

2.0 Summary of Results

AST conducted NESHAP and state permit compliance demonstration testing at the ASA facility in Coldwater, MI on September 11-12, 2013. Testing consisted of determining the emission rates of PM, PM10, D/F, NOx, THC, HCl and HF from multiple sources at the facility.

Tables 2-1 and 2-2 provide a summary of the testing results with comparisons to the applicable NESHAP and/or state permit limits. Table 2-3 provides a summary of the process operating and control system data collected during testing. Any difference between the summary results listed in the following tables and the detailed results contained in Appendix B is due to rounding for presentation.

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Table 2-1
Summary of Results – NESHAP

Run Number	Run 1	Run 3	Run 4	Average ¹
Date	9/11/13	9/12/13	9/12/13	--
Furnace No. 7				
PM Emission Factor, lb/ton	0.013	0.016	0.010	0.013
NESHAP Emission Limit, lb/ton	--	--	--	0.40
Percent of Limit, %	--	--	--	3
HCl Emission Factor, lb/ton	0.0008 ²	0.010	0.051	0.021
NESHAP Emission Limit, lb/ton	--	--	--	0.40
Percent of Limit, %	--	--	--	5
D/F Emission Factor, ug TEQ/MG ³	0.87	0.52	1.4	0.92
NESHAP Limit, ug TEQ/MG	--	--	--	15.0
Percent of Limit, %	--	--	--	6
Furnace No. 8				
PM Emission Factor, lb/ton	0.028	0.020	0.013	0.021
NESHAP Emission Limit, lb/ton	--	--	--	0.40
Percent of Limit, %	--	--	--	5
HCl Emission Factor, lb/ton	0.0017 ²	0.013	0.065	0.027
NESHAP Emission Limit, lb/ton	--	--	--	0.40
Percent of Limit, %	--	--	--	7
D/F Emission Factor, ug TEQ/MG ³	1.9	0.68	1.7	1.4
NESHAP Limit, ug TEQ/MG	--	--	--	15.0
Percent of Limit, %	--	--	--	9

¹ Run 2 was voided – See Section 1.5.

² At least one (1) fraction was “U” flagged – undetected at the limit of quantitation. See Appendix C for all laboratory reporting notes.

³ D/F TEQ values were calculated using 1989 NATO TEFs.

Table 2-2
 Summary of Results – State Permit

Run Number	Run 1	Run 3	Run 4	Average ¹
Date	9/11/13	9/12/13	9/12/13	--
Furnace No.'s 7/8 (BH 1)				
PM Emission Factor, lb/ton	0.009	0.009	0.006	0.008
Permit Limit, lb/ton	--	--	--	0.023
Percent of Limit, %	--	--	--	34
PM10 Emission Factor, lb/ton ²	0.009	0.012	0.018	0.013
Permit Limit, lb/ton	--	--	--	0.036
Percent of Limit, %	--	--	--	36
HCl Emission Factor, lb/ton	0.00055 ³	0.0056	0.029	0.012
Permit Limit, lb/ton	--	--	--	0.001
Percent of Limit, %	--	--	--	> 100
Flue No. 7				
PM Emission Factor, lb/ton	0.057	0.776	0.275	0.370
Permit Limit, lb/ton	--	--	--	0.580
Percent of Limit, %	--	--	--	64
PM10 Emission Factor, lb/ton ²	0.131	1.167	1.457	0.918
Permit Limit, lb/ton	--	--	--	0.489
Percent of Limit, %	--	--	--	> 100
NOx Emission Factor, lb/ton	0.101	0.176	0.180	0.152
Permit Limit, lb/ton	--	--	--	0.231
Percent of Limit, %	--	--	--	66
Flue No. 8				
PM Emission Factor, lb/ton	0.431	1.28	0.667	0.794
Permit Limit, lb/ton	--	--	--	0.909
Percent of Limit, %	--	--	--	87
PM10 Emission Factor, lb/ton ²	2.204	2.630	2.213	2.349
Permit Limit, lb/ton	--	--	--	2.234
Percent of Limit, %	--	--	--	> 100
HCl Emission Factor, lb/ton	1.512	1.284	1.494	1.430
Permit Limit, lb/ton	--	--	--	1.552
Percent of Limit, %	--	--	--	92
HF Emission Factor, lb/ton	0.0092	0.029	0.016	0.018
Permit Limit, lb/ton	--	--	--	0.269
Percent of Limit, %	--	--	--	7
VOC Emission Factor, lb/ton	0.025	0.020	0.020	0.021
Permit Limit, lb/ton	--	--	--	0.049
Percent of Limit, %	--	--	--	43

¹ Run 2 was voided – See Section 1.5.

