and if no exceptions to the Maximum theoretical concentration calculated values occur, will sample twice per quarter in calendar 2006. If there are no exceptions to the MTC calculated values following 2006, Ashland may petition

the agency with primacy over this program for a reduced sampling frequency. The MTC method is demonstrated in section 7.4.5.

**Table 5-2
Analytical Methods for Compliance Parameters**

Parameter	Analytical Method ¹	Anticipated Lower Detection Limits
Total chlorine	SW-846 Methods 5050 and 9056	50-100 mg/kg
Mercury	SW-846 Method 6020, 7470A, or 7471A	2.5 – 10 ug/l
Cadmium	SW-846 Method 6010B or 6020	1.5 – 10 ug/l
Lead	SW-846 Method 6010B or 6020	0.2 – 10 ug/l

¹ The referenced analytical method, or an equivalent method, will be used in determining each parameter.

5.4 Recordkeeping

The laboratory will reduce and validate all of the analytical data. After the analyses have been completed, the laboratory will generate a report. The analytical reports will be maintained in the facility's operating log for a period of five years.

ATTACHMENT 2

email; Eric Hunsberger to Abbie Yant; September 8, 2011

and email thread therein

ASHLAND

Fw: 6/16/11 Email Request - Approval of an Alternative Compliance Method for Determining Total Chlorine in Ashland's Neville Island Waste Feed Stream to its CISWI Unit.

Eric L. Hunsberger To: Yant, Abbie
Cc: Nicole M Hamilton

09/08/2011 04:27 PM

Abbie,

Please see the EPA's response to the information I forwarded to them from EMSL on the XRF method. Find out if they can do what the EPA wants and let me know, so we can continue to pursue this if possible.

Thanks,

Eric

----- Forwarded by Eric L. Hunsberger/CPD/ASCC/Ashland on 09/08/2011 04:24 PM -----

From: Topsale.Jim@epamail.epa.gov
To: Eric L. Hunsberger/CPD/ASCC/Ashland@Ashland
Cc: dmorgan@achd.net, Nicole M Hamilton/EHS/CORP/Ashland@Ashland, Hass.Andrew@epamail.epa.gov
Date: 09/08/2011 04:21 PM
Subject: Re: 6/16/11 Email Request - Approval of an Alternative Compliance Method for Determining Total Chlorine in Ashland's Neville Island Waste Feed Stream to its CISWI Unit.

Eric,

The Bruker Standardless method as it currently stands is not equivalent to Method 9056A. In order for the Bruker method to be equivalent it must pass all the QC as listed in section 9.0 of Method 9056A and have at least a three point calibration curve as listed in section 10.0 of Method 9056A. The QC in Method 9056A includes a successful demonstration of proficiency study and a method blank, matrix spike, duplicate (either duplicate or matrix spike duplicate) and an LCS (laboratory control sample) with each batch. It must also meet the sensitivity as listed in the Waste Analysis Plan (50-100 mg/kg). If Ashland wants to run and submit this data, our chemist is willing to review it for equivalence.

Jim T.

From: "Eric L Hunsberger" <ELHunsberger@ashland.com>
To: Jim Topsale/R3/USEPA/US@EPA
Cc: dmorgan@achd.net, "Nicole M Hamilton" <NMHamilton@ashland.com>
Date: 09/08/2011 12:37 PM
Subject: Re: 6/16/11 Email Request - Approval of an Alternative Compliance Method for Determining Total Chlorine in Ashland's Neville Island Waste Feed Stream to its CISWI Unit.

Jim,

Jian Hu, M.S., Ph.D., Senior Laboratory Scientist from EMSL Analytical, Inc., responded to your concerns about XRF as listed below.

The XRF method we used for your samples was a standardless method, which means no calibration curve was established using standards specifically made for this analysis. The method utilizes the manufacturer-installed library of line intensities obtained when the system was built (drift-corrected to date), and performs the matrix absorption correction by theoretical calculation (FP approach) when applied to the samples of different types of matrix. The method has been shown to be accurate on various kinds of certified standards analyzed over the years. The accuracy was especially high for homogeneous aqueous and glass (oxides) matrix samples. Due to the standardless nature of the used XRF method, we do not have 3 point calibration curve and did not perform matrix spikes (normally used to check for the matrix effect).

