From: <u>Jonathan Walling</u>

To:

Cc: OCE; Holloway, Don; Warnement, Tom E

Subject: Lyondell Chemical Company, Channelview Plant - Performance Test Extension Request

Date: Friday, April 17, 2020 4:28:42 PM

Attachments: 2020 F65630 LYB Performance Test TCEO Notification 30-Jan-20.pdf

Good afternoon, Mr. Mayo:

Your request for enforcement discretion at Lyondell Chemical Company- Channelview Complex, as presented, to extend the date to conduct the performance testing of POSMI-F-630 Hot Oil Heater, as required by 40 Code of Federal Regulations Part 63, Subpart DDDDD and 30 TAC §113.1130, is approved with the following qualifications:

- Enforcement discretion is limited to the Texas Commission on Environmental Quality. It does not limit EPA's ability to enforce federal regulations; and
- This enforcement discretion will be in effect until June 30, 2020. The required testing shall be completed as soon as practicable, as conditions allow, before this date. Should an extension beyond this be needed, please re-submit your request.

Regulated entities must maintain records adequate to document activities related to the noncompliance under enforcement discretion, including details of the regulated entity's best efforts to comply.

Regards,

Jonathan Walling, Area Director Coastal & East Texas Area Texas Commission on Environmental Quality

From: Mayo, Randall S <

Sent: Friday, April 17, 2020 11:51 AM

To: OCE < OCE@tceq.texas.gov >; Ramiro Garcia < ramiro.garcia@tceq.texas.gov > Cc: Holloway, Don < Warnement, Tom E

Subject: Lyondell Chemical Company, Channelview Plant - Performance Test Extension Request

Lyondell Chemical Company, Channelview Plant, is submitting a request to extend the date to conduct the performance testing of POSMI-F-630 Hot Oil Heater by 90 days. Testing is being done to demonstrate compliance with the National Emission Standards for Hazardous Air Pollutants (NESHAP) for Major Sources: Industrial, Commercial, and Institutional (ICI) Boilers and Process Heaters, Title 40 Code of Federal Regulations (CFR) Part 63, Subpart DDDDD and as required by 30 TAC 113.1130.

On January 30, 2020, Lyondell submitted a notification and test plan via email and letter of intention to perform performance testing as required by 40 CFR 63.7515(a) no earlier than May 4, 2020. The test plan and notification letter to Region 12 have been included with this correspondence. This extension request is due to reduced operating rates caused by the COVID-19 pandemic. At the

reduced operating rates the plant is unable to support testing at maximum firing rates required by the test.

Lyondell is requesting until August 3, 2020 to conduct this testing assuming that normal operations and work schedules can resume at that time. Should resources be available and operating rates return to the level necessary to perform the testing earlier, Lyondell will make every effort to perform the testing as soon as possible.

Please feel free to contact me if you have any questions or concerns regarding this matter. Thank you for your consideration.

Scott Mayo Environmental Manager – Waste & Water LyondellBasell Channelview Complex 281-452-8109

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January 30, 2020

CERTIFIED MAIL-7015.0640.0002.0784.9603 RETURN RECEIPT REQUESTED

Air Program, Region XII
Texas Commission on Environmental Quality
5425 Polk, Suite H
Houston, Texas 77023-1423
Attention: Mark Kolkmeier
Stack Test Team

Re:

Notification to Conduct Boiler MACT 5D Performance Test

Lyondell Chemical Company - Channelview Texas, Harris County

TCEQ Air Quality Permit No. 2993 Regulated Entity Number: RN100633650 Customer Reference Number: CN600344402

Account Number: HG-1575-W

Lyondell Chemical Company, Channelview Plant, is providing written notification of a planned performance test on the atmospheric vent source (listed below) in the POSM I production unit. Testing is being done to establish the actual pattern and the quantity of air contaminants being emitted to demonstrate compliance with the National Emission Standards for Hazardous Air Pollutants (NESHAP) for Major Sources: Industrial, Commercial, and Institutional (ICI) Boilers and Process Heaters, Title 40 Code of Federal Regulations (CFR) Part 63, Subpart DDDDD and as required by 30 TAC 113.1130.

Unit	Source ID	Description	
POSM I-F-630	EF65630	Hot Oil Heater	

A formal test plan developed by Focus Environmental has been included in this notification. The source testing will take place no sooner than the week of May 4, 2020. Element Materials Technology will be performing the actual stack test under the direction of Focus Environmental.

If you have any questions regarding this information or desire to conduct a pretest meeting, please contact Susie K. Geraci, Environmental Engineer at 281-452-8866.

Sincerely.

Scott Mayo

Environmental Manager

Filing: 100-610-295

cc:

Executive Director Texas Commission on Environmental Quality P.O. Box 13087 Austin, Texas 78711

CERTIFIED MAIL 70190700000222040372

Director Harris County Public Health and Environmental Services 70190700000222040037 101 South Richey, Suite H Pasadena, Texas 77506

CERTIFIED MAIL

F-65630 COMPLIANCE TEST PLAN LYONDELL CHEMICAL COMPANY

PREPARED FOR:



LYONDELLBASELL INDUSTRIES 2502 SHELDON ROAD CHANNELVIEW, TEXAS 77530

> JANUARY 2020 REVISION: 3 FOCUS PROJECT NO. P-001409

PREPARED BY:



FOCUS ENVIRONMENTAL INC. 4700 PAPERMILL DRIVE KNOXVILLE, TENNESSEE 37909 (865) 694-7517

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Appendix A Appendix B Sampling Method Summaries Example Forms

List of Acronyms

acfm actual cubic feet per minute

acm actual cubic meters

As Arsenic

ASTM American Society for Testing and Materials

Be beryllium

Btu British thermal unit °C degrees centigrade cc or cm³ centimeter cubed

CCB continuing calibration blank

Cd cadmium

CEM continuous emissions monitoring
CEMS continuous emissions monitoring system

CFR Code of Federal Regulations

C-H-N-O carbon, hydrogen, nitrogen, and oxygen (ultimate analysis)

Cl₂ molecular chlorine
CO carbon monoxide
CoC chain of custody
Cr chromium
cSt centistokes

CVAA or

CVAAS cold vapor atomic adsorption spectroscopy

dscf dry standard cubic foot

dscfm dry standard cubic feet per minute

dscm dry standard cubic meter

EPA U.S. Environmental Protection Agency

°F degrees Fahrenheit

g gram gr grains

gpm gallons per minute g/s grams per second

HCl hydrogen chloride (gas) or hydrochloric acid (aqueous)

Hg mercury

HHV higher heating value

HNO₃ nitric acid

H₂O₂ hydrogen peroxide

hr hour

HRA hourly rolling average

H₂SO₄ sulfuric acid

CCV continuing calibration verification
ICI Industrial, Commercial, and Institutional
ICP or ICAP inductively coupled argon plasma
ICV initial calibration verification

inwc inches water column

kg kilogram

KMnO₄ potassium permanganate

I liter lb pound

lb/MMBtu pounds per million British thermal units

LCS laboratory control standard

LCSD laboratory control standard duplicate

LMB laboratory method blank

MACT Maximum Achievable Control Technology

MDL method detection limit

mg milligram

min minute ml milliliter

MMBtu/hr million British thermal units per hour

Mn manganese

mscfm thousand cubic feet per minute

MS matrix spike

MSD matrix spike duplicate

NA not applicable NaOH sodium hydroxide

ND non-detect

NESHAP National Emission Standards for Hazardous Air Pollutants

 $\begin{array}{ll} \text{Ni} & \text{nickel} \\ \text{O}_2 & \text{oxygen} \\ \text{Pb} & \text{lead} \end{array}$

PDS post-digestion spike

PPE personal protective equipment

ppm parts per million

ppmdv parts per million dry volume
ppmv parts per million by volume
psig pounds per square inch, gauge

QA quality assurance
QC quality control
RFA request for analysis
RL reporting limit

RPD relative percent difference
RSD relative standard deviation
scfm standard cubic feet per minute

Se selenium sec second

SSASP Stationary Source Audit Sample Program

SW846 "Test Methods for Evaluating Solid Wastes Physical/Chemical Methods (SW-846),"

Third Edition, 1986 and updates (December 1997).

TSM total select metals which includes: arsenic (As), beryllium (Be), cadmium (Cd),

chromium (Cr), lead (Pb), manganese (Mn), nickel (Ni), and selenium (Se)

 $ug \ or \ \mu g$ micrograms $ng \ or \ \eta g$ nanograms

1.0 TEST PROGRAM SUMMARY

1.1 UNIT AND TEST BACKGROUND

This test plan describes protocol for testing of the LyondellBasell Industries (Lyondell) F-65630 Oil Heater (F-65630) located at the Channelview, Texas facility. The F-65630 in an industrial furnace that burns a combination of liquid and gaseous fuels to heat thermal transfer fluid (hot oil) for use in the manufacturing processes. Testing will demonstrate compliance of F-65630 with the National Emission Standards for Hazardous Air Pollutants (NEHSHAP) for Major Sources: Industrial, Commercial, and Institutional (ICI) Boilers and Process Heaters (Boiler MACT), Title 40 Code of Federal Regulations (CFR) Part 63, Subpart DDDDD.

1.2 TEST PROTOCOL

The F-65630 is fueled by a combination liquid fuel, a vapor stream composed of mostly propane with D-65951 by-product vapors, and natural gas. Testing will be conducted at a single operating condition with three replicate sampling runs. F-65630 will be operated, as nearly as possible, to the maximum liquid fuel and vapor/gas firing rates. The sampling and analyses includes the following:

- Liquid fuel for heating value, ash, chlorine, sulfur, density, ultimate [carbon, hydrogen, oxygen, and nitrogen (C-H-N-O)], total select metals [TSM includes arsenic (As), beryllium (Be), cadmium (Cd), chromium (Cr), lead (Pb), manganese (Mn), nickel (Ni), and selenium (Se)], and mercury (Hg)
- TSM and mercury emissions using EPA Method 29
- Hydrogen chloride/chlorine (HCI/CI₂) emissions using EPA Method 26A
- Carbon Monoxide (CO) emissions using installed continuous emissions monitoring system (CEMS) operating in conformance with 40 CFR 60, Appendix F.

TSM, mercury, and HCl/Cl₂ emissions results will be evaluated on a thermal input basis using EPA Method 19. The liquid fuel analyses will be used to calculate fuel-specific emission factors used in the EPA Method 19 emissions analysis. Historical average analysis data for the composition of the combined propane and D-65951 by-product vapors (fuel gas) stream will be used in performing the Method 19 heat input emissions analysis. The Method 19 default value for natural gas will be used for the natural gas heat input.

1.3 TEST PLAN ORGANIZATION

This test plan is organized into six sections as follows:

- Section 1.0 Test Program Summary;
- Section 2.0 Engineering and Fuel Descriptions;
- Section 3.0 Test Program Objectives, Protocol, and Schedule;

- Section 4.0 Quality Assurance/Quality Control Procedures; and
- Section 5.0 Test Report.

1.4 REFERENCE DOCUMENTS

Reference documents that have been used in developing the plan include the following:

- National Emission Standards for Hazardous Air Pollutants (NEHSHAP) for Major Sources: Industrial, Commercial, and Institutional (ICI) Boilers and Process Heaters, Title 40 Code of Federal Regulations (CFR) Part 63, Subpart DDDDD.
- American Society for Testing and Materials, "Annual Book of ASTM Standards," latest annual edition.
- EPA, "New Source Performance Standards, Test Methods and Procedures," Appendix A, 40 CFR 60.
- EPA, "Test Methods for Evaluating Solid Wastes Physical/Chemical Methods (SW-846)," Third Edition, 1986 and updates.
- EPA Requirements for Quality Assurance Project Plans (EPA QA/R-5 EPA/240/B-01/003), March 2001.
- Interim Guidelines and Specifications for Preparing Quality Assurance Project Plans (QAMS-005/80).

2.0 ENGINEERING AND FUEL DESCRIPTIONS

2.1 ENGINEERING DESCRIPTION

F-65630 engineering design information is summarized in Table 2-1. F-65630 is equipped with twelve (12) liquid and vapor burners that fire natural gas or natural gas/vapor mixture, and liquid fuel. The unit's burners are configured in two concentric rings, with four (4) burners forming the inner ring and eight (8) burners forming the outer ring. The liquid fuel feed to F-65630 burners is pumped from tank TK-65635. Steam is used as the atomizing media for the liquid fuel. The nominally rated firing capacity of the F-65630 is 138 MMBtu/hr including the burner pilots. The flow of natural gas and/or natural gas/by-product gas mixture is varied to control the product oil temperature. The D-65951 vapors (fuel gas) are co-fired with natural gas through both the inner and outer ring burners. The liquid fuel is co-fired with the vapor/natural gas. The F-65630 energy recovery section is constructed of two sections, radiant and convective. F-65630 has its own stack and is not equipped with any emissions control devices. F-65630 has combustion air blowers on the burners. The natural draft provided by the F-65630 stack maintains an induced-draft on the unit's combustion zone.

2.2 FUEL DESCRIPTION

Figure 2-1 is a schematic of the liquid fuel and vapor feed (fuel gas) streams to F-65630. Liquid fuel analyses from the previous test conducted in April 2017 are summarized in Table 2-2. Historical analyses of the vapor vent fuel are summarized in Table 2-3. The fuel values of the streams are calculated via the formula presented in Figure 2-2. Natural gas is used as auxiliary fuel in F-65630. Typical characteristics of the natural gas are summarized in Table 2-4.