² PM10 is the sum of the filterable PM10 and CPM.

³ At least one (1) fraction was "U" flagged – undetected at the limit of quantitation. See Appendix C for all laboratory reporting notes.

Table 2-3
Process/Control System Data

Run Number	Run 1	Run 3	Run 4	Average *
Date	9/11/13	9/12/13	9/12/13	--
Furnace No. 7 Feed Rate, lb/hr	17,524	10,549	10,853	12,975
Furnace No. 8 Feed Rate, lb/hr	8,241	8,147	8,566	8,318
Furnace No. 7 Flux Percent, %	5.7	3.5	3.3	4.2
Furnace No. 8 Flux Percent, %	2.7	3.5	3.7	3.3
Furnace No. 7 Cl ₂ Feed, lb	147	525	600	424
Furnace No. 8 Cl ₂ Feed, lb	441	350	330	374
Lime Injection Rate, lb/hr	126	124	126	125
Baghouse Inlet Temperature, °F	121	116	128	122

* Run 2 was voided – See Section 1.5.

Testing Methodology

3.0 Testing Methodology

The emission testing program was conducted in accordance with the U.S. EPA Reference Test Methods listed in Table 3-1. Method descriptions are provided below while quality assurance/quality control data is provided in Appendix D.

Table 3-1
Source Testing Methodology

Parameter	U.S. EPA Reference Test Methods	Notes/Remarks
Volumetric Flow Rate (VFR)	1 & 2	Full Velocity Traverses
Oxygen/Carbon Dioxide (O ₂ /CO ₂)	3/3A	Integrated Bag / Instrumental Analysis
Moisture Content (BWS)	4	Volumetric / Gravimetric Analysis
Particulate Matter (PM)	5	Isokinetic Sampling
Particulate Matter (PM)/ Condensable PM (CPM)	5/202	Isokinetic Sampling
Nitrogen Oxides (NO _x)	7E	Instrumental Analysis
Dioxin/Furan (D/F)	23/ALT-034	Isokinetic Sampling
Total Hydrocarbons (THC)/Methane (CH ₄)	25A	Instrumental Analysis
Hydrogen Chloride (HCl)/Hydrogen Fluoride (HF)	26A	Isokinetic Sampling
Particulate Matter less 10 microns (PM ₁₀)	201A/202	Constant Rate Sampling

3.1 U.S. EPA Reference Test Methods 1 & 2 -- Volumetric Flow Rate

The sampling location and number of traverse (sampling) points were selected in accordance with U.S. EPA Reference Test Method 1. A full velocity traverse was conducted in accordance with U.S. EPA Reference Test Method 2 to determine the average stack gas velocity pressure, static pressure and temperature. The velocity and static pressure measurement system consisted of an S-type pitot tube and inclined manometer while the stack gas temperature was measured with a K-type thermocouple and pyrometer.

3.2 U.S. EPA Reference Test Method 3/3A – Oxygen and Carbon Dioxide

The oxygen and carbon dioxide concentrations were determined in accordance with U.S. EPA Reference Test Method 3. One (1) integrated Tedlar bag sample was collected during each test run. The bag samples were analyzed on site with a California Analytical Instruments Model 200P O₂/CO₂ analyzer. The remaining stack gas constituent was assumed to be nitrogen for the stack gas molecular weight determination. The quality control measures are described in Section 3.11.

3.3 U.S. EPA Reference Test Method 4 – Moisture Content

The stack gas moisture content was determined in accordance with U.S. EPA Reference Test Method 4. The gas conditioning train consisted of a series of chilled impingers. The impinger contents were pre and post-measured to determine the amount of moisture condensed during each test run.

