



UNITED STATES ENVIRONMENTAL PROTECTION AGENCY

REGION 5  
77 WEST JACKSON BOULEVARD  
CHICAGO, IL 60604-3590

FEB 27 2020

REPLY TO THE ATTENTION OF

ECW-15J

**CERTIFIED MAIL 7019 0140 0000 0721 7573**  
**RETURN RECEIPT REQUESTED**

Mr. Robert Maciel  
Environmental Manager  
ArcelorMittal Burns Harbor, LLC  
250 West U.S. Highway 12  
Burns Harbor, IN 46304

Subject: February 5, 2020 Compliance Evaluation Inspection Report for Microbac Laboratories, Inc. as the Contract Laboratory for ArcelorMittal Burns Harbor (NPDES Permit No: IN0000175)

Dear Mr. Maciel:

Enclosed, please find a copy of the U.S. Environmental Protection Agency Inspection Report that describes and documents the activities at Microbac Laboratories, Inc. as the contract laboratory for ArcelorMittal Burns Harbor, LLC (AMBH) on February 5, 2020.

The purpose of the compliance evaluation inspection at Microbac Laboratories, Inc. was to document sample handling and analysis as the contract laboratory for AMBH's samples.

If you have any questions or concerns regarding this letter, or the inspection report, please contact Joan Rogers at (312) 886-2785 or at rogers.joan@epa.gov.

Sincerely,

A handwritten signature in blue ink, appearing to read "Dean Maraldo".

Dean Maraldo, Acting Chief  
Water Enforcement and Compliance Assurance Branch, Section 2

Enclosure

cc: Ron Misiunas, Director, Laboratory Services  
Microbac Laboratories, Inc.

Nicholas Ream, Environmental Engineer  
Indiana Department of Environmental Management

**CWA COMPLIANCE EVALUATION INSPECTION REPORT  
U.S. ENVIRONMENTAL PROTECTION AGENCY, REGION 5**

**Purpose:**

Compliance Evaluation Inspection

**Facility:**

Microbac Laboratories, Inc  
250 West 84<sup>th</sup> Drive  
Merrillville, Indiana 46410  
219-769-8378

**NPDES Permit Number:**

None

**Date of Inspection:**

February 5, 2020

**EPA Representatives:**

Joan Rogers, Environmental Scientist 312-886-2785  
[Rogers.joan@epa.gov](mailto:Rogers.joan@epa.gov)

Ken Gunter, QAC/Enforcement Officer 312-353-9076  
[Gunter.Ken@epa.gov](mailto:Gunter.Ken@epa.gov)

**State Representatives:**

Nicholas Ream, Indiana Department of Environmental Management 219-730-1691  
Wastewater Inspector  
[Nream@idem.IN.gov](mailto:Nream@idem.IN.gov)

Robert Lugar, Indiana Department of Environmental Management 317-234-6019  
Deputy Assistant Commissioner, Office of Program Support  
[RLugar@idem.IN.gov](mailto:RLugar@idem.IN.gov)

Becky Ruark, Indiana Department of Environmental Management 317-691-1909  
Wastewater Facility Inspector/Lab Proficiency Coordinator  
[BRuark@idem.IN.gov](mailto:BRuark@idem.IN.gov)

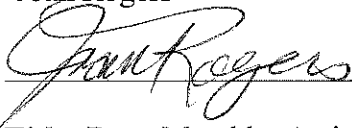
**Facility Representatives:**

Carey Gadzala, Project Manager

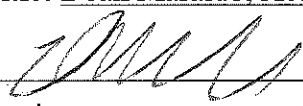
Shon Ahrendt, Operations Manager

Amy Sheehy, Quality Manager

Report Prepared by: Joan Rogers

Inspector Signature: 

Approver Name and Title: Dean Maraldo, Acting Chief, Section 2

Approver Signature: 

Approval Date: 2/27/2020

## 1. BACKGROUND

The purpose of this report is to describe and document the discussion and site inspection at the Microbac Laboratory (Microbac) in Merrillville, Indiana on February 5, 2020. This inspection was performed pursuant to Section 308(a) of the Federal Water Pollution Control Act, as amended, in order to discuss the laboratory's handling and analysis of the samples from its client, ArcelorMittal Burns Harbor.