Table 2-1. F-65630 Engineering Data

Parameter	F-65630
Manufacturer:	KTI Corporation
Model No.:	KTI 75-01-403
Type:	Vertical Cylindrical with Convection
Date of Mfr.:	1976
No. of Burners:	Twelve (12)
Burner Type:	Steam atomized liquid burner
Heated Oil Conditions:	47 psig, 625°F
Maximum Heated Oil Production:	3,600,000 lb/hr
Minimum Heated Oil Production	2,200,000 lb/hr
Maximum Heat Release:	138 MM Btu/hr
CO CEMs Manufacturer/Type	Siemens Ultramat 5F
	Nondispersive Infrared (NDIR) Analyzer
O ₂ CEMs Manufacturer/Type	Servomex Series 1100A Paramagnetic
	Analyzer

Table 2-2. F-65630 Liquid Fuel Analyses

Parameter	Units		Run 1			Run 2			Run 3			Test	
			12-Apr-17	1		13-Apr-17	ı		14-Apr-17	,		Average	
Ash	mg/kg		289			275			249			271	
Density	g/cm ³		1.03			1.03			1.03			1.03	
Heating Value	BTU/lb		15,100			15,200			15,300			15,200	
Total Chlorine	mg/kg		59.1	J	<	98.0	ND		81.3	J	<	79	
Total Sulfur	mg/kg		29.6	J		36.4	J		90.0	J		52	
Carbon	wt%		80.17			80.10			79.80			80.02	
Hydrogen	wt%		8.17			8.10			8.07			8.11	
Nitrogen	wt%		0.72			0.53			0.62			0.62	
Oxygen	wt%		12.17			12.30			12.12			12.20	
F _d (calculated)	scf/MMBtu		9,729			9,635			9,542			9,635	
Arsenic	mg/kg		0.0187	J		0.0285	J		0.0295	J		0.0256	
Beryllium	mg/kg	<	0.0399	ND	<	0.0399	ND	<	0.0399	ND	<	0.0399	ND
Cadmium	mg/kg	<	0.0200	ND	<	0.0200	ND	<	0.0199	ND	<	0.0200	ND
Chromium	mg/kg		0.189			0.192			0.204			0.195	
Lead	mg/kg	٧	0.0599	ND		0.0136	J		0.0118	J	٧	0.0284	
Manganese	mg/kg		0.0695			0.0674			0.0787			0.0719	
Nickel	mg/kg		0.323			0.336			0.358			0.339	
Selenium	mg/kg		0.0334	J		0.0436	J		0.0429	J		0.0400	
Mercury	mg/kg	<	0.00798	ND	<	0.00798	ND	<	0.00797	ND	<	0.00798	ND

ND - Non-detect at the reporting limit (RL) noted.

J - Result less than the RL, but greater than or equal to the method detection limit (MDL): The concentration noted is an approximate value.

Table 2-3. Historical Vapor Vent Fuel Analyses

	Monthly	Average Comp	osition - Analysi	s (wt%)		
MONTH	ACETALDEHYDE	PROPANE	PROPYLENE	BUTANE	PROPYLENE OXIDE	TOTAL
January	5.757	77.458	1.196	6.24	8.183	98.834
February	4.442	83.981	1.583	3.799	5.006	98.811
March	3.270	88.019	2.169	2.208	3.308	98.974
April	4.024	86.812	2.127	2.141	3.673	98.777
May	4.007	85.164	2.003	1.715	6.213	99.102
June	3.429	80.585	8.657	1.465	5.208	99.344
July	2.703	91.083	1.631	0.885	3.065	99.367
August	2.876	90.374	2.972	0.816	2.317	99.355
September	3.229	89.688	2.56	1.163	2.672	99.312
October	3.226	90.844	1.818	0.867	2.738	99.493
November	2.981	90.244	1.651	0.982	3.571	99.429
December	4.306	80.314	1.246	0.814	5.606	92.286
Average	3.688	86.214	2.468	1.925	4.297	98.590

	Monthly A	verage Compo	sition - Normaliz	ed (wt%)		
MONTH	ACETALDEHYDE	PROPANE	PROPYLENE	BUTANE	PROPYLENE OXIDE	TOTAL
January	5.825	78.372	1.210	6.314	8.280	100.0
February	4.495	84.992	1.602	3.845	5.066	100.0
March	3.304	88.931	2.191	2.231	3.342	100.0
April	4.074	87.887	2.153	2.168	3.718	100.0
May	4.043	85.936	2.021	1.731	6.269	100.0
June	3.452	81.117	8.714	1.475	5.242	100.0
July	2.720	91.663	1.641	0.891	3.085	100.0
August	2.895	90.961	2.991	0.821	2.332	100.0
September	3.251	90.309	2.578	1.171	2.691	100.0
October	3.242	91.307	1.827	0.871	2.752	100.0
November	2.998	90.762	1.660	0.988	3.592	100.0
December	4.666	87.027	1.350	0.882	6.075	100.0
Average	3.740	87.447	2.503	1.952	4.358	100.0

Gas Component	Vol%	HHV, Btu/lb	MW	lb/scf	Btu/scf	С	Н	0				
Propane	87.439	20,769	44.0	0.11420	2,372	3	8	0				
Butane	1.949	19,669	58.0	0.15053	2,961	4	10	0				
Propylene Oxide	4.370	13,226	58.0	0.15053	1,991	3	6	1				
Propylene	2.495	19,693	42.0	0.10901	2,147	3	6	0				
Acetaldehyde	3.747	10,786	44.0	0.11420	1,232	2	4	1	wt%C	wt%H	wt%O	Total
TOTAL	100.0	20,017	44.8	0.11636	2,329	2.98	7.75	0.08	79.8%	17.3%	2.9%	100.0%
		F _d	=	9,178	scf/	MMBt	u					

Table 2-4. Typical Characteristics of Natural Gas

Constituent/Duomonty	Unita		Value	
Constituent/Property	Units		Typical	Range
	Primary Com	nponents		
Methane	vol%		93.7	93.4 - 93.9
Ethane	vol%		3.3	2.8 - 3.6
Propane	vol%		0.5	0.5
i-Butane	vol%		0.07	0.06 - 0.1
n-Butane	vol%		0.09	0.08 - 0.1
i-Pentane	vol%		0.03	0.02 - 0.05
n-Pentane	vol%		0.02	0.02 - 0.03
Hexane (plus)	vol%		0.05	0.04 - 0.06
Hydrogen sulfide	ppmv	<	1	0 - 1
Phy	sical/Chemic	al Properties		
Heating Value	Btu/scf		1,028 - 1,033	1,030
Vapor Specific Gravity	NA		0.593 - 0.595	0.594
Тур	ical Ultimate	Composition		
Carbon	wt%		74.	8
Hydrogen	wt%		24	
Oxygen	wt%		0	
Nitrogen	wt%		1.2	2
Sulfur	wt%		0	
Chlorine/Chloride	ug/m³	<	1.6	3

Source: "Analysis of Trace Level Compounds in Natural Gas" Gas Research Institute, Document Number GRI-99/0111, February, 2000

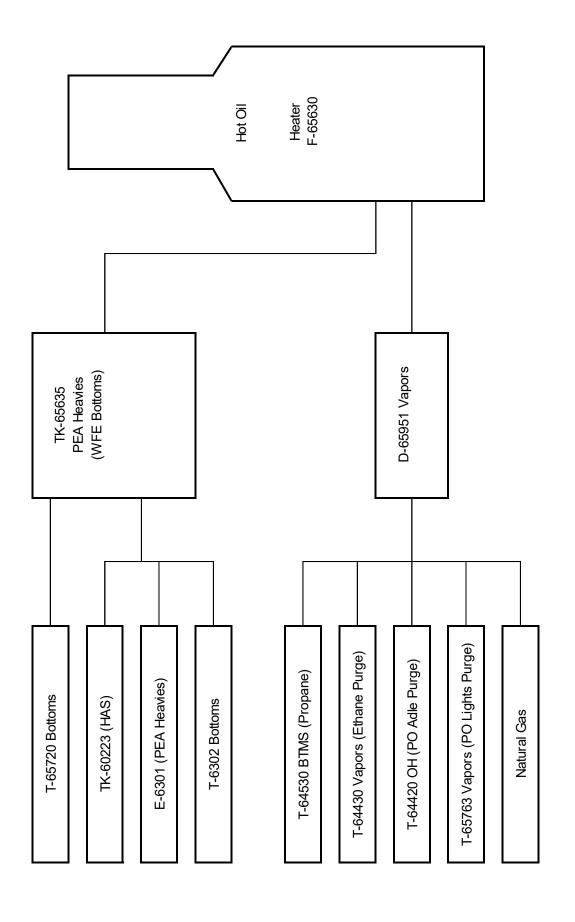


Figure 2-1. Hot Oil Heater F-65630 Feed Streams

Calculation of Volume Factor based on Fuel Analysis

Volume Factor for Mixed Fuel Firing

C_d = Pollutant concentration, dry basis, lb/scf.
 E = Pollutant emission rate, lb/million Btu.
 O₂ = Oxygen concentration, % dry volume

Figure 2-2. Fuel Feed Stream Heating Value Formula

3.0 TEST PROGRAM OBJECTIVES, PROTOCOL, AND SCHEDULE

3.1 TEST OBJECTIVES OVERVIEW

The test program will demonstrate compliance of the F-65630 with the following 40 CFR 63, Subpart DDDDD emissions standards:

- Emissions of total select metals [TSM, which includes arsenic (As), beryllium (Be), cadmium (Cd), chromium (Cr), lead (Pb), manganese (Mn), nickel (Ni), and selenium (Se)] less than 2.0E-04 pounds per million Btu (lb/MMBtu) of fuel firing
- Mercury (Hg) emissions less than 2.0E-06 lb/MMBtu of fuel firing
- Hydrogen chloride/chlorine (HCI/Cl₂) emissions less than 1.1E-03 lb/MMBtu of fuel firing
- Carbon monoxide (CO) emissions less than 130 parts per million, dry basis corrected to three percent oxygen (ppm, dry @ 3% O₂).

3.2 TEST PROTOCOL

The test will be composed of a single, maximum fuel firing test condition with three replicate sampling runs. The following sampling and analyses will be performed:

- Liquid fuel for heating value, ash, moisture, chlorine, sulfur, density, ultimate [carbon, hydrogen, oxygen, and nitrogen (C-H-N-O)], TSM, and mercury (Hg)
- TSM and Hg emissions using EPA Method 29
- HCI/Cl₂ emissions using EPA Method 26A
- CO emissions using installed continuous emissions monitoring system (CEMS) operating in conformance with 40 CFR 60, Appendix F.

Sampling and analysis methods are summarized in Table 3-1. Summary sampling procedures are included as Appendix A. The target sampled stack volumes for Method 26A and 29 are as specified in Table 2 of 40 CFR 63, Subpart DDDDD. A schematic of the stack sampling location with dimensions is presented as Figure 3-1.

Lyondell has the option to comply with either the particulate or TSM emissions standard. Lyondell has elected to comply with the TSM emissions standard. Method 29 will be used to demonstrate compliance with the TSM and mercury emissions standards. Method 26A will be used to demonstrate compliance with the HCI/Cl₂ emissions standard.

3.3 TEST RESPONSIBLE PARTIES

Lyondell will operate F-65630 at maximum fuel firing rate conditions during the test. Experienced Lyondell operators wearing appropriate personal protective equipment (PPE) will collect the liquid fuel samples. Stack gas samples will collected by contractor personnel. The following paragraphs identify the contractors and their test program responsibilities.

LyondellBasell Industries, Channelview, TX F-65630 Compliance Test Plan Revision: 3, January 2020

3.3.1 Test Management

Focus Environmental, Inc. (Focus), Knoxville, Tennessee will be responsible for overall management and coordination of the on-site testing activities including direction of the sampling contractor. Focus will be responsible for managing the test samples. Focus will transport the liquid fuel and emissions samples directly to the analytical laboratory. Focus will also coordinate the post-test data reduction and reporting by the stack sampling and analytical laboratory contractors.

3.3.2 Stack Gas Sampling

Element Materials Technology (Element), Houston, Texas will responsible for providing stack gas sampling services related to the test program. Element's scope includes providing a report of the stack gas sampling results.

3.3.3 Sample Analysis

The TestAmerica (TestAmerica), Knoxville, Tennessee laboratory will be responsible for analysis of the liquid fuel and all the stack gas samples collected by Lyondell, Focus, and Element. TestAmerica will provide the pre-labeled sample containers for all of the process and stack gas samples. Preparation of the pre-labeled sample containers will include preparation of an associated sample data base and numbering system for tracking samples from field collection through analysis.

3.4 TEST SCHEDULE

Lyondell is targeting the week of May 4, 2020 for performing the testing. Equipment set up and preparations for the testing will begin on Monday. The Method 26A and Method 29 sampling will be performed concurrently. The test will consist of three test runs. Completion of the sampling is expected to take two days (Tuesday and Wednesday). Thursday will serve as a contingency day if there are any delays or interruptions in testing.