3.4 U.S. EPA Reference Test Method 5 – Particulate Matter

The particulate matter testing was conducted in accordance with U.S. EPA Reference Test Method 5. The complete sampling system consisted of a glass or Teflon coated nozzle, heated glass-lined probe, pre-weighed Teflon filter, gas conditioning train, pump and calibrated dry gas meter. The gas conditioning train is described in Section 3.9.

Following the completion of each test run, the sampling train was leak checked at a vacuum pressure greater than or equal to the highest vacuum pressure observed during the run. The probe and nozzle were rinsed three (3) times with acetone to remove any adhering particulate matter. The front half of the filter holder was rinsed three (3) times with acetone, and these rinses were added to the probe/nozzle rinse. The pre-weighed filter was carefully removed and placed in its sample container. All containers were sealed, labeled and liquid levels marked for transport to AST's laboratory in Decatur, Alabama.

The mass of particulate matter collected in the probe and nozzle was determined by evaporating the acetone rinse in a pre-weighed glass beaker and then weighing the residue until a constant weight was obtained. The filter loading was determined by subtracting the initial constant filter weight from the final constant weight. The total particulate loading, which was used to calculate the concentration of particulate matter in the exhaust gas, was determined by adding these two (2) weights. All weight measurements were made on the same balance (accurate to 0.1 mg).

3.5 U.S. EPA Reference Test Methods 5/202 – Particulate Matter

The particulate matter testing was conducted in accordance with U.S. EPA Reference Test Methods 5 and 202. The complete sampling system consisted of a glass nozzle, heated glass-lined probe, pre-weighed quartz filter, gas conditioning train, pump and calibrated dry gas meter. The gas conditioning train consisted of five (5) chilled impingers. The first, second and fourth impingers were initially empty, the third contained 100 milliliters (mL) of de-ionized water and the last impinger contained approximately 200-300 grams of silica gel. An un-weighed 90 mm Teflon filter was placed between the second and third impingers.

Following the completion of each test run, the sampling train was leak checked at a vacuum pressure greater than or equal to the highest vacuum pressure observed during the run. The nitrogen purge was omitted from the sample clean due to minimal condensate collected in the dry impingers.

The contents of the impingers were measured gravimetrically to determine the moisture gain. The contents of impinger 1 and 2 were recovered in Container 1. Impingers 1 and 2, the coil condenser and all connecting glassware were rinsed with water and then rinsed with acetone and hexane. The water rinses were added to Container 1 while the solvent rinses were recovered in Container 2. The un-heated Teflon filter was removed from the filter holder and placed in Container 3. The front half of the filter holder was rinsed with water and then with acetone and hexane. The water rinse was added to Container 1 while the solvent rinses were added to Container 2. All containers were sealed, labeled and liquid levels marked for transport to AST's laboratory in Decatur, Alabama for condensable particulate matter analysis.

The pre-weighed quartz filter was carefully removed and placed in Container 4. The probe, nozzle and front half of the filter holder were rinsed three (3) times with acetone to remove any adhering particulate matter, and these rinses were recovered in Container 5. All containers were sealed, labeled and liquid levels marked for transport to AST's laboratory in Decatur, Alabama for filterable particulate matter analysis.

The mass of filterable particulate matter collected in the probe and nozzle was determined by evaporating the water rinse in a pre-weighed glass beaker and then weighing the residue until a constant weight was obtained. The filter loading was determined by subtracting the initial constant filter weight from the final constant weight. The filterable particulate loading was determined by adding these two (2) weights. All weight measurements were performed on the same balance (accurate to 0.1 mg). The total particulate loading was determined by adding the filterable particulate mass and the condensable particulate mass.

3.6 U.S. EPA Reference Test Method 7E -- Nitrogen Oxides

The concentration of nitrogen oxides was determined in accordance with U.S. EPA Reference Test Method 7E. Data was collected online and reported in one-minute averages. The sampling system consisted of a heated stainless steel probe, heated Teflon sample line(s), gas conditioning system and California Analytical Instruments Model 400CLD analyzer. The gas conditioning system was a non-contact condenser to remove moisture from the stack gas. The quality control measures are described in Section 3.12.