The ArcelorMittal Burns Harbor (AMBH) facility is one of the largest fully integrated steel mills in North America, with the capacity to produce approximately 5 million tons of raw steel per year. They operate under NPDES Permit No. IN0000175, which was issued on May 27, 2016 and expires on June 30, 2021.

On August 11, 2019, AMBH had a failure of the pump system for its Blast Furnace Gas Closed Water Recycle System (BFGCWS) which required the facility to draw in Lake Michigan water to use in the Blast Furnace Gas (BFG) Air Scrubbers. Since the pumps to recycle the BFG Air Scrubber water were not functioning, the water became "once-through" water and the facility discharged many more millions of gallons than during normal operations. The once-through water was not able to be treated due to the high volume and flowed out its internal Outfall 011 and then through its final Outfall 001 to the East Arm of the Little Calumet River.

Due to this pump failure, the Burns Harbor facility had effluent exceedances of its NPDES permit. Following this event, Indiana Department of Environmental Management (IDEM) required the facility to conduct additional sampling from Outfalls 001, 011 and from AMBH's Outfall 002, an outfall that is permitted for non-contact cooling water and storm water. Results of the additional sampling from Outfall 002 showed the existence of pollutants in that wastewater that were not regulated for that outfall, including cyanide, ammonia, boron and phenols.

On several occasions, AMBH provided final reports of sample analysis that differed from the preliminary analysis provided in a draft report. AMBH stated that these samples had been reanalyzed. IDEM and EPA conducted this focused inspection at Microbac to learn the processes in place at the lab that would cause the final analysis to differ from the preliminary analysis.

## **2. SITE INSPECTION**

### **Site Entry and Opening Conference – February 5, 2020**

EPA and IDEM arrived at the laboratory at 9:15 A.M. Mr. Lugar stated the purpose of the inspection and requested to look at the analysis, including initial analysis, Level 2 and Level 3 reviews and final analysis for a couple of the samples from AMBH. Mr. Lugar stated that the inspection was not a full lab audit, but a review of the processes in relation to samples received from AMBH.

EPA and IDEM followed Ms. Gadzala, Mr. Ahrendt, and Ms. Sheehy to a conference room in an adjacent building.

### **Records Review**

#### **October 29, 2019 Sample from Outfall 002**

The discussion began with the results from October 29, 2019 for Outfall 002. The initial sample analysis for cyanide provided by AMBH in a Microbac Preliminary Report was 0.0070 mg/L. After reanalyzing the sample, the cyanide level was 0.0020 mg/L. The Microbac lab report number for the samples on this date was 19J1517.

EPA and IDEM discussed the deficiencies in the chain of custody (COC), including that the sample was taken by one person, but relinquished by someone else. The date and time that the sample was relinquished by the sampler was not on the chain of custody. Additionally, no preservative was listed or time of collection. Microbac stated that the times were listed on the bottles and the samples are brought into Microbac unpreserved in one large bottle. Microbac's receiving personnel split the sample into the separate bottles and does the preservation. Mr. Ahrendt stated that this happens within one hour of delivery, but the times are not recorded. Ms. Gadzala stated that the practice of AMBH bringing in the large bottle and Microbac splitting it into the separate bottles is historic but not common amongst their clients. Mr. Ahrendt stated that typically, the client takes their samples in pre-preserved bottles provided by the lab.