Table 3-1. F-65630 ICI Boiler and Process Heaters MACT Sampling and Analysis

Sample Location/	g Sampling	Sampling Reference Method ¹	Sample Size/Frequency	Analytical Parameters	Analytical Reference Method ¹
Tap on line		ASTM E-300-86	Collect a 250 mL grab sample at each 30-minute interval	Heating Value Density	ASTM D-240 ASTM D-1475
	grab		during each test run. The grab	Ash Content Total Chloride	ASTM D-482 SW846 5050/90564
	L glass jug;		run composite sample in a 4L	C-H-N-O	ASTM D-3176
	250 and 500 mL		jug. At the end of the test run, collect the master sample list	Sulfur Moisture	ASTM D-129 ASTM D-4928
	glass		required sample aliquots for		
	pottles		zed	TSM: As, Be, Cd, Cr, Pb, Mn, Ni, &	TSM: ICAP (Method 3050B & 6010C)
				e N	
				Mercury (Hg)	Hg: CVAAS (Method 7471A)
Isokinetic	Method 29	40 CFR 60; App	Minimum sampled volume of	As, Be, Cd, Cr,	EPA Method 29; SW846 Method
Port		A, Method 29	four (4) dry standard cubic meters. 2, 3	Pb, Mn, Hg, Ni, & Se	6010C, 7471A
Isokinetic		40 CFR 60; App	Minimum sampled volume of	HCI/Cl ₂	EPA Method 26A and SW846 Method
Port	26A	A, Method 26A	two (2) dry standard cubic meters. 2, 3		9056A
CEMS Po	CEMS Port Installed	40 CFR 60,	Continuous	CO and O ₂	40 CFR 60, Appendix F
	CO and O ₂	Appendix F			

¹ Reference Method Sources:

- "ASTM" refers to American Society for Testing Materials, Annual Book of ASTM Standards, Annual Series
 - "SW846" refers to Test Methods for Evaluating Solid Waste, Third Edition, November 1986, and Updates.
- "EPA Method" refers to New Source Performance Standards, Test Methods and Procedures, Appendix A, 40 CFR 60.

- Analysis of oxygen and carbon dioxide concentrations to determine stack gas molecular weight (EPA Method 3A)
- Performing stack gas velocity, pressure and temperature profile measurements for each sampling location (EPA Method 2) Determining the moisture content of the stack gas for each sampling train sample (EPA Method 4)

²The exact volume of gas sampled will depend on the isokinetic sampling rate.

³ Isokinetic sampling trains include:

Figure 3-1. F-65630 Stack Sampling Locations

4.0 QUALITY ASSURANCE/QUALITY CONTROL PROCEDURES

4.1 DATA QUALITY OBJECTIVES

Table 4-1 summarizes the test program analytical data quality objectives (DQOs).

4.2 SAMPLE COLLECTION

Table 4-2 summarizes the test program field samples, including quality control (QC) samples, to be collected. Example forms for the field data collection and sample management are presented in Appendix B.

4.3 SAMPLE PRESERVATION

Table 4-3 summarizes the sample containers, preservation, and holding times.

4.4 SAMPLE ANALYSES

Table 4-4 summarizes the all analyses to be performed, including QC analyses.

4.5 ANALYTICAL INSTRUMENT QUALITY CONTROL CHECKS

Table 4-5 summarizes the analytical instrument quality control checks, frequencies, acceptance criteria, and corrective actions.

4.6 SAMPLING EQUIPMENT CALIBRATIONS

Table 4-6 summarizes the sampling equipment calibration requirements.

4.7 ANALYTICAL DATA PACKAGES

Analytical data packages will be organized in accordance with the laboratory standard operating procedures. The complete analytical data package is a stand-alone deliverable that includes the final analysis results, raw analytical instrument data, initial and continuing calibration data, parameter-specific quality control documentation, sample preparation documentation, and records of sample traceability. These data are sufficient for performing independent verification of the final analytical results. Every analytical data package includes the following:

- Cover Page Identifies the laboratory-assigned lot number, project identification, laboratory project manager, and issue date.
- Table of Contents Organization of the data package.
- Sample Summary Cross reference to project sample identifications and laboratory sample identifications.
- Analytical/Preparation Methods Summary Identifies the methods used to prepare and analyze the samples.

- Narrative Summarizes the project-specific information and any pertinent information concerning data quality. The narrative documents sample delivery and condition upon receipt, and any analytical difficulties or anomalies encountered during sample preparation and analysis.
- QC Data Association Summary Comparison of sample results to the project and laboratory DOQs and association of project samples to laboratory QC.
- Analytical Data Report Summarized analytical data for all samples and associated quality assurance samples including as appropriate: data flags, duplicate analysis results, surrogate recovery results, method blank results, laboratory control sample results, and matrix spike/matrix spike duplicate/post digestion spike (MS/MSD/PDS) results.
- Chain-of-custody documentation

Each analytical data package will include the identification and signature or initials of each analyst who handles the test samples. The Laboratory Analysis Coordinator will certify via signature the contents of each analytical data package.

4.7.1 Metals Analyses

The metals analytical data packages will include the following:

- Sample Results
- Quality Control Summary
 - Method Blank Report
 - Sample Duplicate Report
 - MS/MSD/PDS Data and Evaluation Report
 - Laboratory Control Sample Data and Evaluation Report
- Quality Control Summaries
 - Initial Calibration Verification and Blank Results
 - Continuing Calibration Verification and Blank Results
 - Interference Check Sample
 - PDS Sample Recovery
 - Serial Dilutions Documentation for ICAP
 - Instrument Detection Limits
 - Correction Factors
 - Linear Ranges
- Raw Data
 - Data Review Checklist
 - Sample, Standards, and Quality Control Data
 - Digestion/Extraction Sheets
 - Preparation Log
 - Analysis Run Log.

4.7.2 Other Inorganic Analyses

The data packages for other inorganic analyses will include the following:

- Sample Results
- QC Summary
 - Method Blank Report
 - Sample Duplicate report
 - MS/MSD Data and Evaluation Report(s)

- Laboratory Control Sample Data and Evaluation Report
- Raw Data
 - Data Review Checklist
 - Sample, Standards, and Quality Control Data
 - Distillation, Extraction, and Sample Preparation Sheets
 - Standards Preparation Logs
 - Run Logs

4.8 ASSESSMENT PROCEDURES FOR ACCURACY, PRECISION, & COMPLETENESS

4.8.1 General

The QA activities implemented in this test program will provide a basis for assessing the accuracy and precision of the analytical measurements. The generalized forms of the equations that will be used to calculate accuracy and precision are presented below.

4.8.2 Accuracy

When a reference standard material is used in the analysis, percent Accuracy (A) will be calculated as follows:

$$A = \frac{\text{Found concentration}}{\text{True concentration}} \times 100$$

Percent analyte Recovery (R) will be calculated as follows:

$$R = \frac{X - N}{S} \times 100$$

Where X is the experimentally determined value, N is the amount of native material in the sample, and S is the amount of spiked material of the species being measured. Recoveries are used to determine accuracy when standards are not available, or are not appropriate for a given matrix.

4.8.3 Precision

When less than three analyses of the same parameter are available, precision will be calculated as a Relative Percent Difference (RPD) from the average of replicate measurements according to:

$$RPD = \frac{(X_1 - X_2)}{\text{Average } X} \times 100$$

Where X₁ and X₂ are the highest and lowest results of replicate measurements.

Where three or more analyses of the same parameter are available, the precision will be determined as the Relative Standard Deviation (RSD) according to:

$$RSD = \frac{Standard deviation}{Average X} \times 100$$

4.8.4 Completeness

Completeness of data generated from a test program is usually calculated as follows:

% Completeness =
$$\frac{\text{Valid data}}{\text{Expected data}} \times 100$$

Data completeness is defined as the percentage of valid data collected from the total number of valid tests conducted. Three valid test runs are required for the test to be completed. If an individual sample from a test run is lost or broken, the data for that individual analytical parameter may not be 100% complete. This, however, may not invalidate the test run. The completeness objective for this test program is to generate sufficient data for the regulatory agencies to judge the performance of the system.

4.9 PERFORMANCE AUDITS

EPA has established the Stationary Source Audit Sample Program (SSASP). The program requires a minimum of two EPA SSASP providers. However, at the time of this test plan publication, the SSASP has been indefinitely suspended by EPA due to one of the two approved providers withdrawing from the program. If the program is re-instated, Lyondell will obtain as may be available audit samples for the Method 26A (HCI/Cl₂) and Method 29 (Multi-Metals) sampling trains. EPA does not currently have or

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provide any other audit samples. Audit samples are included in the sampling and analytical summary tables of this test plan in the event the SSASP program is re-instated and samples become available by the time of testing.

Table 4-1. Test Analytical Data Quality Objectives

Parameter	QC type	Precision	Accuracy
	Liquid Waste Fe	eds	
Heating Value, Density, Ash, Total Chlorine, Total Sulfur, and	Duplicates	≤ 10% RPD	
Ultimate	Reference Sample		<u>+</u> 10%
Total Chlorine and Total Sulfur	Matrix Spikes	10% RPD	<u>+</u> 10%
Metals	Duplicates	<u><</u> 20% RPD	
	Matrix Spikes	<u><</u> 20% RPD	75-125%
	Method 26A Sampling T	rain HCI/CI ₂	
Hydrogen Chloride/Chlorine	Duplicates	≤ 25% RPD	
	Matrix Spikes	≤ 35% RPD	<u>+</u> 30%
	Reference Sample		<u>+</u> 10%
	Audit Samples		70-130%
	Method 29 Samplin	g Train	
Method 29 Metals	Duplicates	<u><</u> 20% RPD	
	Matrix Spikes and Post- Digestion Spikes	≤ 20% RPD	75-125%
	Reference Sample		70-130%
	Audit Samples		70-130%
	Continuous Emissions	s Monitors	
Carbon Monoxide	40 CFR 60, Appendix F	<u>+</u> 3% of Span	<u>+</u> 5% of Span
Oxygen	40 CFR 60, Appendix F	<u>+</u> 0.5% Oxygen	<u>+</u> 0.5% Oxygen
	Method 3A Anal	yses	
Carbon Dioxide	Certified Calibration Gas		± 0.5% CO ₂ for Three Point Calibration
Oxygen	Certified Calibration Gas		± 0.5% O ₂ for Three Point Calibration

Table 4-2. Sample Collection Methods, Equipment, and Frequency

		•					
Sample Name	Sampling Reference Method	Sample Container	Analysis	General Procedure/Frequency	Test	Field QC	Total Field Samples
Liquid Fuel	ASTM D-4057, E-300-86	500 mL glass bottle	Properties, Chloride, Sulfur, and Water	Collect a 250 mL grab sample at each 30-minute interval during each test	8	I	ဧ
		500 mL glass bottle	Ultimate (C-H-O- N)	run. The grab samples will be used to build a run	က	ı	က
		250 mL glass bottle	As, Be, Cd, Cr, Pb, Mn, Hg, Ni, & Se	jug. At the end of the test run, collect the master sample list required sample aliquots for analysis from the homogenized composite sample.	က	:	ဇ
Method 29 Particulate Filter	EPA Method 29	Petri Dish	As, Be, Cd, Cr, Pb, Mn, Hg, Ni, & Se	Sample a minimum volume of four (4) dry standard cubic meters of	က	1- blank train	4
Method 29 Front Half Nitric Acid Rinses		250 mL glass sample bottle	As, Be, Cd, Cr, Pb, Mn, Hg, Ni, & Se	stack gas.	8	1- blank train	4
Method 29 Acidified Peroxide Impingers and Rinses		1-liter glass sample bottle	As, Be, Cd, Cr, Pb, Mn, Hg, Ni, & Se		င	1- blank train	4
Method 29 Empty Impinger and Rinses		250 mL glass sample bottle	Mercury Only		က	1- blank train	4
Method 29 Acidified Permanganate Impingers and Rinses		500 mL glass sample bottle	Mercury Only		က	1- blank train	4
Method 29 Acidified Permanganate Impingers 8N HCI Rinses		250 mL glass sample bottle	Mercury Only		က	1- blank train	4
Method 29 Particulate Filter Reagent Blank	EPA Method 29	Petri Dish	As, Be, Cd, Cr, Pb, Mn, Hg, Ni, & Se	Reagent Blank	:	-	-

Table 4-2. Sample Collection Methods, Equipment, and Frequency

			cample concern meaned adaptively and to adaptive	faa.ha			
Sample Name	Sampling Reference Method	Sample Container	Analysis	General Procedure/Frequency	Test	Field	Total Field Samples
Method 29 Nitric Acid Reagent Blank	EPA Method 29	250 mL glass sample bottle	As, Be, Cd, Cr, Pb, Mn, Hg, Ni, & Se	Reagent Blank	1	_	~
Method 29 Acidified Peroxide Reagent Blank	EPA Method 29	250 mL glass sample bottle	As, Be, Cd, Cr, Pb, Mn, Hg, Ni, & Se	Reagent Blank	1	_	~
Method 29 Acidified Permanganate Reagent Blank	EPA Method 29	250 mL glass sample bottle	Mercury Only	Reagent Blank	1	~	-
Method 29 8N HCI Reagent Blank	EPA Method 29	250 mL glass sample bottle	Mercury Only	Reagent Blank	ŀ	~	~
Multi-Metals Audit Sample Filter	SW846 Method 6010C	Spiked Filter in Petri Dish	As, Be, Cd, Cr, Pb, Mn, Ni, & Se	Obtained from SSASP supplier	-	1	1
Multi-Metals Audit Sample Liquid	SW846 Method 6010C	Ampoule with spiked Liquid	As, Be, Cd, Cr, Pb, Mn, Ni, & Se	Obtained from SSASP supplier	-	1	1
Mercury Audit Sample Filter	SW846 Method 7470	Filter in Petri Dish with Liquid Ampoule	Mercury Only	Obtained from SSASP supplier		1	1
Mercury Audit Sample Liquid	SW846 Method 7470	Ampoule with spiked Liquid	Mercury Only	Obtained from SSASP supplier	-	1	-
Method 26A Sulfuric Acid Impingers and Rinses	Method 26A	1-liter glass sample bottle	Chloride Ion	Sample a minimum volume of two (2) dry standard cubic meters of	3	1	8
Method 26A Sodium Hydroxide Impingers and Rinses		500 mL glass sample bottle	Chloride Ion	stack gas.	3	ı	ဇ
Method 26 Deionized Water Reagent Blank	Method 26A	250 mL glass sample bottle	Chloride Ion	Reagent Blank	1	~	~
Method 26 Sulfuric Acid Reagent Blank	Method 26A	250 mL glass sample bottle	Chloride Ion	Reagent Blank	1	1	1
Method 26A Sodium Hydroxide Reagent Blank	Method 26A	250 mL glass sample bottle	Chloride Ion	Reagent Blank	:	~	_

Table 4-2. Sample Collection Methods, Equipment, and Frequency

	l able 4-2.	l able 4-z. Sample Collection Methods, Equipment, and Frequency	Metnods, Equipment	, and r requency			
Sample Name	Sampling Reference Method	Sample Container	Analysis	General Procedure/Frequency	Test	Field	Total Field Samples
Chloride in Audit Sample	Method 26A	Ampoule with spiked liquid	Chloride Ion	Obtained from SSASP supplier	1	1	1
Carbon Monoxide	40 CFR 60, Appendix F	Installed	Carbon Monoxide	Continuous during each	1	:	1
		Emissions Monitor					
Oxygen	40 CFR 60,	Installed	Oxygen	Continuous during each	:	:	1
	Appendix F	Continuous		test run			
	:	EIIIISSIOIIS MOIIIGI			1		
Oxygen and Carbon Dioxide	EPA Method	Temporary	Oxygen and	Continuous during each	1	ı	1
	3A	Continuous	Carbon Dioxide	test run			
		Emissions Monitor					
TOTAL SAMPLES							52

Notes:

ASTM - American Society for Testing and Materials

EPA Method - New Source Performance Standards, Test Methods and Procedures, Appendix A, 40 CFR 60.

