

STACK EMISSIONS MONITORING REPORT



Unit 5 Crown Industrial Estate
Kenwood Road
Stockport
SK5 6PH
Tel: 0161 443 0980
Fax: 0161 443 0989

Your contact at ESG

Mark Woodruff
Business Manager - North
Tel: 0161 443 0982
Email: mark.woodruff@esg.co.uk

Operator & Address:

Qioptiq Ltd
Glascoed Road
St Asaph
Denbighshire
LL17 0LL

Permit:

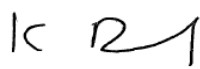
EPR Permit: EPR/BX4666IZ/V002

Release Point:

A1 - BP Plant (EA Permit JP3536LS)

Sampling Date(s):

18th November 2015

ESG Job Number:	LNO 12696
Report Date:	4th January 2016
Version:	1
Report By:	Dominic Houghton
MCERTS Number:	MM 04 529
MCERTS Level:	MCERTS Level 2 - Team Leader
Technical Endorsements:	1, 2, 3 & 4
Report Approved By:	Keith Bird
MCERTS Number:	MM 07 825
Business Title:	MCERTS Level 2 - Team Leader
Technical Endorsements:	1, 2 3 & 4
Signature:	



CONTENTS

EXECUTIVE SUMMARY

Stack Emissions Monitoring Objectives

- Plant
- Operator
- Stack Emissions Monitoring Test House

Emissions Summary

Monitoring Times

Process Details

Monitoring Methods

Analytical Methods

- Sampling Methods with Subsequent Analysis
- On-Site Testing

Sampling Location

- Sampling Plane Validation Criteria
- Duct Characteristics
- Sampling Lines & Sample Points
- Sampling Platform
- Sampling Location / Platform Improvement Recommendations

Sampling and Analytical Method Deviations

APPENDICES

APPENDIX 1 - Monitoring Schedule, Calibration Checklist & Monitoring Team

APPENDIX 2 - Summaries, Calculations, Raw Data and Charts

APPENDIX 3 - Measurement Uncertainty Budget Calculations

EXECUTIVE SUMMARY

MONITORING OBJECTIVES

Qioptiq Ltd operates a boron phosphide manufacturing process at St Asaph which is subject to EPR Permit EPR/BX4666IZ/V002, under the Environmental Permitting Regulations 2010.

ESG were commissioned by Qioptiq Ltd to carry out stack emissions monitoring to determine the release of prescribed pollutants from the following Plant under normal operating conditions.

The results of these tests shall be used to demonstrate compliance with a set of emission limit values for prescribed pollutants as specified in the Plant's EPR Permit, EPR/BX4666IZ/V002.

Plant

A1 - BP Plant (EA Permit JP3536LS)

Operator

Qioptiq Ltd
Glascoed Road
St Asaph
Denbighshire
LL17 0LL

EPR Permit: EPR/BX4666IZ/V002

Stack Emissions Monitoring Test House

ESG - Stockport Laboratory
Unit 5 Crown Industrial Estate
Kenwood Road
Stockport
SK5 6PH
UKAS and MCERTS Accreditation Number: 1015

Opinions and interpretations expressed herein are outside the scope of UKAS accreditation.
MCERTS accredited results will only be claimed where both the sampling and analytical stages are UKAS accredited.
This test report shall not be reproduced, except in full, without written approval of ESG.

EXECUTIVE SUMMARY

EMISSIONS SUMMARY					
Parameter	Units	Result	Calculated Uncertainty +/-	Limit	MCERTS accredited result
Phosphine	mg/m ³	<i>0.01</i>	0.02	-	✗
Diborane	mg/m ³	<i>0.03</i>	0.03	-	✗

ND = None Detected,

Results at or below the limit of detection are highlighted by bold italic text.

The above volumetric flow rate is calculated using data from the preliminary survey. Mass emissions for non isokinetic tests are calculated using these values. For all isokinetic testing the mass emission is calculated using test specific flow data and not the above values.

Reference conditions are 273K, 101.3kPa without correction for water vapour

EXECUTIVE SUMMARY

MONITORING TIMES			
Parameter	Sampling Date(s)	Sampling Times	Sampling Duration
Phosphine Run 1	18 November 2015	06:15 - 12:00	345 minutes
Diborane Run 1	18 November 2015	06:15 - 12:00	345 minutes

EXECUTIVE SUMMARY

PROCESS DETAILS

Parameter	Process Details
Description of process	Boron Phosphide Manufacturing
Continuous or batch	Batch
Product Details	Boron Phosphide
Part of batch to be monitored (if applicable)	Whilst running
Normal load, throughput or continuous rating	Normal throughput
Fuel used during monitoring	N/A
Abatement	None
Plume Appearance	None visible

EXECUTIVE SUMMARY

Monitoring Methods

The selection of standard reference / alternative methods employed by ESG is determined, wherever possible by the hierarchy of method selection outlined in Environment Agency Technical Guidance Note (Monitoring) M2. i.e. CEN, ISO, BS, US EPA etc.