Mr. Lugar then asked to see the analysis and Levels 2 and 3 reviews for this sample in order to understand why this sample was re-analyzed. During the review, there was nothing in the first analysis or the Level 2 Peer Review that indicated that the sample needed to be re-run. Ms. Gadzala stated that sometimes the client will request a sample to be reanalyzed and that was what happened for this sample. On November 8, 2019, Mr. Gary Amendola, of Amendola Engineering, Inc., instructed Ms. Joyce Casillas, from Environmental Process Technologies, Inc., to ask for the reanalysis.

During the discussion, EPA and IDEM learned that it was not Microbac's practice at the time to put in a reanalysis request form if the report was still in draft form. It also was not their practice to put the information on who requested the reanalysis in the final report.

Regarding this sample, re-distillation was only done in duplicate and not in triplicate, as is the usual practice. There were matrix spike errors, but they were not valid for this

sample because Microbac did not use the AMBH sample to run the matrix spike. The reanalysis result was 0.002 mg/L and because there was no laboratory determined reason to reanalyze the sample, both values were listed on the final report. Microbac confirmed that any reanalysis is run on the retained sample at the laboratory.

### **December 21, 2019 Sample from Outfall 011**

EPA and IDEM then requested to review the analysis and reanalysis for the sample taken on December 21, 2019 for Outfall 011. The lab report number was 19L1117 for this day's samples. The preliminary analysis was 0.052 mg/L for total cyanide. After calculating for mass concentration, as per the permit limit, AMBH issued a Noncompliance 24-hour Notification Report to IDEM. The mass of cyanide was 31 lbs/day, over the permit limit of 21 lb/day. After reanalyzing the sample, the total cyanide level was non-detect.

Ms. Sheehy stated that although there was a detection of cyanide in the method blank, it was between the Method Detection Limit (MDL) and the reporting limit (RL). There were also matrix spike errors, but again, the matrix spike was not performed using AMBH sample, so any errors do not relate to the AMBH sample. Microbac's typical procedures would not have required reanalysis.

Ms. Gadzala stated that once she emailed the 0.52 mg/L result to "the group", she received an email request from Ms. Teri Kirk to reanalyze the sample. Ms. Gadzala stated that "the group" consisted of AMBH Company representatives, Mr. Rob Maciel, Ms. Teri Kirk, Ms. Morgan Swanson, and Mr. Gary Amendola. Microbac created a re-evaluation form even though the report was still in draft form because Microbac had changed their practice to create one every time.

Mr. Ahrendt explained that on this day, there was a glassware issue. That morning, Microbac received solid samples from another client. One of the samples was found to have high cyanide levels after they were analyzed. The glassware for that client was cleaned as per the "regular" cleaning that is done between samples and the glassware was put back into use. (Typically, if a sample has high cyanide, the glassware would be cleaned in a more robust way, with soaking for several days in an acid bath.) Microbac did not know of the high cyanide detection in the other client's sample until the AMBH sample was already being analyzed.

Mr. Ahrendt stated that only one of the samples for the other client was higher than "typical." He then explained that the glassware for the other client "must have been" the set that was used for the AMBH sample because of the high detect for total cyanide in AMBH's Outfall 011 sample.

Although Microbac did not have a way to record which glassware was used for the other client, they assumed that the glassware for the first client contaminated the AMBH sample. When asked if he assumed that the glassware for the high cyanide sample from the first client was the same set used for the AMBH Outfall 011 sample and that the regular cleaning was ineffective, Mr. Ahrendt stated "yes."

The final report for the AMBH sample did not reflect the reason for the reanalysis, only that there was a false positive. The final report also did not reflect who requested the reanalysis. The original analysis was completed on December 23, 2019 and the reanalysis was completed one week later, on December 30, 2019. The lab result from the sub-contracted cyanide Method 1677 analysis showed  $< 0.91 \mu\text{g/L}$ .

Microbac has begun to label the glassware since this occurrence. All four pieces that are used together for cyanide analysis will be kept together as a unit and each labeled with the letter of that unit. Microbac's corporate office is looking into using disposable single use plasticware.