SW846 - Test Methods for Evaluating Solid Waste, Third Edition, November 1986, and Updates.

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Table 4-3. Sample Containers, Preservation, and Holding Times

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Parameter	Sample Name/Matrix	Sample Containers	Preservation	Maximum Holding Time
Physical Properties	Liquid Fuel	Amber glass Boston round bottles with Teflon-lined	NA	180 days
Chlorine and Sulfur	Liquid Fuel	Amber glass Boston round bottles with Teflon-lined caps	AA	30 days
Ash Content	Liquid Fuel	Amber glass Boston round bottles with Teflon-lined caps	ΨZ	180 days
Water Content	Liquid Fuel	Amber glass Boston round bottles with Teflon-lined caps	Ϋ́	180 days
Ultimate (C-H-N-O)	Liquid Fuel	Amber glass Boston round bottles with Teflon-lined caps	٩٧	180 days
Metals	Liquid Fuel	Amber glass Boston round bottles with Teflon-lined caps	Chill to 4°C for Hg	Non-Hg Metals: 180 days; Hg: 28 days
Stack Gas Metals	Method 29 filter	Glass or plastic Petri dish	Chill to 4°C for Hg	Non-Hg Metals: 180 days; Hg: 28 days
	Method 29 liquids	Amber glass Boston round bottles with Teflon-lined caps	Chill to 4°C for Hg	Non-Hg Metals: 180 days; Hg: 28 days
Stack Gas Chloride	Method 26A liquids	Polyethylene bottles	Chill to 4°C	30 days

References: Quality Assurance/Quality Control (QA/QC) Procedures for Hazardous Waste Incineration, EPA/625/6-89/023, January 1990 and promulgated method.

Table 4-4. Summary of Test Program Analyses

				· (
		Ë	Field	Reference	1 - 2 17 1 - 2 V		၁ ၂	ç	ŀ
Analysis	Sample Matrix	Test	samples est QC	Preparation Method	Kererence Analytical Method	QC Analysis	Analysis Frequency	طا Analyses	l otal Analyses
Heat Content	Liquid Fuel	3	:	NA	Bomb calorimeter (ASTM Method D-240)	Duplicate	One per stream	L	4
Density				NA	Gravimetric/volumetric (ASTM Method D-1475)	Duplicate	One per stream	←	4
Ash Content				NA	Residue after muffle furnace combustion (ASTM Method D-482)	Duplicate	One per stream	~	4
Water Content				NA	Titration (ASTM Method D-4928)	Duplicate	One per stream	~	4
Total Chloride				Bomb or flask combustion (SW-	Ion chromatography of residue (SW-846 Method	Duplicate	One per stream	_	4
				846 Method 5050)	9056A)	MS/MSD	One per stream	2	2
Total Sulfur				Bomb or flask combustion	Ion chromatography of residue (SW-846 Method	Duplicate	One per stream	1	4
				(ASTM D-129)	9056A)	MS/MSD	One per stream	2	2
Ultimate	Liquid Fuel	3	1	NA	ASTM Method D-3176	Duplicate	One per stream	1	4
Metals by ICP	Liquid Fuel	က	1	Digestion (SW- 846 Method 3050B)	ICP (SW-846 Method 6010C)	MS/MSD	One per stream	2	5
	Analytical system QC	Ϋ́	Ϋ́	Digestion (SW- 846 Method 3050B)	ICP (SW-846 Method 6010C)	SOT	One per batch/ matrix specific	1 or more	₩
						Serial dilution	One per batch/ matrix specific	1 or more	_

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Table 4-4. Summary of Test Program Analyses

Γ										
	Total Analyses	-	5	_	~	4	4	4	4	4
	QC Analyses	1 or more	2	1 or more	1 or more	~	_	_	_	_
	QC Analysis	One per batch/ matrix	specific One per stream	One per batch/ matrix specific	One per batch/ matrix specific	One per test	One per test	One per test	One per test	One per test
	QC Analysis	Method Blank	MS/MSD °	rcs	Method Blank	PDS	PDS	PDS	PDS	PDS
	Reference Analytical Method		CVAA (SW-846 Method 7471A)	CVAA (SW-846 Method 7471A)	,	ICP (SW-846 Method 6010C)	CVAA (SW-846 Method 7470A)	ICP (SW-846 Method 6010C)	CVAA (SW-846 Method 7471A)	CVAA (SW-846 Method 7471A)
	Reference Preparation		CVAA (SW-846 Method 7471A)	CVAA (SW-846 Method 7471A)		Method 29		Method 29		Method 29
2	Field Samples		:	Y V		1- blank train				
	Sam		က	₹ Z		ო		က		ო
	Sample Matrix		Liquid Fuel	Analytical system QC		Method 29 front half: filter, and nitric acid probe and front half	Tilter holder rinses	Method 29 10%HNO ₃ /5%H ₂ O ₂	impinger contents and rinses	Method 29 initially empty contents and rinses
	Analysis		Mercury by CVAA			Metals by Method 29				

Table 4-4. Summary of Test Program Analyses

		1		۱				
Analysis	Sample Matrix	Field Samples	Reference Preparation	Reference Analytical Method	QC Analysis	QC Analysis	QC Analyses	Total Analyses
Metals by Method 29 (conťd)	Method 29 4%KMnO ₄ /10%H ₂ SO ₄ impinger contents and rinses	m	Method 29	CVAA (SW-846 Method 7471A)	PDS	One per test	-	4
	Method 29 4%KMnO4/10%H2SO4 8N HCI rinses	ю	Method 29	CVAA (SW-846 Method 7471A)	PDS	One per test	~	4
	Method 29 filter reagent blank	←	Method 29	ICP (SW-846 Method 6010C)	Reagent Blank	Only analyze if required	1	~
				CVAA (SW-846 Method 7471A)	Reagent Blank	Only analyze if required	1	~
	Method 29 HNO3 reagent blank	-	Method 29	ICP (SW-846 Method 6010C)	Reagent Blank	Only analyze if required	1	_
				CVAA (SW-846 Method 7471A)	Reagent Blank	Only analyze if required	1	~
	Method 29 10%HNO3/5%H2O2 reagent blank	-	Method 29	ICP (SW-846 Method 6010C)	Reagent Blank	Only analyze if required	-	~
	,			CVAA (SW-846 Method 7471A)	Reagent Blank	Only analyze if required	-	~
	Method 29 4%KMnO ₄ /10%H ₂ SO ₄ reagent blank	~	Method 29	CVAA (SW-846 Method 7471A)	Reagent Blank	Only analyze if required	~	~
								-

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Table 4-4. Summary of Test Program Analyses

Γ	Ś								
1040F	Analyses	₩	-	-	~	~	~	~	-
Ç	Analyses	1	-	-	~	-	-	~	-
S	Analysis	Only analyze if required	One for test program	One for test program	One for test program	One for test program	One per batch/ matrix specific	One per batch/ matrix specific	One per batch/ matrix
	QC Analysis	Reagent Blank	Audit Sample	Audit Sample	Audit Sample	Audit Sample	rcs	Serial dilution	Method blank
Defending your control of the party of the p	Neierence Analytical	CVAA (SW-846 Method 7471A)	ICP (SW-846 Method 6010C)	ICP (SW-846 Method 6010C)	CVAA (SW-846 Method 7471A)	CVAA (SW-846 Method 7471A)	ICP (SW-846 Methods 6010C)		
Bofozogo	Preparation	Method 29	Method 29	Method 29	Method 29	Method 29	NA		
71013	rield Samples						∀ Z		
1	Sam	—	-	—	_	-	Υ Υ		
	Sample Matrix	Method 29 8N HCI reagent blank	Spike filter audit sample for metals	Liquid audit sample for metals	Filter with liquid ampoule audit sample for mercury	Liquid audit sample for mercury	Analytical system QC		
	Analysis	Metals by Method 29 (cont'd)							

Table 4-4. Summary of Test Program Analyses

Analysis		ü	Field	Poforonco	Poforonce Analytical		۲	J	Total
	Sample Matrix	Samples	ples	Preparation	Method	QC Analysis	Analysis	Analyses	Analyses
	Analytical system QC			NA V	CVAA (SW-846 Method 7471A)	rcs	One per batch/ matrix specific	1	~
·						Serial dilution	One per batch/ matrix specific	~	_
						Method blank	One per batch/ matrix specific	~	~
1	Method 26A H2SO4 impingers	က	ı	NA	Ion chromatography (EPA Method 26A/ SW-846 Method 9056A)	Duplicate	One for test program	~	9
						MS/MSD	1 per batch (assuming all samples batched together)	2	
	Method 26A H2SO4 reagent blank	1	~	NA	Ion chromatography (EPA Method 26A/ SW-846 Method 9056A)	Reagent Blank	One for test program	~	₩
	Method 26A NaOH impingers	က	ŀ	NA	lon chromatography (EPA Method 26A/ SW-846 Method 9056A)	Duplicate	One for test program	~	9
						MS/MSD	1 per batch (assuming all samples batched together)	2	
	Method 26A NaOH reagent blank	1	_	NA	Ion chromatography (EPA Method 26A/ SW-846 Method 9056A)	Reagent Blank	One for test program	1	1

Table 4-4. Summary of Test Program Analyses

			ם	able 4-4. Summary of	+-+. Summany of restriction Amanyses				
oioylou	Some Modern	Field	ple	Reference	Reference Analytical	Signature of	၁ဗ	gc	Total
Allalysis	Sample Maura	Sam	Samples	Preparation	Method	ac Allalysis	Analysis	Analyses	Analyses
CI ion by Method 26A	Method 26A deionized water reagent blank	-	-	NA	Ion chromatography (EPA Method 26A/ SW-846 Method 9056A)	Reagent Blank	One for test program	1	-
Cl- ion chromatography	Chloride in Audit Sample	1	-	NA	Ion chromatography (EPA Method 26A/ SW-846 Method 9056A)	Duplicate	One for test program	-	2
Cl-ion chromatography	Analytical system QC	۷ ۲	NA	NA	lon chromatography (EPA Method 26A/ SW-846 Method 9056A)	LCS/LCSD Method Blank	1 per batch following initial calibration (separate calibration for each matrix) One per batch/ matrix specific - analyzed in duplicate	1	r
TOTAL									113

ASTM - American Society for Testing and Materials

CVAA - cold vapor atomic absorption

EPA Method - New Source Performance Standards, Test Methods and Procedures, Appendix A, 40 CFR 60.

ICP - inductively coupled argon plasma

LCS - Laboratory Control Sample

MS - Matrix spike

MSD - Matrix spike duplicate

PDS - Post-digestion spike

NA - not applicable

SW846 - Test Methods for Evaluating Solid Waste, Third Edition, November 1986, and Updates.

Table 4-5. Summary of Laboratory Analytical Quality Control Checks, Frequencies, Acceptance Criteria, and Corrective Actions

Parameter/Method	Quality Control Check	Method of Determination	Frequency	Acceptance Criteria	Corrective Action
Heat Content (Btu) (ASTM D-240)	Laboratory Control Sample/Laboratory Control Sample Duplicate (LCS/LCSD)	LCS/LCSD analysis	Once per sample batch (maximum 20 samples)	Accuracy:%Recovery: 98 – 102%, Precision: RPD < 2%	Retest. If the retest fails, correct the cause and retest all samples.
	Duplicate Analyses	Duplicate analysis	Once prior to sample batch analysis (maximum 20 samples), minimum of 1 per set of test samples	Precision: RPD < 10%	(1) Flag data, (2) Discuss in report narrative
Density (ASTM D- 1475)	Laboratory Control Sample	LCS analysis	Once per sample batch (maximum 20 samples)	Accuracy: %Recovery: 99 – 101%	Determine cause and retest all
	Duplicate Analyses	Duplicate analysis	Once every 10 samples, minimum of 1 per set of test samples	Precision: < 10% RPD	Flag data
Percent Ash (ASTM D-482)	Continuing Calibration Blank (CCB) or Laboratory Method Blank (LMB)	CCB/LMB analysis	Once per sample batch (maximum 20 samples)	Concentrations: Result < Reporting Limit (RL) or < 0.1 sample result	Return all samples to muffle furnace and re-fire them. Reweigh after cooling.
	Laboratory Control Sample (LCS)	LCS analysis	Once per sample batch (maximum 20 samples)	Accuracy: %Recovery: 90 – 110%	Refire all samples and reanalyze if recovery exceeds upper limit.
	Laboratory Control Sample Duplicate (LCSD)	LCSD analysis	Once per sample batch (maximum 20 samples)	Accuracy: %Recovery: 90 – 110%, Precision: RPD ≤ 10%	Refire all samples and reanalyze. If RPD value continues to exceed 10%, reanalyze all samples.