MONITORING METHODS						
Species	Method Standard Reference Method / Alternative Method	ESG Technical Procedure	UKAS Lab Number	MCERTS Accredited Method	Limit of Detection (LOD)	Calculated MU +/- %
Phosphine	SRM - BS EN 13649	AE 118	1015	Yes	0.007 mg/m ³	127 %
Diborane	SRM - BS EN 13649	AE 118	1015	Yes	0.015 mg/m ³	118 %

EXECUTIVE SUMMARY

Analytical Methods

The following tables list the analytical methods employed together with the custody and archiving details:

SAMPLING METHODS WITH SUBSEQUENT ANALYSIS							
Species	Analytical Technique	Analytical Procedure	UKAS Lab Number	UKAS Accredited Lab Analysis	Analysis Lab (ESG or Subcontract)	Sample Archive Location	Archive Period
Phosphine	Wet chemistry	In House	-	No	SAL	SAL	3 months
Diborane	ICP-MS	In House	1015	No	ESG - Bretby	ESG - Bretby	3 months

ON-SITE TESTING							
Species	Analytical Technique	Analytical Procedure	UKAS Lab Number	MCERTS Accredited Analysis	Laboratory	Data Archive Location	Archive Period
-	-	-	-	-	-	-	-

EXECUTIVE SUMMARY

SAMPLING LOCATION					
Sampling Plane Validation Criteria	Value	Units	Requirement	Compliant	Method
Lowest Differential Pressure	-	Pa	≥ 5 Pa	-	BS EN 15259
Lowest Gas Velocity	-	m/s	-	-	-
Highest Gas Velocity	-	m/s	-	-	-
Ratio of Gas Velocities	-	: 1	$< 3 : 1$	-	BS EN 15259
Mean Velocity	-	m/s	-	-	-
Maximum angle of flow with regard to duct axis	-	°	$< 15^\circ$	-	BS EN 15259
No local negative flow	-	-	-	-	BS EN 15259

DUCT CHARACTERISTICS		
	Value	Units
Shape	Circular	-
Depth	0.25	m
Width	-	m
Area	0.05	m ²
Port Depth	0	mm

SAMPLING LINES & POINTS		
	Isokinetic	Non-Iso & Gases
Sample port size	-	1 inch port
Number of lines used	-	1
Number of points / line	-	1
Duct orientation	-	Vertical
Filtration	-	Out Stack

SAMPLING PLATFORM	
General Platform Information	
Permanent / Temporary Platform / Ground level / Floor Level / Roof	Ladder
Inside / Outside	Outside

M1 Platform requirements	
Is there a sufficient working area so work can be performed in a compliant manner	Yes
Platform has 2 levels of handrails (approximately 0.5 m & 1.0 m high)	No
Platform has vertical base boards (approximately 0.25 m high)	No
Platform has removable chains / self closing gates at the top of ladders	No
Handrail / obstructions do not hamper insertion of sampling equipment	No
Depth of Platform = $\geq$ Stack depth / diameter + wall and port thickness + 1.5m	Yes

Sampling Platform Improvement Recommendations (if applicable)

The sampling location should ideally be improved to meet all the requirements as specified in EA Guidance Note M1.

EXECUTIVE SUMMARY

Sampling & Analytical Method Deviations

Mass Emission

Under the work permit conditions, we are not allowed to be in the area of the sample location whilst the plant is running, therefore a flow traverse was not possible and a mass emission rate could not be calculated.

APPENDICES

CONTENTS

APPENDIX 1 - Monitoring Schedule, Calibration Checklist & Monitoring Team

APPENDIX 2 - Summaries, Calculations, Raw Data and Charts

APPENDIX 3 - Measurement Uncertainty Budget Calculations

APPENDIX 1 - Monitoring Schedule, Calibration Checklist & Monitoring Team

MONITORING SCHEDULE					
Species	Method Standard Reference Method / Alternative Method	ESG Technical Procedure	UKAS Lab Number	MCERTS Accredited Method	Number of Samples
Phosphine	SRM - BS EN 13649	AE 118	1015	Yes	1
Diborane	SRM - BS EN 13649	AE 118	1015	Yes	1

APPENDIX 1 - Monitoring Schedule, Calibration Checklist & Monitoring Team

CALIBRATEABLE EQUIPMENT CHECKLIST					
Extractive Sampling		Instrumental Analyser/s		Miscellaneous	
Equipment	Equipment I.D.	Equipment	Equipment I.D.	Equipment	Equipment I.D.
Control Box DGM	-	Horiba PG-250 Analyser	-	Laboratory Balance	-
Box Thermocouples	-	FT-IR	-	Tape Measure	LNO 24-DJH
Meter In Thermocouple	-	FT-IR Oven Box	-	Stopwatch	LNO 17-DJH
Meter Out Thermocouple	-	Bernath 3006 FID	-	Protractor	-
Control Box Timer	-	Signal 3030 FID	-	Barometer	LNO 08-DJH
Oven Box	-	Servomex	-	Digital Micromanometer	LNO 01-DJH
Probe	-	JCT Heated Head Filter	-	Digital Temperature Meter	LNO 03-DJH
Probe Thermocouple	-	Thermo FID	-	Stack Thermocouple	-
Probe	-	Stackmaster	-	Mass Flow Controller	LNO 29-13
Probe Thermocouple	-	FTIR Heater Box for Heated Line	-	MFC Display module	LNO 29-11
S-Pitot	-	Anemometer	-	1m Heated Line (1)	-
L-Pitot	-	Ecophysics NOx Analyser	-	1m Heated Line (2)	-
Site Balance	-	Chiller (JCT/MAK 10)	-	1m Heated Line (3)	-
Last Impinger Arm	-	Heated Line Controller (1)	-	5m Heated Line (1)	-
Dioxins Cond. Thermocouple	-	Heated Line Controller (2)	-	10m Heated Line (1)	-
Callipers	-	Site temperature Logger	-	10m Heated Line (2)	-
Small DGM	-		-	15m Heated Line (1)	-
Heater Controller	-		-	20m Heated Line (1)	-
Inclinometer (Swirl Device)	-		-	20m Heated Line (2)	-