### **January 17, 2019 Microbac Letter to AMBH**

EPA and IDEM then asked to discuss the letter from Mr. Ron Misiunas, Microbac Laboratory Director, to Mr. Cary Mathias of AMBH. Mr. Ahrendt stated that the lab realized that it did not have an SOP for reanalysis. He stated that Mr. Misiunas would be addressing that when he got back from vacation.

Mr. Lugar suggested that IDEM would like the lab to consider putting the information on why a sample was reanalyzed into the final report. Mr. Lugar also suggested that the chain of custody forms be improved with AMBH.

The letter from Microbac to AMBH discusses the reanalysis of two days' of samples, December 24, 2019 and December 25, 2019. The lab report numbers for those dates are 19L1184 and 19L1986, respectively. On December 24, 2019, the preliminary analysis of total cyanide for Outfalls 001, 002, and 011 was .0076 mg/L, .0052 mg/L, and .0020 mg/L respectively. After reanalysis, all three results for cyanide were non-detect. For the December 25, 2019 samples, the preliminary total cyanide results were .0038 mg/L, .0040 mg/L, and .0059 mg/L for Outfalls 001, 002, and 011, respectively. Again, the results for cyanide were non-detect after reanalysis.

The letter from Mr. Misiunas stated that after both the 1<sup>st</sup> Level Review and 2<sup>nd</sup> Level Review, all batch related QC parameters were acceptable. During the 3<sup>rd</sup> Level Review, the Project Manager recognized that "the reported data, spanning multiple locations and sampling days involving sample points not connected with each other, presented nearly uniform cyanide results" and requested the reanalysis.

According to Mr. Ahrendt, due to staffing issues around the holidays, and some poor decisions by those at the lab, no duplicates were done on reanalysis #1. After reanalysis #1 was complete, the December 24, 2019 Outfall 002 and the December 25, 2019 Outfall 011 samples still had detections of cyanide. Reanalysis #2 was done and following that reanalysis, the levels of cyanide were 0.002 mg/L or non-detect. The letter from Mr. Misiunas described a finding of a baseline shift early in the analysis sequence, which may have directly contributed to a low-level bias of approximately 0.002 mg/L.

Microbac has initiated a Corrective Action Report (CAR) to address the formal reanalysis policy. Mr. Lugar requested to see any new policy related to this CAR when completed.

Mr. Gunter asked if a reanalysis/holding time study was ever done for cyanide. Mr. Ahrendt stated that Microbac had never done one. Mr. Gunter stated that collecting cyanide samples using an auto sampler is not an accepted practice. A review of the AMBH permit allows the use of composite sampling for cyanide. Mr. Gunter also expressed concern over the amount of time that elapsed between collection and preservation as cyanide and ammonia/nitrogen are unstable pollutants and degradation can occur. Mr. Gunter also noted that some of the chain of custody forms appeared to show that analysis was completed *before* the sample was prepped. Mr. Ahrendt stated that he would look into that documentation.

EPA and IEPA concluded the records review and asked to have a tour of the laboratory. Mr. Ahrendt stated that he would conduct the tour.

### **Walkthrough of the Laboratory**

At 12:01 P.M. EPA and IDEM followed Mr. Ahrendt through the laboratory. During the tour, Mr. Gunter interviewed two of the analysts. EPA and IDEM observed the new labeling of the glassware units and the cart with the AMBH daily sample being prepped for analysis.

EPA and IDEM inspectors exited the laboratory at approximately 1:30 P.M.

### **3. LIST OF DOCUMENTS RECEIVED FROM FACILITY**

No documents were received from the laboratory.