Table 4-5. Summary of Laboratory Analytical Quality Control Checks, Frequencies, Acceptance Criteria, and Corrective Actions

Parameter/Method	Quality Control Check	Method of Determination	Frequency	Acceptance Criteria	Corrective Action
Percent Ash (ASTM D-482) (continued)	Duplicate Analyses	Duplicate analysis	Once prior to sample batch analysis (maximum 20 samples), minimum of 1 per set of test samples	Precision: RPD ≤ 10%	(1) Flag data (2) Discuss results in report narrative
Moisture Analysis (ASTM D-4928)	Laboratory Control Sample (LCS)	LCS analysis	Once per sample batch (maximum 20 samples)	Accuracy: %Recovery: 90 – 110%	Refire all samples and reanalyze if recovery exceeds upper limit.
	Duplicate Analyses	Duplicate analysis	Once per sample batch (maximum 20 samples)	Precision: RPD ≤ 35%	(1) Repeat analysis, (2) Flag data
Ultimate Analysis (ASTM D-3176)	Duplicate Analyses	Duplicate analysis	Once per sample batch (maximum 20 samples)	Precision: RPD ≤ 35%	(1) Repeat analysis, (2) Flag data
Chloride and Sulfate by Ion Chromatography (SW846 9056A)	Initial Calibration	4 standards bracketing expected concentrations Note: Separate calibrations are required for the acid and alkaline sample	Prior to sample analysis	Linear correlation coefficient >0.995	Recalibrate
	Retention time check for ion identification	Determine average retention time for ions of interest or relative retention time of several ions for every calibration curve	Prior to sample analysis	Average Retention Time - Sample identification is positive if results are within retention time window of standards	lons of interest are not present if criteria are not met.
				Relative Retention Time – Sample identification is positive if results are within 3 SD of average RRT	

Table 4-5. Summary of Laboratory Analytical Quality Control Checks, Frequencies, Acceptance Criteria, and Corrective Actions

Parameter/Method	Quality Control Check	Method of Determination	Frequency	Acceptance Criteria	Corrective Action
Chloride and Sulfate by Ion Chromatography (SW846 9056A)	Control check sample	Midlevel independent standard analyzed in duplicate	Beginning and end of each analysis period and after every 10 samples	90 – 110% of theoretical value	Repeat calibration check. If second check fails criteria, regenerate analytical system and reanalyze all samples since last acceptable calibration check.
	Reagent blank (ICB and CCBs)	Analysis of blanks	Immediately following the ICV and following each CCV. CCV performed after every 10 analyses.	Less than 1 mg/L	Contamination source must be found and corrected. All samples analyzed since the last acceptable CCB must be reanalyzed.
Metals by ICP or CVAA (SW846 6010C and SW846 7471A)	Initial calibration	Multiple standards (CVAA) or 1 standard (ICP) and a calibration blank, bracketing the expected concentrations. Critical level should be at least twice the lowest calibration standard.	Prior to sample analysis	Correlation coefficient of linear plot >0.995 (CVAA). Not applicable for ICP.	Recalibrate
	Reagent blank	Analysis of blank	After every 10 samples and at end of analysis	Less than instrument detection limit (IDL)	Reanalyze if greater than the reporting limit and discuss in case narrative if greater than the IDL
	Calibration check	Analysis of independent calibration check standard	Once after initial calibration	90 - 110% of theoretical value	Reanalyze and recalibrate, if necessary
	Post digestion spike	Analysis of post digest spike, spiked at 2 to 5 times the original sample value	One sample per test	85 - 115% of theoretical value	Flag data; discuss in case narrative

Table 4-5. Summary of Laboratory Analytical Quality Control Checks, Frequencies, Acceptance Criteria, and Corrective Actions

Parameter/Method	Quality Control Check	Method of Determination	Frequency	Acceptance Criteria	Corrective Action
Metals by ICP or CVAA (SW846 6010C and	Calibration accuracy (ICP only)	Reanalysis of high level standard	After every initial calibration	90 - 110% of theoretical value	Recalibrate and recheck
SVV 840 747 1A)	Interference check (ICP only)	Analysis of interference sample	After every initial calibration and at the end of each run	80 - 120% of theoretical value	Recalibrate and recheck
	Continuing calibration	Midlevel standard and blank	Beginning and end of each analysis period and after every 10 samples	ICP – Midlevel standard 90 - 110% of theoretical value; blank <50% of lowest calibration standard or within 3 SD of average blank.	Identify and correct problems; reanalyze samples run since last acceptable continuing calibration check.

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Table 4-6. Sampling Equipment Calibration Requirements

Stack Sampling Equipment	Acceptance Criteria	Measurement Frequency	Action If Criteria Are Not Met
	ν .	Volumetric Flow Measurements	
Type S pitot tube inspection	All dimension specifications met	Calibrate prior to test and visually inspect after each field test	Use pitot tubes that meet face opening specifications; repair or replace as required
Type S pitot tube calibration	± 3% for volumetric flows >1,000 fpm fpm ± 5-6% for volumetric flows >600 and <1,000 fpm	Refer to Section 10.0 of Method 2: If D _t is between 0.48 and 0.95 cm (3/16 and 3/8 in.), and if P _A and P _B are equal and between 1.05 and 1.50 D _t , there are two possible options: (1) the pitot tube may be calibrated according to the procedure outlined in Sections 10.1.2 through 10.1.5 of Method 2 before and after the test, or (2) a baseline (isolated tube) coefficient value of 0.84 may be assigned to the pitot tube with pitot tube inspection before and after the test.	Check for blockage. If blockage is significant, recalculate calibration coefficient.
Barometers	±0.1 inches Hg (± 2.5mm Hg) of mercury-in-glass barometer	Calibrate initially versus mercury-in-glass barometer; check before and after field test	Adjust to agree with a certified barometer
Stack gas temperature measurement system	Capable of measuring within ± 2°F (± 1°C) of mercury-in-glass thermometer	Calibrate prior to test and after each field use	Adjust to agree with Hg bulb thermometer; construct calibration curve, correct readings
Pressure sensors (excludes inclined manometer)	Agree within ± 5 percent of inclined manometers	Prior to and after field use	Adjust to agree with Hg bulb thermometer; construct calibration curve, correct readings
Wet test meter	$Y_{mi} = Y_m \pm 0.030 \text{ Y (before test)}$ Y_{mt}/Y_{mi} is 0.95 to 1.05 (after test)	Calibration prior to test Check calibration after test	Before test: Adjust until specifications are met. After test: Recalculate calibration coefficient.
Dry gas meters	$Y_i = Y \pm 0.02 \text{ Y (before test)}$ Ymf/Ymi is 0.95 to 1.05 (after test)	Calibration versus wet test meter initially, and when post-check exceeds $\Delta Y \pm 0.05$	Before test: Repair or replace and then calibrate After test: Recalculate calibration coefficient.

Table 4-6. Sampling Equipment Calibration Requirements

Stack Sampling Equipment	Acceptance Criteria	Measurement Frequency	Action If Criteria Are Not Met
Assembled isokinetic sampling train leakage	0.02 cfm (0.00057 m³/min) at vacuum of ≥ 10 inches Hg (250 mm Hg) before the start of stack	Just prior to start of first sampling traverse (required); after first sampling traverse (recommeded); after moving	Before stack sampling or at post port change: Isolate and repair leak point(s); repeat leak check.
	sampling, and > maximum vacuum value recorded during sampling run for post sampling leak checks	sampling train from first traverse port to second traverse port (recommended); end of second sampling traverse (required).	End of first traverse or end of second traverse: Determine leak rate; If <4% of sampling rate, correct sample volume per procedures in Section 6.3 of Method 5.
			If >4% of sampling rate, invalid test sample; discard sample and repeat sampling run for invalid train.
Tedlar bags	Any water manometer displacement after 10 minutes when inflated to 2 to 4 inches (5 to 10 cm) water column pressure	Every bag	Discard bag
Analytical balance	± 1 mg of Class-S weights	Check with Class-S weights upon receipt and daily	Adjust or repair
	Sampling Tr	Sampling Train Heating Systems and Thermocouples	
Probe heating system (isokinetic sampling trains)	Capable of maintaining 248° ± 25°F (120°C ± 14°C) at a flow of 0.75 cfm (21.2 L/min)	Calibrate initially by APTD-0576(11) or use published calibration curves	Repair, or replace, and then verify the calibration
Probe nozzle (isokinetic sampling trains)	Average of three ID measurements of nozzle within 0.001 inches (0.0025 mm); difference between high and low 0.002 inches (0.0050 mm)	Use a micrometer to measure to nearest 0.025mm (0.001 in.); check before and after field test	Recalibrate, reshape, and sharpen when nozzle becomes nicked, dented, or corroded
Thermocouples (stack gas meters and final impinger)	Impinger thermocouple ± 1°C (2°F) [Method 5]; dry gas thermocouple ± 3°C (5.4°F) [Method 0010]; stack thermocouple within ± 1°C(± 2°F) of absolute temperature.	Calibrate prior to test against a mercury thermometer	Adjust; determine a correction factor or reject

Table 4-6. Sampling Equipment Calibration Requirements

Sources

Quality Assurance Handbook for Air Pollution Measurement Systems, Volume III, Stationary Source Specific Methods, EPA-600/R-94/038c, September

Maintenance, Calibration, and Operation of Isokinetic Source Sampling Equipment, APTD-0576, U.S. EPA, Office of Air Programs, March 1972.

Construction of Isokinetic Source Sampling Equipment, APTD-0581, U.S. EPA, Office of Air Programs, April 1971.

Method 0030, Volatile Organics Sampling Train and Method 0010, Modified Method 5 Sampling Train, Test Method for Evaluating Solid Waste Physical/Chemical Methods (SW-846), U.S. EP A Office of Solid Waste, Update III, December 1997. <u>Determination of Particulate Emissions from Stationary Sources</u>, U.S. EPA, Test Methods and Procedures, New Source Performance Standards, 40 CFR 60, Appendix A.

5.0 TEST REPORT

The final test report will be postmarked before the close of business on the 60th day after completion of the test. The final test report will be a comprehensive test report that contains a discussion of the test objectives; sampling, analysis, and QA/QC activities performed; summaries of process operating conditions; and the results of the test determinations. The proposed report outline is presented in Table 5-1.

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Table 5-1. Example Test Report Outline

- 1.0 **EXECUTIVE SUMMARY**
 - 1.1 SUMMARY OF COMPLIANCE TEST RESULTS
 - 1.2 TEST PROTOCOL
 - 1.3 CONCLUSIONS
- 2.0 PROCESS DESCRIPTION
- 3.0 TEST PROGRAM OBJECTIVES AND TEST PROTOCOL
 - 3.1 TEST OBJECTIVES OVERVIEW
 - 3.2 TEST PROTOCOL
 - 3.3 TEST RESPONSIBLE PARTIES
 - 3.4 TEST CHRONOLOGY
 - 3.5 OPERATING DATA CONDITIONS SUMMARY
- 4.0 SAMPLING AND ANALYSIS RESULTS
 - 4.1 LIQUID AND GASEOUS FUEL SAMPLING AND ANALYSES
 - 4.2 METALS EMISSIONS RESULTS
 - 4.3 HCL/CL₂ EMISSIONS RESULTS
 - 4.4 CEMS MONITORING RESULTS FOR CARBON MONOXIDE
- TEST SUMMARY AND CONCLUSIONS 5.0
 - 5.1 METHOD 19 ANALYSIS
 - 5.2 CONCLUSIONS
- QUALITY ASSURANCE/QUALITY CONTROL RESULTS 6.0
 - 6.1 PROJECT PERSONNEL AND RESPONSIBILITIES
 - 6.2 QA/QC ACTIVITIES AND IMPLEMENTATION
 - 6.3 ANALYTICAL AUDITS
 - 6.4 DATA QUALITY
 - 6.5 CONCLUSIONS

APPENDICES

Appendix A	Process Operating Data
Appendix B	Stack Sampling Report

Appendix C Liquid Fuel Sampling Documentation

Appendix D **Example Calculations**

Field Notes and List of Samples Appendix E Appendix F **Summary Analytical Data**

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APPENDIX A SUMMARY SAMPLING PROCEDURES

Sampling Procedures

Table A-2.	Method 26A Stack Gas Hydrogen Halide and Halogen Samplin
	Procedure (Isokinetic)

Table A-3. Method 29 Stack Gas Multiple Metals Sampling Procedure

Liquid Fuel Sampling Procedure

Table A-1.