Also because of the standardless nature of the used XRF method, the reported detection limit was calculated by Bruker Spectraplus Software based on theoretical calculation and evaluation of the obtained spectra data from the sample. The reference standards we used for QC purpose were selected to verify the validation of the factory-installed standardless calibration and instrument performance. We normally do not seek to confirm the calculated detection limit for standardless methods.

For the future, if needed, we can modify our method to include QC standards with concentrations close to the calculated detection limit or low enough to match the 50-100 ppm limit as in the mentioned Ashland Waste Analysis Plan. We can also perform matrix spikes if necessary. There should not be technical barriers for adding the above procedures. It is also possible to develop a XRF method using calibration standards for the same sample matrix, so the theoretical matrix correction calculation will not be involved.

If they modified their method to include QC standards with concentrations close to the calculated detection limit or low enough to match the 50-100ppm limit, would it be possible for EPA Region 3 to consider X-ray Fluorescence Spectrometry (XRF) using the Bruker Standardless method to be an equivalent or better method for determining total chlorine content than the currently approved methods by the Air Protection Division, SW-846 Methods 5050 and 9056?

Please let me know your thoughts on this.

Thanks,

Eric

From: Topsale.Jim@epamail.epa.gov
To: Eric L Hunsberger/CPD/ASCC/Ashland@Ashland
Cc: dmorgan@achd.net
Date: 09/06/2011 02:39 PM

Subject: Re: 6/16/11 Email Request - Approval of an Alternative Compliance Method for Determining Total Chlorine in Ashland's Neville Island Waste Feed Stream to its CISWI Unit.

Eric,

After consulting again with an EPA analytical chemist, we have determined that Method 9251 is an old method from 1986. It is outdated. Although the method is sensitive enough and there is some QC, it does not specify QC limits. It is our determination that Method 9056A is a better method because it contains more complete and vigorous QC requirements. We are willing to recommend to our management the approval a request to amended the 2004 Ashland Waste Analysis Plan (WAP) to include Method 9056A, which was written in 2007.

As noted in my 8/31/11 email, a formal request for an amended WAP should be submitted to the Region 3 Air Protection Division Director, Diana Esher.

If you have any further questions, please let me know.

Jim Topsale
Environmental Engineer
EPA Region 3
(215) 814-2190

From: "Eric L Hunsberger" <ELHunsberger@ashland.com>

To: Jim Topsale/R3/USEPA/US@EPA

Cc: dmorgan@achd.net

Date: 09/01/2011 10:05 AM

Subject: Re: 6/16/11 Email Request - Approval of an Alternative Compliance Method for Determining Total Chlorine in Ashland's Neville Island Waste Feed Stream to its CISWI Unit.

Jim,

Test America has been using SW 846 Method 9251 for as far back as my records go. Do you have any problem with that method's quality control, calibration and sensitivity?

Thanks,

Eric

From: Topsale.Jim@epamail.epa.gov
To: Eric L Hunsberger/CPD/ASCC/Ashland@Ashland
Cc: dmorgan@achd.net
Date: 08/31/2011 12:39 PM
Subject: 6/16/11 Email Request - Approval of an Alternative Compliance Method for Determining Total Chlorine in Ashland's Neville Island Waste Feed Stream to its CISWI Unit.