3.7 U.S. EPA Reference Test Method 23/Alternative Method 034 – Dioxin/Furan

The dioxin and furan concentrations were determined in accordance with EPA Reference Method 23 with guidance from Alternative Method 034. All glassware leading to the XAD adsorbing resin was cleaned at AST's laboratory before mobilizing to the site. Glassware cleaning consisted of washing with warm soapy water and rinsing with distilled water and acetone. Once the glassware was dry, the open ends were sealed with Teflon tape. SGS Analytical Perspectives in Wilmington, North Carolina provided the pre-cleaned filters and pre-cleaned, packed and spiked XAD resin traps.

The impinger train was assembled in the sample recovery area. The first impinger (shortened stem) was empty and used for a knockout impinger. The next two (2) impingers were standard Greenberg-Smith impingers with each containing 100 mL of high performance liquid chromatography grade water. The fourth impinger was empty while the fifth impinger was charged with approximately 300 grams of indicating silica gel. The pre-cleaned glass fiber filter was placed in a glass filter holder with a Teflon-coated filter support and connected to the condenser coil. All open ends of the sampling train were sealed with Teflon tape prior to complete assembly at the sampling location.

The complete sampling system consisted of a glass nozzle, heated glass-lined probe, glass filter holder with pre-cleaned glass-fiber filter, condenser coil, XAD resin trap, gas conditioning train, pump and calibrated dry gas meter. The probe and filter box temperatures were maintained at approximately 250°F. The sorbent module resin and impinger temperatures were maintained at or below 68°F throughout the testing.

Following the completion of each test run, the sampling train was leak checked at a vacuum pressure greater than or equal to the highest vacuum pressure observed during the run. The filter was removed from the filter holder and placed in sample Container 1. The XAD sorbent module was sealed on both ends and placed on ice. The nozzle, probe liner, filter holder, condenser and all connecting glassware were triple-rinsed with acetone, and these rinses were recovered in sample Container 2. All glassware cleaned for sample Container 2, except the condenser, was also triple-rinsed with toluene. Three (3) 5-minute soaks with toluene were conducted on the condenser. The toluene rinses were recovered in sample Container 3.

3.8 U.S. EPA Reference Test Method 25A – Total Hydrocarbons & Methane

The concentration of total hydrocarbons was determined in accordance with U.S. EPA Reference Test Method 25A. Data was collected online and reported in one-minute averages. The sampling system consisted of an in-stack filter, heated stainless steel probe, heated Teflon sample line(s) and CAI Model 300 HFID THC analyzer. The quality control measures are described in Section 3.13.

Methane was determined by direct extraction with a Method 25A sampling system concurrently with each Method 25A test run. A CAI Model 300M HFID operating in non-methane mode was used to quantify the concentration of methane in the stack gas. The 300M HFID analyzer has current methane and ethane penetration factors as required by NSPS Subpart JJJJ. The methane concentration was subtracted from the THC concentration to determine the source non-methane THC concentration.

3.9 U.S. EPA Reference Test Method 26A - Hydrogen Chloride/Hydrogen Fluoride

The hydrogen chloride and hydrogen fluoride concentrations were determined in accordance with U.S. EPA Reference Test Method 26A and in combination with the Method 5 sample train. The complete sampling system consisted of a glass or Teflon nozzle, heated glass-lined probe, Teflon filter, gas conditioning train, pump and calibrated dry gas meter. The gas conditioning train consisted of four (4) impingers contained in an ice/water bath. The first and second impingers contained 100 mL of 0.1 N H₂SO₄, the third was empty and the last impinger contained approximately 200 grams of silica gel. The probe and filter box temperatures were maintained above 250°F, and the impinger temperature was maintained below 68°F throughout the testing.

Following the completion of the test run, the sampling train was leak checked at a vacuum pressure greater than or equal to the highest vacuum pressure observed during the run. After determining the amount of condensed moisture in each impinger, the contents of the first, second and third impingers were placed into a sample container. The back-half of the filter holder, first, second and third impingers and all glassware leading to the outlet of the third impinger were triple-rinsed with DI water, and these rinses were recovered in the sample container. The samples were sealed, labeled and liquid levels marked for transport to Maxxam Analytics, Inc. of Mississauga, Ontario, Canada.