Sample Name: Liquid Fuel

Locations: Sample tap on fuel feed line

Equipment: 250 mL (~4 oz.) grab sample bottle

4 - liter amber jug

250 and 500 mL amber glass bottles with Teflon-lined screw lids Coolers for containing the composite sample between sampling events

Gloves, eye protection, splash protection, and respiratory protection (if needed).

Frequency: 30-minute intervals during each test run.

Procedures: At each time interval, purge the tap by allowing a small amount of liquid to flow into a

waste container. Using a 250 grab sample bottle, collect approximately 250 mL of sample and transfer the liquid to a 4-liter amber glass jug to build a run composite. Between grab samples, the 4-liter amber glass jug in which the run sample is being composited will be capped and maintained in a cooler. Record time each grab sample is

taken on sample collection sheet and approximate volume of final sample.

At the end of the run, vigorously mix the composite sample in the 4-liter amber jug and fill the test plan prescribed number 250 and 500 mL sample bottles from the 4-liter jug. Attach sample numbers to the samples with the sample name, date, run number, and time. The balance of the composite sample may be then discarded.

The Sample Custodian accepts custody of all samples and confirms the sample number and identities with the master samples list, request for analysis, and chain of custody. Samples are placed in shipping containers or coolers which are stored in the sample holding area separate from the stack gas samples.

References: ASTM E 300-86, Section 23 and 24, American Society for Testing and Materials, Annual Book of ASTM Standards, Philadelphia, Pennsylvania, Annual Series.

Sample name: Stack Gas Method 26A

Sampler: Stack sampling team

Locations: Exhaust stack

Equipment: Method 5 sampling train components assembled as shown in Figure 1; amber glass

sample jars with Teflon-lined lids; petri dishes with Teflon mat particulate filters [or quartz fiber filters if the stack gas temperature exceeds 410°F (210°C)]; balance; glass

graduated cylinder; ambient air conditioning tube containing Ascarite II.

Frequency: Continuous sampling for a minimum of 60 minutes during each test run.

Procedure Summary:

This procedure is used to determine hydrogen halide and halogen concentrations and emission rates when the source may contain liquid droplets, but is known <u>not</u> to emit acid particulate matter. The stack gases are sampled under isokinetic conditions to collect hydrogen halides and halogens in absorbing solutions and to determine the stack gas flow rate. The halide content of the absorbing solutions is determined by ion chromatography.

The Method 26A sampling train includes a glass probe nozzle, a heated sample probe with borosilicate or quartz glass probe liner, a quartz fiber or Teflon mat particulate filter, a Teflon filter frit, a condensate knockout impinger (optional), two acid impingers, an empty impinger (optional), two alkaline impingers, and a silica gel impinger. All impingers are placed in an ice bath.

All train components, reagents, and cleaning solutions are prepared according to the procedures specified in the methods referenced below. The pretest preparations include soaking the train glassware in hot (≥50°C) soapy water, rinsing it three times each with hot water, distilled/deionized water, and acetone, and allowing it to dry in air. When glassware is dry, all openings are covered with Teflon film until sample train assembly.

The sampling train is assembled in a clean area and train components are handled in a manner that minimizes potential for contamination from air or direct contact. A Teflon mat (or quartz fiber) filter is placed in the filter holder. When sampling a stack gas that has high moisture content, a condensate knockout impinger containing 50 ml of 0.1 N sulfuric acid may be included as the first impinger in the train. The next two impingers each contain 100 ml of 0.1 N sulfuric acid. An optional empty impinger may be placed after the sulfuric acid impingers to collect any liquid carryover. The next two impingers each contain 100 ml of 0.1 N sodium hydroxide solution. The last impinger is filled with 200 to 300 grams of indicating silica gel.

Stack sampling point locations are determined in accordance with Method 1. An initial traverse is made with a pitot tube at each sample point following Method 2 to establish stack gas velocity profile, temperature, and flow rate, and to check for cyclonic flow (cyclonic flow is checked only on the first day of testing). An Orsat analyzer or continuous emissions monitoring system (CEMS) is used to determine stack gas oxygen, carbon dioxide, and dry molecular weight according to either Method 3, 3A, or 3C procedures. The stack gas moisture content is determined according to Method 4. Pretest and post-test leak checks, isokinetic sampling rate, filter change outs (if needed), and data recording are performed according to Method 5 procedures.

The stack gas sampling is conducted following the general procedure given in Method 5 with the exception that the temperature of the sample probe and filter assembly is maintained at a minimum of $248^{\circ}F$ (instead of $248^{\circ}F \pm 25^{\circ}F$). As required by Method 5, the isokinetic sampling rate is maintained within $\pm 10\%$ of 100%.

After sampling, the probe is removed from the stack and the post-test leak check is performed. The filter surface is inspected for visible moisture which if present, is recovered by the procedure specified in Section 8.1.6 of Method 26A. While visible moisture is not expected to be present under normal sampling conditions, this procedure involves attaching the ambient air conditioning tube to the nozzle tip and operating the train for the time(s) needed to evaporate the moisture and to capture the gases in the impinger solutions.

The sampling train is allowed to cool and when the probe temperature is safe to handle, it is disconnected from the train and the inlet to the filter is cleaned and capped. The probe and the filter/impinger assembly are transported to the sample recovery area. The sample recovery and sampling train cleanup procedures are summarized below.

- Particulate Filter and Front Half Rinse The particulate matter filter is recovered and discarded. The probe and front half of the filter housing are rinsed with deionized water and the rinse is discarded.
- Acid Impinger Liquid -- The liquid contents of the condensate knockout impinger (if present), the two sulfuric acid impingers, and the initially empty impinger (if present), are measured to the nearest milliliter or weighed to the nearest 0.5 g and placed into a sample bottle (Container 3). Any condensate found in the back half of the particulate filter holder or the connecting glassware (or flexible tubing) is also measured or weighed and transferred to Container 3. The back half of the filter holder, acid impingers and all connecting glassware are rinsed with deionized water, and the rinses are added to Container 3. The final liquid level is marked on the sample container. Any color or film observed is noted on the sample recovery sheet.
- Alkaline Impinger Liquid -- The liquid contents of the sodium hydroxide impingers are measured to the nearest milliliter or weighed to the nearest 0.5 g and placed into a sample bottle (Container 4). The sodium hydroxide impingers and all connecting glassware are rinsed with deionized water, and the rinses are added to Container 4. A sodium thiosulfate solution is added to this container in the amount of 0.7 mg per ppm of halogen anticipated in the stack gas times the stack gas sample volume in dscf, (i.e., 0.7 mg/ppm-dscf). The container is sealed, shaken to mix and the final liquid level marked on the sample container. Any color or film observed is noted on the sample recovery sheet.
- Silica Gel -- The silica gel contents of the last impinger are weighed to the nearest 0.5 g. The color and condition of the silica gel is noted on the sample recovery sheet.

A schematic presentation of the above sample recovery and train cleanup procedures is provided in Figure 2. Following sample recovery, the train glassware may be reused at the same sampling location.

Samples of the absorbing reagent solutions equivalent to the amounts used in the sampling train are collected once during the test for reagent blanks. Deionized water

Table A-2. Method 26A Stack Gas Hydrogen Halide and Halogen Sampling Procedure (Isokinetic) (Continued)

from the wash bottle is added to these samples to bring the volumes up to the corresponding recovered sample volumes. The same ratio of sodium thiosulfate solution used for Container 2 is added to the sodium hydroxide solution blank. A blank sample of the deionized water in the wash bottle is also collected.

All of the sample containers are assigned numbers and labeled with the date and test-run number. The samples are turned over to the Sample Coordinator who records the appropriate data in the field logbook and packs the samples in insulated cold chests. Samples are stored in the sample holding area separate from the container supply area.

References:

"Method 26A - Determination of Hydrogen Halide and Halogen Emissions from Stationary Sources – Isokinetic Method."

"Method 1 - Sample and Velocity Traverses for Stationary Sources,

"Method 2 – Determination of stack Gas Velocity and Volumetric Flow Rate (Type S Pitot Tube)"

"Method 3 – Gas Analysis for Carbon Dioxide, Oxygen, Excess Air, and Dry Molecular Weight"

"Method 4 - Determination of Moisture Content in Stack Gases"

"Method 5 – Determination of Particulate Emissions from Stationary Sources"

Source: Appendix A - Test Methods, New Source Performance Standards, 40 CFR 60.

Table A-2. Method 26A Stack Gas Hydrogen Halide and Halogen Sampling Procedure (Isokinetic) (Continued)

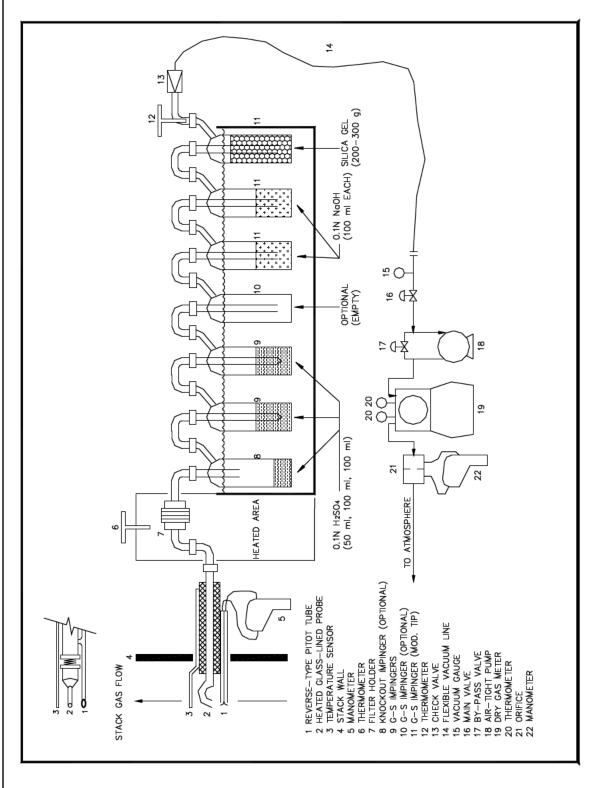


Figure 1. Method 26A Sample Train for Hydrogen Halide and Halogen

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Table A-2. Method 26A Stack Gas Hydrogen Halide and Halogen Sampling Procedure (Isokinetic) (Continued)

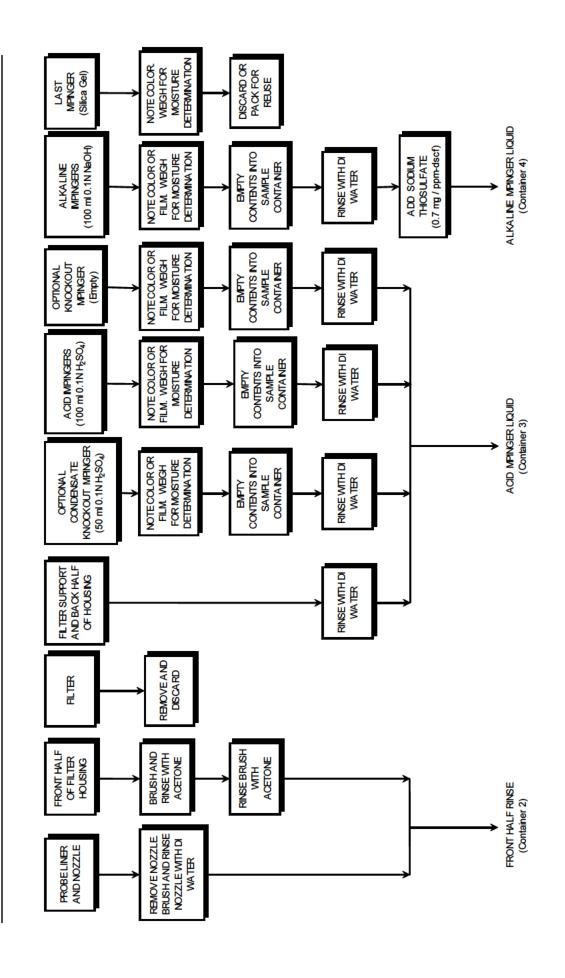


Figure 2. Method 26A Hydrogen Halide and Halogen Sample Recovery

Sample name: Stack Gas Method 29 (MM, Hg, No PM)

Sampler: Stack sampling team

Locations: Exhaust stack

Equipment: Method 29 multiple metals sampling train as shown in Figure 1 of this table; amber glass

sample jars with Teflon-lined lids; low-metals content particulate filters sealed in petri

dishes; balance; glass graduated cylinder.

Frequency: Sample a minimum of three (3) dry standard cubic meters of stack gas.

Procedure Summary:

Gases are sampled isokinetically to collect the metals on a particulate filter and in absorbing solutions. The filter and absorbing solutions are recovered for subsequent analyses by inductively coupled plasma emission spectroscopy (ICP) for non-mercury metals and cold vapor AAS (CVASS) for mercury. [Note: This procedure does <u>not</u> include collection of particulate matter (PM) samples for determination of PM concentration.]

The Method 29 multiple metals train includes a heated sample probe with borosilicate or quartz nozzle and liner, a heated particulate filter holder with Teflon filter support, a condensate knockout impinger (optional), two acidified hydrogen peroxide impingers, an empty impinger, two acidified potassium permanganate impingers, and a silica gel impinger. All impingers are placed in an ice bath. An air tight gas pump, dry gas meter, and manometer complete the train.

All train components, reagents, and cleaning solutions are prepared according to the procedures specified in the methods referenced below. The pretest preparations include rinsing the train glassware with hot tap water, washing with hot soapy water, rinsing three times each with tap water and distilled/deionized water (ASTM D1193-99, Type II). The glassware is then soaked in 10% (v/v) nitric acid solution for a minimum of four hours, rinsed three times with distilled/deionized water, a final rinse with acetone, and allowed to air dry. All glassware openings are covered with Teflon film until sample train assembly.