Eric,

In consultation with an EPA analytical chemist, I have reviewed the subject request, and the supplemental information you submitted on 8/19/11. It is our opinion that X-ray Fluorescence Spectrometry (XRF) using the Bruker Standardless method is not an equivalent or better method for determining total chlorine content than the currently approved methods by the Air Protection Division, SW-846 Methods 5050 and 9056. Our biggest concerns are quality control, calibration and sensitivity. The proposed method lacks some of the QC that Method 9056 has (initial demonstration of proficiency, a 3 point calibration curve and matrix spikes). The 2004 Ashland Waste Analysis Plan lists detection limits as 50-100 mg/kg. The previously submitted table of analysis results also has units in ppm and results reported from 25-1300 ppm. The Bruker Standardless method uses reference standards in the range of 0.1 to 1%, or 1000 to 10,000 ppm. As you know, the maximum theoretical concentration (MTC) for total chlorine concentration in the waste feed stream to Ashland's CISWI unit is 345 ppm. Reported values for samples using the Bruker method were <17, <18, <19 and <40 ppm. We are concerned that standards are not being analyzed at the reporting limit and that the reference standards are being analyzed at concentrations much higher than most sample results.

If you wish to pursue this further, you can submit a formal determination request to:

Diana Esher, Director
Air Protection Division
U.S. Environmental Protection Agency
Region 3
1650 Arch Street
Philadelphia, PA 19103

If you have any questions, let me know.

Jim Topsale
Environmental Engineer
EPA Region 3
(215) 814-2190

From: "Eric L Hunsberger" <ELHunsberger@ashland.com>
To: Jim Topsale/R3/USEPA/US@EPA
Cc: Mike Gordon/R3/USEPA/US@EPA, Andrew Hass/R3/USEPA/US@EPA,
"Nicole M Hamilton" <NMHamilton@ashland.com>
Date: 08/19/2011 10:01 AM
Subject: Re: Fw: CISWI - Total Chlorine Results on Aqueous Waste
Samples, Alternative XRF Method

Jim,

The information below is one response to your questions from EMSL Analytical, Inc., the laboratory subcontracted for XRF by our normal laboratory, Test America.

For the analysis of your samples, we use XRF Standardless Analysis method with Bruker AXS Spectraplus software package. The method is a fundamental parameters (FP) method based on library lines installed by the manufacturer (Bruker) and using Bruker's fundamental parameter software package for calculation. We have a general internal SOP that we follow for all XRF standardless analyses, but the SOP is not specifically detailed for chlorine in aqueous solution.

We run QC and reference standards with each sample (or sample batch) to check the calibration and calculation. The reference standards (with chlorine% in 0.1 to 1% range) that we use are of two different types. One type is the XRF fused glass standards with certified amount of chlorine. The other is aqueous standard solution with known amount of chlorine. In addition, lab blanks (typically lab DI water) are also run using the same procedure when needed.

The specific details for chlorine in aqueous solution / the SOP are attached below.

Test American, Savannah, responded to the question (Were any QC or reference samples from a source different from the calibration standards analyzed?) regarding the method they have been using, SW-846 and 9251, as follows:

The chloride calibration curve is run with Sodium Chloride. The LCS for Total Chlorine is 2,4,6 Trichlorophenol. Our LCS for Total Chlorine is a 2nd source to the calibration curve used for analysis.

I hope this information answers your questions. If you need any more information, please let me know.

Thanks,

Eric

From: Eric L Hunsberger/CPD/ASCC/Ashland
To: Topsale.Jim@epamail.epa.gov
Cc: Gordon.Mike@epamail.epa.gov, Hass.Andrew@epamail.epa.gov
Date: 08/17/2011 11:29 AM
Subject: Re: Fw: CISWI - Total Chlorine Results on Aqueous Waste Samples, Alternative XRF Method

Jim,

I had been waiting for responses from the labs. I received their responses late last week. I will be putting them together and sending them to you by the end of this week. I should have let you know that I and the labs were working on your request.

Eric

From: Topsale.Jim@epamail.epa.gov
To: Eric L Hunsberger/CPD/ASCC/Ashland@Ashland
Cc: Gordon.Mike@epamail.epa.gov, Hass.Andrew@epamail.epa.gov
Date: 08/17/2011 11:26 AM
Subject: Fw: CISWI - Total Chlorine Results on Aqueous Waste Samples, Alternative XRF Method

Eric,

Good morning! It has been over a month since I requested certain information from Ashland, as per the 7/14 email below. I have no record of its receipt. If you need a clarification on the request, please let me know. EPA cannot prepare a response to your 6/16/11 questions until our receipt of all the requested information.