3.10 U.S. EPA Reference Test Methods 201A/202 – Particulate Matter

The particulate matter < 10 microns was determined in accordance with U.S. EPA Reference Methods 201A and 202. The complete sampling system consisted of a stainless steel nozzle, in-stack cyclone, in-stack 47-mm quartz filter, heated stainless-lined probe, gas conditioning train, pump and calibrated dry gas meter. For the reverb flue sampling train, an out-of-stack 83-mm quartz filter was used instead of the in-stack 47-mm filter. The gas conditioning train consisted of a condenser and five (5) impingers. The first and second impingers were initially empty, the third contained 100 milliliters (mL) of deionized water, the fourth was initially empty and the fifth impinger contained approximately 200-300 grams of silica gel. An un-weighed 90-mm Teflon filter was placed between the second and third impinger.

Following the completion of each test run, the sampling train was leak checked at a vacuum pressure greater than or equal to the highest vacuum pressure observed during the run. Since there was no visible water in the first two (2) impingers, a nitrogen purge was not conducted.

The contents of impingers 1 and 2 were recovered in Container 1. Impingers 1 and 2, the coil condenser and all connecting glassware were rinsed with water and then rinsed with acetone and hexane. The water rinses were added to Container 1 while the solvent rinses were recovered in Container 2. The un-heated Teflon filter was removed from the filter holder and placed in Container 3. The front half of the condensable filter holder was rinsed with water and then with acetone and hexane. The water rinse was added to Container 1 while the solvent rinses were added to Container 2. All containers were sealed, labeled and liquid levels marked for transport to AST's laboratory in Decatur, AL for condensable particulate matter analysis.

The pre-weighed filter was carefully removed and placed in Container 4. The back-half of the PM10 cyclone and the connecting stainless tubing were rinsed three (3) times with acetone, and these rinses were recovered in Container 5. All containers were sealed, labeled and liquid levels marked for transport to AST's laboratory in Decatur, Alabama for filterable particulate matter analysis.

To determine the condensable particulate matter mass, the filter from Container 3 was placed in an extraction tube, rinsed with water then acetone and hexane and sonicated. The water rinse was added to Container 1 while the solvent rinse was added to Container 2. The contents of Containers 1 was added to a separatory funnel and mixed with hexane. Hexanes extractions were conducted, and the organic fraction was drained from the funnel and added to Container 2. After the organic and inorganic fractions were separated, the mass of each fraction was determined by evaporating the applicable solvents in pre-weighed beakers and then weighing the residue in each beaker until a constant weight was obtained. All weight measurements were performed on the same balance (accurate to 0.1 mg)

The mass of filterable PM10 was determined by evaporating the acetone rinses from Container 5 in separate pre-weighed dishes and then weighing the residue until constant weights was obtained. The filter loading was determined by subtracting the initial constant filter weight from the final constant weight. All weight measurements were performed on the same balance (accurate to 0.1 mg).

3.11 Quality Assurance/Quality Control - U.S. EPA Reference Test Method 3A

Cylinder calibration gases were supplied by AirGas – South or NexAir which met Protocol 1 (+/- 2%) standards.

Low Level gases were introduced directly to analyzer. After adjusting the analyzer to the Low Level gas concentration and once the analyzer reading was stable, the analyzer reading was recorded. This process was repeated for the High Level gas. Next, Mid Level gases were introduced directly to analyzer and reading was recorded. All recording readings were within +/- 2 percent of the Calibration Span.

All data was reviewed by the Field Team Leader before leaving the facility. Once arriving at AST's office, all written and electronic data was relinquished to the report coordinator and then a final review was performed by the Project Manager.

3.12 Quality Assurance/Quality Control – U.S. EPA Reference Test Method 7E

U.S. EPA Protocol 1 Calibration Gases – Cylinder calibration gases were supplied by a certified supplier which meet Protocol 1 (+/- 2%) standards. Copies of all calibration gas certificates can be found in the Quality Assurance/Quality Control Appendix.

3.8 U.S. EPA Reference Test Method 25A – Total Hydrocarbons & Methane

The concentration of total hydrocarbons was determined in accordance with U.S. EPA Reference Test Method 25A. Data was collected online and reported in one-minute averages. The sampling system consisted of an in-stack filter, heated stainless steel probe, heated Teflon sample line(s) and CAI Model 300 HFID THC analyzer. The quality control measures are described in Section 3.13.