The sampling train is assembled in a clean area at the test site and the train components are handled in a manner that minimizes contamination from air or by direct contact. A clean and inspected filter is placed in the filter holder. The first impinger is initially empty and serves as a condensate knockout impinger. (This impinger may be eliminated if the moisture to be collected is less than 100 ml.) The next two impingers each contain 100 ml of 5% HNO₃/10% H₂O₂ solution. The carryover impinger is empty and the next two impingers each contain 100 ml of 4% KmnO₄/10% H₂SO₄ solution. The last impinger contains 200 to 300 g of indicating silica gel.

Stack sampling point locations are determined in accordance with Method 1. An initial traverse is made with a pitot tube at each sample point following Method 2 to establish stack gas velocity profile, temperature, and flow rate, and to check for cyclonic flow (cyclonic flow is checked only on the first day of testing). An Orsat analyzer is used to determine stack gas oxygen, carbon dioxide, and dry molecular weight according to Method 3 procedures. The stack gas moisture content is determined according to Method 4.

The sample train is operated according to Method 5 procedures for pretest and post-test leak-checks, isokinetic sampling rate, and data recording. The train leak rate must be the

less than 0.02 cfm, or less than 4% of the average sampling rate. The sampling rate is adjusted during the test to maintain isokinetic conditions at the probe nozzle within \pm 10% of 100%.

After sampling, the probe is removed from the stack and a post-test leak check (Method 5) is conducted. The probe nozzle is wiped to remove PM and covered loosely. After cooling, the probe is wiped to remove external PM, disconnected from the train, and both ends are capped. The probe and filter box-impinger assemblies are transported to the sample recovery area. The sample recovery and sample train cleanup are summarized below.

- Particulate Filter A non-metallic tool is used to remove the particulate filter from its holder and place it in the original petri dish (Container 1). A nylon bristle brush is used to remove any PM or filter fibers from the filter gasket or the holder onto the filter, and the petri dish is sealed with tape and placed in a plastic bag.
- Front Half Rinse -- The internal surfaces of the nozzle, probe, and front half of the filter holder are cleaned by rinsing, brushing, and final rinsing with exactly 100 ml of 0.1N nitric acid. All rinses are placed into an sample bottle (Container 3). [Note: There is no Container 2 (Acetone Rinse) because the collection of PM samples is not included in this procedure.]
- Acidified Peroxide Impingers -- The liquid contents of the condensate knockout impinger (if used) and the two HNO₃/H₂O₂ impingers are measured to the nearest milliliter or weighed to the nearest 0.5 g and placed into a sample bottle (Container 4). The back half of the particulate filter holder is inspected for condensate, which if observed, is measured and/or weighed as described above and transferred to Container 4. The back half of the filter holder, the filter support, the optional condensate knockout impinger (if used), and the two HNO₃/H₂O₂ impingers, and all connecting glassware are rinsed with exactly 100 ml of 0.1N nitric acid, and the rinses are added to Container 4.
- Carryover impinger The liquid contents of the initially empty impinger is measured to the nearest 0.5 ml and placed into a separate sample bottle (Container 5A). The impinger is then rinsed with exactly 100 ml of 0.10N nitric acid solution and the rinse is added to Container 5A.
- Acidified Permanganate Impingers -- The liquid contents of the acidified permanganate impingers are measured to the nearest 0.5 ml and placed into a separate sample bottle (Container 5B). These impingers and any connecting glassware are then rinsed a minimum of three times using a total of exactly 100 ml of fresh acidified potassium permanganate solution, and the rinses are added to Container 5B, being careful to also transfer any loose precipitated materials into the container. Triple rinsing of the acidified permanganate impingers is then repeated using a total of exactly 100 ml of water. The water rinses are also placed into Container 5B. If visible deposits remain in the impingers following the water rinses, they are rinsed with 25 ml of 8N hydrochloric acid, and the rinse is placed into a separate container (Container 5C) which contains 200 ml of water.

Silica Gel -- The silica gel contents of the last impinger are weighed to the nearest 0.5 g. The color and condition of the silica gel is noted on the sample recovery sheet.

A schematic diagram of the above sample train recovery and cleanup procedures is provided in Figure 2 of the table. Following sample recovery, the train may be reused at the same sampling location.

The following reagent blank samples are collected once during the test program: one unused particulate filter; 300 ml of the 0.1N nitric acid solution; 100 ml of the water used in sample recovery; 200 ml of the acidified hydrogen peroxide solution, and 100 ml of the acidified potassium permanganate solution. If the acidified potassium permanganate impingers from any run are rinsed with HCl, then a 25 ml blank sample of the 8N HCl solution is also collected and added to 200 ml of water in a separate sample bottle.

Once during the test program, one complete blank train is assembled in a clean area, brought up to operating temperatures, leak tested, and allowed to sit idle (no sample gas flow) for the duration of one sampling run. The train samples are recovered as described above and subsequently used in matrix spike and matrix spike duplicate analyses to evaluate conformance with analytical data quality objectives.

All of the sample containers are assigned numbers and labeled with the date and test-run number. The samples are turned over to the Sample Coordinator who records the appropriate data in the field logbook and pack the samples in insulated cold chests. Samples are stored in the sample holding area separate from the container supply area.

References:

- "Method 29 Determination of Metals Emissions form Stationary Sources."
- "Method 1 Sample and Velocity Traverses for Stationary Sources"
- "Method 2 Determination of stack Gas Velocity and Volumetric Flow Rate (Type S Pitot Tube)"
- "Method 3 Gas Analysis for Carbon Dioxide, Oxygen, Excess Air, and Dry Molecular Weight"
- "Method 4 Determination of Moisture Content in Stack Gases"
- "Method 5 Determination of Particulate Emissions from Stationary Sources"

Source: Appendix A, Test Methods and Procedures, New Source Performance Standards, 40 CFR 60.

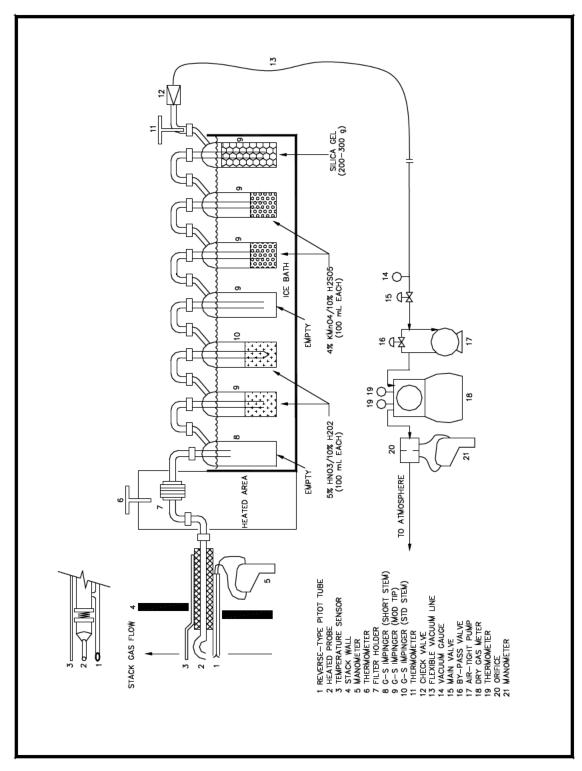


Figure 1. Method 29 Sample Train for Multiple Metals

DETERMINATION CONTENTS INTO RINSE 3X WITH CONTAINER WEIGH FOR 0.1N HNO₃ HNO₃/H₂O₂ IMPINGERS MOISTURE SAMPLE EMPT? PEROXIDE MPINGERS **DETERMINATION** CONTENTS INTO RINSE 3X WITH CONDENSATE CONTAINER WEIGH FOR 0.1N HNO₃ KNOCKOUT MOISTURE SAMPLE MPINGER (EMPTY) EMPT≺ NOTE USE EXACTLY 100 ML TOTAL FOR THESE RINSES AND BACK HALF FILTER SUPPORT RINSE 3X WITH

0.1NHNO₃

SEAL PETRI DISH WITH

ĭAÆ

DETERMINATION

DETERMINATION

DETERMINATION

WEGHFOR

WEGH FOR MOISTURE

WEIGH FOR

MOISTURE

NON-METALLIC TWEEZERS AND PLACE IN PETRI

REMOVE WITH

RINSEWITH 0.1N

Š H

BRUSH LOOSE

PARTICULATE ONTO FILTER

NONMETALLIC BRUSH AND RINSE 3X WITH

0.1NHNO

BRUSH WITH

MOISTURE

(SILICA GEL)

MPINGERS

ACIDIFIED

KNOCKOUT

(BMPTY)

OF HOUSING

FLTER

PROBELINER, NOZZLĘ

AND FRONT HALF OF (NOTE: USE EXACTLY

FILTER HOUSING

100 ML OF 0.1N HNO₃)

KMnO₄

MPINGER

AND CONDITION

CONTENTS INTO

CONTENTS INTO

EMPI→

CONTAINER

CONTAINER

SAMPLE MPI√

SAMPLE

DISCARD OR

PACK FOR

EXACTLY 100 ML ACIDIFIED KMnO₄

EXACTLY 100 ML

0.1N HNO₃

RINSE 3X WITH

SOLUTION

RINSE 3X USING

REUSE

NOTE COLOR OF SILICA GEL REMOVE ANY DEPOSITS WITH

EXACTLY 100 ML

WATER B

RINSE 3X USING

F NEEDED,

25 ML 8N HCL

RECOVER INTO

SAMPLE

HOLDING 200 ML

WATER

CONTAINER

KMnO₄ MPINGERS

HCL RINSE

MPINGERS (KMnO₄) **PERMANGA NATE**

MPINGER (HNO₃)

(HNO₃/H₂O₂)

≯HEE

FRONT HALF RINSE (3)

CARRYOVER

(₂C)

Table A-3. Method 29 Stack Gas Multiple Metals Sampling Procedure (Continued)

Page 5 of 5

Figure 2. Method 29 Multiple Metals Sample Recovery

WITH 0.1N HNO₃

RINSEBRUSH

RECOVER INTO

SAMPLE

CONTAINER

APPENDIX B EXAMPLE FORMS

Lyonde IIBase II Industries Channel view, TX

F-65630 COMPLIANCE TEST MASTER SAMPLE LIST

Test America, Knoxville, TN April 27, 2020

Sal	Field ample	Test	Run	Field Sample Test Run RFA/COC				Analytical	Analytical	
_	No.	No.	No.	No.	Sample Source	Sample Description	Sample Container	Parameters	Laboratory	QC Analysis
S-	8001	1	-	100	Liquid Fuel	Organic Liquid	500 mL amber bottle	Phys/Chem Properties TA, Knoxville	TA, Knoxville	DUP
S.	8002	1	-	002	Liquid Fuel	Organic Liquid	250 mL amber bottle	TSM & Hg	TA, Knoxville	DUP/MS/MSD
S-	8003	1	-	003	Liquid Fuel	Organic Liquid	500 mL amber bottle	Ultimate Analysis	TA, Knoxville	DUP
S-	8004	1	-	900	Method 26A	H2SO4 Impingers	1-Liter polyethylene bottle	Chloride Ion	TA, Knoxville	DUP
S-	8005	1	-	900	Method 26A	NaOH Impingers	500 mL polyethylene bottle Chloride lon		TA, Knoxville	DUP
S	8006	1	-	005	Method 29	M29 Particulate Filter	Petri dish	TSM & Hg	TA, Knoxville	
S	8007	1	-	005	Method 29	M29 Nitric Acid Probe Rinses	250 mL amber bottle	TSM & Hg	TA, Knoxville	
S	8008	1	-	005	Method 29	M29 Acidfied Peroxide Impingers	1-liter amber	TSM & Hg	TA, Knoxville	
S	8009	1	-	005	Method 29	M29 Empty Impinger	250 mL amber bottle	. BH	TA, Knoxville	
S-	8010	1	1	005	Method 29	M29 Acidfied Permanganate Impingers 500 mL amber bottle	500 mL amber bottle	Hg	TA, Knoxville	
ပ်	8011	1	-	002	Method 29	M29 Permanganate Imp. 8N HCI Rinse 250 mL amber bottle	250 mL amber bottle	Hg	TA, Knoxville	

Example Master Sample List

PROCESS SAMPLE DATA COLLECTION SHEET

Client:	LyondellBasell Industries		
Location:	Channelview, TX	Sheet No.:	1 of 1
Program:	F-65630 Compliance Test	Run Start:	0800
Stream:	Liquid Fuel	Run End:	1144
Sampler:	CEM	Sample Frequency:	30 minutes
Date:	5-May-20	Sample Type:	Grab/Composite
Test No.:	1	Equip/Container:	250 mL grab/ 4-L Composite
Run No.:	1	Source:	Tap on feed line

Sample No.	Sample Time	Comment	Sample No.	Sample Time	Comment
1	0800	Run Start 0800	26		
2	0830		27		
3	0900		28		
4	0930		29		
5	1000		30		
6	1030		31		
7	1100		32		
8	1130		33		
9	1200		34		
10	1230	Run End 1224	35		
11	\nearrow		36		
12			37		
13			38		
14			39		
15			40		
16			41		
17			42		
18			43		
19			44		
20			45		
21			46		
22			47		
23			48		
24			49		
25			50		

Total No. of Grabs:	10	
Comments/Notes:		
Sample aliquots for ana	alysis were prepared at the end of t	he test run from the homogenized composite sample.
The balance of the com	nposite remaining was discarded.	