Thanks.

Jim Topsale
Environmental Engineer
EPA Region 3
(215) 814-2190

----- Forwarded by Jim Topsale/R3/USEPA/US on 08/17/2011 11:16 AM -----

From: Jim Topsale/R3/USEPA/US

To: elhunsberger@Ashland.com

Date: 07/14/2011 09:38 AM

Subject: Fw: CISWI - Total Chlorine Results on Aqueous Waste Samples, Alternative XRF Method

Eric,

Good morning! As we briefly discussed today, I'm in the process of working with our Analytical Lab folks in evaluating your subject request. In order to continue our evaluation, please provide a response the following:

- 1) A copy of the XRF method proposed; and
- 2) EPA notes a large difference in the results for the various methods compared. Were any QC or reference samples from a source different from the calibration standards analyzed? Please explain.

Jim Topsale
Environmental Engineer
EPA Region 3
(215) 814-2190

----- Forwarded by Jim Topsale/R3/USEPA/US on 07/13/2011 03:40 PM -----

From: Jim Topsale/R3/USEPA/US

To: Eric L Hunsberger/CPD/ASCC/Ashland@Ashland

Cc: dmorgan@achd.net, Nicole M Hamilton/EHS/CORP/Ashland@Ashland

Date: 07/13/2011 03:32 PM

Subject: CISWI - Total Chlorine Results on Aqueous Waste Samples, Alternative XRF Method

Eric,

I'm in the process of working with our Analytical Lab folks on evaluating your subject request. In order to continue our evaluation, please provide a response the following:

- 1) A copy of the XRF method proposed; and

2) A discussion regarding the use of QC or reference samples, if any, that were analyzed using the current method, and as proposed with the XFR method.

Thanks,

Jim Topsale
Environmental Engineer
Air Protection Division
(215) 814-2190

From: Eric L Hunsberger/CPD/ASCC/Ashland
To: topsale.james@epa.gov
Cc: dmorgan@achd.net, Nicole M Hamilton/EHS/CORP/Ashland@Ashland
Date: 06/16/2011 12:29 PM
Subject: CISWI - Total Chlorine Results on Aqueous Waste Samples

Mr. Topsale,

I spoke with Dan Morgan at the Allegheny County Health Dept. this morning about this, and he recommended I contact you. The table below lists results for total chlorine and chloride on aqueous waste samples taken under our Waste Analysis Plan (WAP) since last September. I am sending you this table to illustrate the inconsistency and unreliability of the traditional test methods used for total chlorine, as well as the consistency and reliability of the X-ray Fluorescence Spectrometry (XRF) method. Table 5-2 in section 5 of our WAP lists SW-846 Methods 5050 and 9056, or an equivalent method, as the analytical method for total chlorine.

Our normal outside/contract laboratory has been using SW-846 and 9251 since at least 2005. On many of our sampling events, we have been sending split samples to another laboratory. They have been using ASTM D808/512 for total chlorine. Since last September, our normal outside/contract laboratory has been sending a split sample of what we send them to a subcontracted laboratory for chlorine analysis by X-ray Fluorescence Spectrometry (XRF) (X-ray fluorescence elemental analysis by XRF Standardless Analysis method with Bruker AXS Spectraplus software package).

Do you agree that XRF is an equivalent (or better) method? Do we need to update the WAP to include XRF and submit it for approval? What additional information, such as a copy of the test method, do you need from us in order for us to be able to use the XRF method exclusively? The inconsistent, periodically high results have caused us to have to perform a lengthy review, investigation and resampling process each time the testing indicates we exceeded the MTC for HCl. Please note, there is no source of chlorine in the waste stream at the levels periodically being reported.

Dan Morgan told me that he will be contacting you to discuss this sometime next week. Please call me if you would like to discuss this.

Respectfully,

Eric Hunsberger

Environmental, Health and Safety Manager
Ashland Inc.
Neville Island Plant
Pittsburgh, PA 15225
412-778-6205

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deleted by Eric L Hunsberger/CPD/ASCC/Ashland]

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