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3.9 U.S. EPA Reference Test Method 26A - Hydrogen Chloride/Hydrogen Fluoride

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Following the completion of the test run, the sampling train was leak checked at a vacuum pressure greater than or equal to the highest vacuum pressure observed during the run. After determining the amount of condensed moisture in each impinger, the contents of the first, second and third impingers were placed into a sample container. The back-half of the filter holder, first, second and third impingers and all glassware leading to the outlet of the third impinger were triple-rinsed with DI water, and these rinses were recovered in the sample container. The samples were sealed, labeled and liquid levels marked for transport to Maxxam Analytics, Inc. of Mississauga, Ontario, Canada.

3.10 U.S. EPA Reference Test Methods 201A/202 – Particulate Matter

The particulate matter < 10 microns was determined in accordance with U.S. EPA Reference Methods 201A and 202. The complete sampling system consisted of a stainless steel nozzle, in-stack cyclone, in-stack 47-mm quartz filter, heated stainless-lined probe, gas conditioning train, pump and calibrated dry gas meter. For the reverb flue sampling train, an out-of-stack 83-mm quartz filter was used instead of the in-stack 47-mm filter. The gas conditioning train consisted of a condenser and five (5) impingers. The first and second impingers were initially empty, the third contained 100 milliliters (mL) of deionized water, the fourth was initially empty and the fifth impinger contained approximately 200-300 grams of silica gel. An un-weighed 90-mm Teflon filter was placed between the second and third impinger.

Following the completion of each test run, the sampling train was leak checked at a vacuum pressure greater than or equal to the highest vacuum pressure observed during the run. Since there was no visible water in the first two (2) impingers, a nitrogen purge was not conducted.

The contents of impingers 1 and 2 were recovered in Container 1. Impingers 1 and 2, the coil condenser and all connecting glassware were rinsed with water and then rinsed with acetone and hexane. The water rinses were added to Container 1 while the solvent rinses were recovered in Container 2. The un-heated Teflon filter was removed from the filter holder and placed in Container 3. The front half of the condensable filter holder was rinsed with water and then with acetone and hexane. The water rinse was added to Container 1 while the solvent rinses were added to Container 2. All containers were sealed, labeled and liquid levels marked for transport to AST's laboratory in Decatur, AL for condensable particulate matter analysis.

The pre-weighed filter was carefully removed and placed in Container 4. The back-half of the PM10 cyclone and the connecting stainless tubing were rinsed three (3) times with acetone, and these rinses were recovered in Container 5. All containers were sealed, labeled and liquid levels marked for transport to AST's laboratory in Decatur, Alabama for filterable particulate matter analysis.

To determine the condensable particulate matter mass, the filter from Container 3 was placed in an extraction tube, rinsed with water then acetone and hexane and sonicated. The water rinse was added to Container 1 while the solvent rinse was added to Container 2. The contents of Containers 1 was added to a separatory funnel and mixed with hexane. Hexanes extractions were conducted, and the organic fraction was drained from the funnel and added to Container 2. After the organic and inorganic fractions were separated, the mass of each fraction was determined by evaporating the applicable solvents in pre-weighed beakers and then weighing the residue in each beaker until a constant weight was obtained. All weight measurements were performed on the same balance (accurate to 0.1 mg)

The mass of filterable PM10 was determined by evaporating the acetone rinses from Container 5 in separate pre-weighed dishes and then weighing the residue until constant weights was obtained. The filter loading was determined by subtracting the initial constant filter weight from the final constant weight. All weight measurements were performed on the same balance (accurate to 0.1 mg).

3.11 Quality Assurance/Quality Control - U.S. EPA Reference Test Method 3A

Cylinder calibration gases were supplied by AirGas – South or NexAir which met Protocol 1 (+/- 2%) standards.

Low Level gases were introduced directly to analyzer. After adjusting the analyzer to the Low Level gas concentration and once the analyzer reading was stable, the analyzer reading was recorded. This process was repeated for the High Level gas. Next, Mid Level gases were introduced directly to analyzer and reading was recorded. All recording readings were within +/- 2 percent of the Calibration Span.

All data was reviewed by the Field Team Leader before leaving the facility. Once arriving at AST's office, all written and electronic data was relinquished to the report coordinator and then a final review was performed by the Project Manager.

3.12 Quality Assurance/Quality Control – U.S. EPA Reference Test Method 7E

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