Example Process Sampling Record

Isokinetic Stack Sample Data Collection Sheet

			SOMING IN STRUK	Jampie Dak	Somiletic Stack Sample Data Collection Silect		
Contract No.	P-001210		Method: Method 26A HCI/CI2	A HCI/CI2		Page 1 of 2	
Facility Lyonde	-yondell, Channelview, TX	v, TX	Initial Leak Rate (ft 3 @ in.Hg)	in.Hg)	<0.010 @ 10"	Operator R. DiRaimo	
Source F-65603	13		Final Leak Rate (ft ³ @ in.Hg)	in.Hg)	<0.010 @ 10"	Pitot No. K-004	
Date	5-May-20	Start Time		0800 Meter No.	1442 PTCF	PTCF 0.84	
Condition No.	1	End Time	1010	1010 DGMCF	1.004	1.004 Init. Pitot Leak Check	0.0
Run No.	1	Duration (min)	72 (nin	Ø H ∇	1.645	1.645 Final Pitot Leak Check (0.0
Stat. Press. (in. H ₂ O) -0.94	2O) -0.94	Bar. Press. (in.Hg)		29.73 Nozzle Dia.(")	(") 0.251 Kf		2.3

	(~ 7. · · · · ·)			(6)							i	
Point	Time	Volume	ΔР	Ч∇			Ter	Temperatures ((°F)			Vacuum
o N	(24 Hr)	(ft³)	(in. H ₂ O)	(in. H ₂ O)	Flue Gas	Probe	Filter	Impingers	Meter in	Meter Out	Cond. Exit	(in. Hg)
Start	0800	701.03										
1-1	9080	704.25	0.78	1.79	294	225	258	89	103	105	VΝ	0.9
1-1	0810	707.88	0.78	1.79	294	260	257	29	105	104		6.0
1-2	0815	711.50	0.74	0.17	294	257	261	99	106	104		0.9
1-2	0820	714.96	0.74	1.7	294	261	258	99	107	104		0.9
1-3	0825	718.09	89.0	1.56	292	258	257	64	108	104		0.9
1-3	0830	721.50	0.68	1.56	292	257	258	63	109	104		0.9
1-4	0835	725.36	0.77	1.77	292	258	258	29	104	104		6.0
1-4	0840	729.35	0.77	1.77	292	258	256	29	109	103		0.9
1-5	0845	733.20	0.77	1.7	293	258	259	61	110	104		0.9
1-5	0820	737.10	0.77	1.7	289	256	258	61	108	104		0.9
1-6	0855	740.67	0.74	1.43	285	259	255	62	106	105		0.9
1-6	0060	744.12	0.74	1.43	290	258	255	62	105	103		6.0
2-1	0910	747.21	0.62	1.77	292	255	259	99	96	92		7.0
2-1	0915	747.42	0.62	1.77	767	255	259	99	96	92		7.0
2-2	0920	750.94	0.62	1.77	292	259	257	92	96	62		7.0
2-2	0925	754.76	0.77	1.43	293	257	257	62	66	62		7.0
2-3	0630	758.21	0.77	1.29	289	257	258	62	102	92		6.0
2-3	0935	761.52	0.62	1.04	285	258	257	62	104	26		0.9
2-4	0940	764.52	0.56	1.1	290	257	253	61	106	66		7.0
2-4	0945	767.79	0.45	1.5	296	257	257	63	109	100		0.9
2-5	0360	767.92	0.56	1.5	296	253	258	64	111	101		0.9
2-5	0955	774.51	0.45	0.99	296	257	259	61	112	103		0.9
2-6	1000	777.52	0.44	0.94	297	258	258	09	112	104		5.5
2-6	1005	780.47	0.65	0.85	262	259	258	61	113	105		5.5
End	1010	783.55										
Comments:												

Example Stack Sampling Record

	LyondellBasell Channelview, X				
Sample Type:	Method 29 Filter	Sample No.:	S-8006		
Test No1	Run No1	Container(s):	1_ of1		
Analysis Required:	Total Select Metals and	d Mercury			
Analysis Laboratory:	Test America, Knoxville, TN				
Date:	5-May-20	Initials:	RD		
Time:	1330	Preservation:	NA		

Example Sample Label Format

Request for Analysis/Chain of Custody No. 004 Channelview, TX LyondellBasell

F-65630 Compliance Test Dr. William C. Anderson XXXXXX Chris E. McBride 865-694-7517 x3041 865-291-3010 865-291-3080 Patti Bales Test America Project Mgr: Test America Project No: Client Project Mgr: Test America Contact: Project Description: Client Project No.:

Data Package Due Date: 30 days from lab receipt Holding Time: 180 days from collection to analysis Analytical Due Date: 21 days from lab receipt

Test America-Knoxville Project No. XXXXXXX

Laboratory Deliverable Requirements

Test America-Knoxville Lot No. _H-XXXXXXXX

Analytical Testing QC Requirements:

Test America-Knoxville 5815 Middlebrook Pike Knoxville, TN 37921 Laboratory Destination (865)-291-3000 Laboratory Destination: Hand deliver

Courier:

Project Deliverables: Report analytical results on R-02 Reports and in data packages. Include "Field Number", "Sample Type", "test Number", and "Run Number" on all R-02 Reports.

TSM (As Be Cd Cr Ph Mn Se & Ni) 180 days from collection to analysis	
1014 (13, E3, C3, C1, 1.E), 1411, C3, C141)	ariarysis
Mercury (Hg) 28 days from collection to analysis	nalysis

	Project QC	Requirements						
		Analysis Specifications R	Method 29: Acid digest with probe rinse; Analyze for TSM & Hg	Method 29: Acid digest with filter; Analyze for TSM & Hg	Mothed 20: Acid diacet: Acclude for TOM 9 Ha	Intelliou 29. Acid digest, Alialyze for Tolvi & Fig.	Method 29: Actid algest, Arialyze for ISM & rig Method 29: Perpare & Analyze for Hg	Method 29: Perpare & Analyze for Hg Method 29: Perpare & Analyze for Hg Method 29: Perpare & Analyze for Hg
		Sample Description	M29 Particulate Filter	M29 Nitric Acid Probe Rinses		Mzs Acidiled Peroxide Impingers	M29 Acidiled Peroxide Impingers M29 Empty Impinger	M.29 Acidired Peroxide Impingers wetnod 29: Acid digest, Analyze for I Method 29: Perpare & Analyze for Hg M29 Acidifed Permanganate Impingers Method 29: Perpare & Analyze for Hg
		Sample Container	Petri dish	250 mL amber bottle	300	I-III atilipei	nber bottle	mber bottle mber bottle
	Sample	No. Collection Date	5-May-20 F	5-May-20	7 Nov-20		- (4	
	Run	Š	-	-	_		-	
	Test	<u>8</u>	_	-	_		-	
Field	Sample Test	è.	9008	8007	8008)	8009	8009
	Ø		ပ်	ပ်	က်		ဟ်	ဟ် ဟ်

Example Request for Analysis

Request for Analysis/Chain of Custody No. 004 LyondellBasell Channelview, TX

Sample Receipt L	og and Condition of the Samples U	Jpon Receipt		
Please fill in the foll	lowing information:		Comments	
(1)	Record the identities of any samples Request for Analysis form but were shipment	s that were listed on the	se write "NONE" if no comment is applica	ble.)
(2)	Record the sample shipping cooler t transporting samples listed on the Re			
(3)	Record any apparent sample loss or	r breakage.		
(4)	Record any unidentified samples tra shipment of samples.	insported with this		
(5)	Indicate if all samples were received project's required specifications (i.e.			
Custody Transfer				
Relinquished by:	Name	Commons	Date/Time	
Accepted by:		Company	·	
Relinquished by:	Name	Company	Date/Time	
Teiliquisiled by.	Name	Company	Date/Time	
Accepted by:	Name	Company	Date/Time	
Relinquished by:	Name	Company	Date/Time	
Accepted by:				
	Name	Company	Date/Time	

Example Chain of Custody

<u>Texas Commission on Environmental Quality (TCEQ)</u> <u>Source Testing Information Form</u>

E-mailed to: Mark Kolkmeier	Mark.kolkmeier@tceq.texas.gov	4 pages
Region 12 Stack Test Team	R12Stack@tceq.texas.gov	

The requested information will be reviewed by Regional Emissions Evaluator Team to determine if a Pretest meeting and/or test observation will be necessary. General guidance for major issues is also included. If you have any questions please contact the Region 12 office at Regional Phone # 713 767-3700

Returned To	Region 12 Stack Test Team	
From	Susie Geraci	
Date	January 30, 2020	4_Pages

Section I - SOURCE INFORMATION

Company	Lyondell Che	mical Co	mpany			
Contact/Title/Phone/ FAX/E-mail	Susie Geraci	Environ	mental Repr	esentative / 281-452-8866 /		
Facility Name	Lyondell Che	mical Co	mpany – Cha	annelview Facility		
Mailing Address	2502 Sheldon	Road ,Cl	nannelview,	TX		
Physical Address	same	same				
City (Source)	Channelview		County	Harris		
Account No.	HG-1575-W	Permit No.	2993	PSD No.		
Regulated Entity No.	RN10063365	0	Customer No.	CN600344402		

Section II - UNIT INFORMATION

Unit/EPN/ Testing Company	POSM I (EPN) EF65630		
Applicable Rules/Regs.	40 CFR 63 Subpart DDDD	DD and 30 TAC Chapter 113	
Testing Required By	40 CFR 63 Subpart 5D		
Construction Date		Operational Date	
Test Parameter(s)	See Test Plan- Performan	ice test for liquid fuel fired furnace per BMACT 5D	
Test Deadline Date	May 12, 2020 per 63.7515(b)		
Safety training/equipment required for unit entry	() Verbal briefing () Safety video (x) Hard hat () Other:	(x) Safety shoes(x) Safety glasses/side shields(x) Flame retardant clothing	

Section III - TEST PROTOCOL GUIDANCE

The following is a general checklist for items that should be included in the Test Protocol. This document must clearly describe the operational and testing procedures. Deviations from the Test Protocol may result in rejection of the results. Chapter 4 of the TCEQ Sampling Procedures Manual discusses the Test Protocol in more detail. Test Protocols must be received fourteen (14) days prior to a Pretest meeting. If you have any questions please contact the Region 12 office or the Engineering Services Team in Austin.

Confidential material should be clearly marked and submitted under a separate cover. All confidential material will be handled according to guidance in the <u>TCEQ Records Management Manual</u>.

Item #		(T)
1	Source Test company and Contact information (phone/FAX/E-mail).	See Test Plan
2	Proposed test method changes. Please note that changes must be submitted in writing to the Regional office no later than 30 days prior	See Test Plan

	to testing. Major changes will require written TCEQ or EPA approval prior to testing.		
3	Source/stack schematic illustrating sampling and CEMS locations, ports, and accessibility.	ing and CEMS locations, ports, (T)	
4	Process description/flow diagram detailing measurement sites, process controls monitoring devices, etc.	(T)	
5	Design, normal, maximum, and proposed unit operating rates, including conditions represented in the Permit Application.	(T)	
6	Design, normal, and maximum control device(s) operating rates, including conditions represented in the Permit Application.	(T)	
7	Unit/control device(s) operating parameters to be monitored, i.e., fuel flow meter, scale, etc.	(T)	
8	Proposed EPA, TCEQ, or alternative test methods.	(T)	

Section IV - GENERAL GUIDANCE

The following items are very brief discussions of topics which often present problems during the compliance testing and reporting process. If you have any questions please contact the Regional ** office or the Engineering Services Team in Austin.

Item #		(T)
9	Understanding and complying with regulatory, sampling, and reporting requirements is the responsibility of the Source.	(T)
10	It is the responsibility of the Source to insure the final report meets the requirements of Chapter 14 of the TCEQ Sampling Procedures Manual which is available from any Regional office.	
11	TCEQ requires separate filterable and condensible particulate matter emissions calculations in the test report.	
12	Field data sheets should be completely filled out by the end of each sample run.	
13	Although there is no requirement to calculate individual point isokinetics, excessive deviations may invalidate a sample run.	

14	If a unit is fueled with pipeline quality natural gas, one fuel analysis during <u>a test series</u> will be required. Any other fuel will require a minimum of one fuel analysis during <u>each</u> sample run.	
15	Parameters which determine production or operating rate shall be monitored <u>during</u> each sample run. Operating rates should not vary more than <u>+</u> 10% during a sample run or over the course of three sample runs.	
16	Calibration data for process instrumentation is required.	
17	Process data must be clearly labeled in the units of measure.	
18	Electronically recorded data must meet the functional traceability of data manually recorded in indelible ink.	
19	One NOx converter efficiency check is required for each source or daily if multiple sources are tested in a 24-hour period.	
20	All collected samples not analyzed on site require a Chain of Custody.	

FINAL INSTRUCTIONS

Submit this form, the Test Protocol and any proposed changes to the Emissions Evaluator Team in the Region 12 office. If a Pretest meeting is deemed necessary or specifically requested by the Facility, scheduling will be coordinated with the Facility Contact.

TCEQ Contact:

Mark Kolkmeier
Texas Commission on Environmental Quality
Stack Test Team Coordinator
Region 12 Air Section- Houston, TX
5425 Polk Ave Ste H
Houston, TX 77023-1452
T:713-767-3719
Mark.kolkmeier@tceq.texas.gov

SECTION V - SCHEDULING						
Notification Date	January 30, 2020	Protocol submittal Date	January 30, 2020			
Proposed Pretest Date	TBD	Proposed Test Date	Week of May 4, 2020			