### **4. AREAS OF CONCERN**

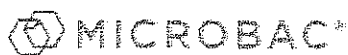
- A. Chain of Custody forms for samples received by AMBH are inaccurate and have omissions. For example:
- a. COC Records 153828 and 1533948 are associated with AMBH samples taken on 12/25/19 and 12/24/19 and were incorrectly completed, omitting the duration (time) composites were collected.
  - b. Samples were not appropriately identified as being for compliance monitoring purposes.
  - c. COC does not document sample preservation as required in 40CFR 136 Table II.
  - d. The pH of composite samples is not noted on the COC form.
  - e. For the COC for the October 29, 2019 samples, the samples were relinquished by Warren Howard, but sampling was conducted by someone else who did not relinquish the samples to Warren Howard.
- B. Composite sampling with an automatic sampler for cyanide, phenols and other parameters is not an accepted practice. The NPDES Compliance Inspection Manual (Chapter 5 page 4) provides that "...Some parameters that are **not** to be collected by automatic samplers, but must be hand collected are dissolved oxygen, total residual chlorine, oil and grease, coliforms, purgeable organics, sulfides, **cyanide**, and total phenols".

- C. Microbac staff report that composite samples are split into aliquots at the lab and appropriate preservatives were then added prior to cyanide and ammonia/nitrogen analysis.
- D. Microbac does not document the pH of the composite sample prior to splitting and after preservation.
- E. Microbac Laboratory Data Reports (19L1184 and 19L1186) show that the sodium hydroxide preservatives were added to cyanide samples from **30 to 58 hours** after collection of composites (see attached hold time reports). Proper preservation and holding times are essential to ensure sample integrity. Unstable pollutants require immediate (e.g., within 15 minutes) preservation and/or analysis. The general acceptable practice for cyanide samples is to add preservatives to sample bottles prior to or immediately following sample collection.
- F. Microbac Laboratory Data Reports (19L1184 and 19L1186) appear to show that some cyanide samples were analyzed from 5 to 8 hours prior to the time noted for sample preparation (see attached certificate of analysis reports).
- G. Microbac Laboratory Data Reports (19L1184 and 19L1186) case narratives relate that preliminary reports of cyanide results were false positives and therefore were not reported. The narrative does not provide the lab's rationale for suspicion of false positive results and likewise does not define the criteria used to confirm that the initial results were false positives.
- H. Microbac did not have an SOP for reanalysis of samples.
- I. Microbac's report format for reports where Microbac is waiting for additional analysis from the sub-contracted laboratory states that the report is a "Preliminary Report: Data Subject to Change" when, in practice, according to Microbac, the report is a "Partial" report.
- J. During reanalysis of AMBH samples, Microbac does not regularly choose AMBH samples for the matrix spike. Microbac chooses a sample randomly. Any matrix spike errors resulting from the spiked sample cannot be attributed to the validity of the AMBH sample.
- K. Frequently, there are detections of pollutants, including cyanide and ammonia, in Microbac's blanks.

##### **5. LIST OF ATTACHMENTS**

- A) Microbac Certificate of Analysis from Laboratory Report Number: 19L1186.
- B) Microbac Hold Time from Laboratory Report Number: 19L1184.





Laboratory Report Number: 19L1186

CERTIFICATE OF ANALYSIS  
FORM I

Client Project ID: NPDES Parameters 12/25/19

Microbac Laboratories, Inc. - Chicagoland

Wet Chemistry

Client ID: 001-Composite	Collection Date: 12/25/19 00:00
Laboratory ID: 19L1186-01RE1	Prep Date: 12/27/19 09:35
Matrix: Aqueous	Analyzed: 12/27/19 03:33
Batch / Sequence: B150063 / S050382	Analytical Method: SM 4500-CN C/E-1999
Instrument: Lachat-4	Units: mg/L
Analyst: ABG	Dilution: 1
Calibration: UNASSIGNED	File ID: OM_12-27-2019_03-19-43PM.csv-006

Analyte	CAS Number	Result	MDL	RL	Flag	Qualifier
Cyanide, Total	57-12-5	ND	0.0020	0.0050	U	

**Notes and Definitions**

MDL: Method Detection Limit

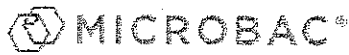
RL: Reporting Limit

mg/L: Milligrams per Liter

U: The analyte was analyzed for but was not detected above the reported quantitation limit. The quantitation limit has been adjusted for any dilution or concentration of the sample.

Microbac Laboratories, Inc.

250 West 84th Drive | Merrillville, IN 46410 | 219.769.8378 | www.microbac.com



Laboratory Report Number: 19L1184  
Client Project ID: NPDES Parameters 12/24/19  
Microbac Laboratories, Inc. - Chicagoland  
Wet Chemistry

CERTIFICATE OF ANALYSIS  
FORM I

Client ID: 011-Composite	Collection Date: 12/24/19 00:00
Laboratory ID: 19L1184-03RE1	Prep Date: 12/27/19 09:35
Matrix: Aqueous	Analyzed: 12/27/19 03:31
Batch / Sequence: B150063 / S050382	Analytical Method: SM 4500-CN C/E-1999
Instrument: Lachat-4	Calibration: UNASSIGNED
Analyst: ABG	Units: mg/L
	Dilution: 1
	File ID: OM_12-27-2019_03-19-43PM.csv-007

Analyte	CAS Number	Result	MDL	RL	Flag	Qualifier
Cyanide, Total	57-12-5	ND	0.0020	0.0050	U	

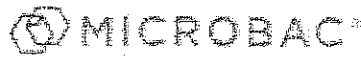
Notes and Definitions

MDL: Method Detection Limit

RL: Reporting Limit

mg/L: Milligrams per Liter

U: The analyte was analyzed for but was not detected above the reported quantitation limit. The quantitation limit has been adjusted for any dilution or concentration of the sample.



Specific Method: SM 4500-CN C/E-1999

Hold Time

Laboratory Report Number: 19L1184

Matrix: Aqueous

Client Project ID: NPDES Parameters 12/24/19

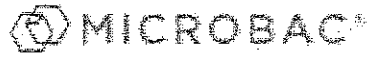
Microbac Laboratories, Inc. - Chicagoland

Laboratory ID	Date Collected	Date Received	Date Prepared	Days to Prep	Max Days to Prep	Date Analyzed	Days to Analysis	Max Days to Analysis	Q
001-Composite	12/24/19 00:00	12/26/19 10:35	12/27/19 09:35	3.00	14.00	12/27/19 15:28	0.25		
002-Composite	12/24/19 00:00	12/26/19 10:35	12/30/19 11:16	6.00	14.00	12/30/19 15:15	0.17		
011-Composite	12/24/19 00:00	12/26/19 10:35	12/27/19 09:35	3.00	14.00	12/27/19 15:31	0.25		

\* - Holding time exceeded.

Microbac Laboratories, Inc.

250 West 84th Drive | Merrillville, IN 46410 | 219.769.8378 | [www.microbac.com](http://www.microbac.com)



Specific Method: SM 4500-CN C/E-1999

Hold Time

Laboratory Report Number: 19L1186

Matrix: Aqueous

Client Project ID: NPDES Parameters 12/25/19

Microbac Laboratories, Inc. - Chicagoland

Laboratory ID	Date Collected	Date Received	Date Prepared	Days to Prep	Max Days to Prep	Date Analyzed	Days to Analysis	Max Days to Analysis	Q
001-Composite	12/25/19 00:00	12/26/19 10:35	12/27/19 09:35	2.00	14.00	12/27/19 15:33	0.25		
002-Composite	12/25/19 00:00	12/26/19 10:35	12/27/19 09:35	2.00	14.00	12/27/19 15:34	0.25		
011-Composite	12/25/19 00:00	12/26/19 10:35	12/27/19 09:35	2.00	14.00	12/27/19 16:01	0.27		

\* - Holding time exceeded.

Microbac Laboratories, Inc.

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