NOTE: If the equipment I.D is represented by a dash (-), then this piece of equipment has not been used for this test.

CALIBRATION GASES					
Gas (traceable to ISO 17025)	Cylinder I.D Number	Supplier	ppm	%	Analytical Tolerance +/- %
-	-	-	-	-	-

STACK EMISSIONS MONITORING TEAM

MONITORING TEAM								
Personnel	MCERTS Number	MCERTS		TE / H&S Qualifications and Expiry Date				
		Level	Expiry	TE1	TE2	TE3	TE4	H&S
Dominic Houghton	MM 04 529	MCERTS Level 2	Dec-16	Dec-16	Dec-16	Mar-18	Dec-16	Sep-19
Mark Derbyshire	MM 07 824	MCERTS Level 1	Dec-17	-	-	-	-	Nov-18

APPENDIX 2 - Summaries, Calculations, Raw Data and Charts

PHOSPHINE SUMMARY					
Test	Sampling Times	Concentration mg/m ³	LOD mg/m ³	Limit mg/m ³	Emission Rate g/hr
Run 1	06:15 - 12:00 18 November 2015	<i>0.015</i>	0.007	-	-
Field Blank	-	0.01	-	-	-

Reference conditions are 273K, 101.3kPa without correction for water vapour

Please note figures in bold italic font are at the limit of detection

PHOSPHINE QUALITY ASSURANCE CHECKLIST

Leak Test Results	Mean Sampling Rate l/min	Pre sampling leak rate l/min	Post sampling leak rate l/min	Acceptable leak rate l/min	Leak Tests Acceptable?
Run 1	0.2	0.001	0.002	0.004	Yes

	Type of tube	Max. Tube Temperature °C	Max. Storage / Transit Temp. °C
Run 1	226-80	19	16

Tube sampling temperature should be < 40°C

PHOSPHINE ADSORPTION EFFICIENCY

Parameter	Total ug	Back ug	Adsorption Efficiency %	Acceptable Adsorption Efficiency %	Adsorption Efficiency Acceptable ?
Run 1	1	0.5	50	95	N/A - No ELV

N/A - As Phosphine does not have an emission limit an absorption efficiency is not required.

APPENDIX 2 - Summaries, Calculations, Raw Data and Charts

DIBORANE SUMMARY					
Test	Sampling Times	Concentration mg/m ³	LOD mg/m ³	Limit mg/m ³	Emission Rate g/hr
Run 1	06:15 - 12:00 18 November 2015	0.029	0.015	-	-
Field Blank	-	0.01	-	-	-

Reference conditions are 273K, 101.3kPa without correction for water vapour

Please note figures in bold italic font are at the limit of detection

DIBORANE QUALITY ASSURANCE CHECKLIST

Leak Test Results	Mean Sampling Rate l/min	Pre sampling leak rate l/min	Post sampling leak rate l/min	Acceptable leak rate l/min	Leak Tests Acceptable?
Run 1	0.2	0.002	0.003	0.004	Yes

	Type of tube	Max. Tube Temperature °C	Max. Storage / Transit Temp. °C
Run 1	Charcoal	19	16

Tube sampling temperature should be < 40°C

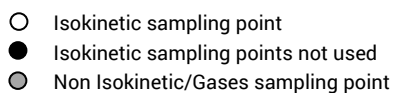
DIBORANE ADSORPTION EFFICIENCY

Parameter	Total ug	Back ug	Adsorption Efficiency %	Acceptable Adsorption Efficiency %	Adsorption Efficiency Acceptable ?
Run 1	2	1	50	95	N/A - No ELV

N/A - As Diborane does not have an emission limit an absorption efficiency is not required.

APPENDIX 2 - Summaries, Calculations, Raw Data and Charts

	Value	Units
Stack Depth	0.25	m
Stack Width	-	m
Area	0.05	m ²



Non-Isokinetic/Gases Sampling			
Sampling Point	Distance (% of Depth)	Distance into Stack	Units
A	50	0.13	m

[illegible]

APPENDIX 3 - Measurement Uncertainty Budget Calculations

MEASUREMENT UNCERTAINTY BUDGET - PHOSPHINE

Run	Sampled Volume m ³	Sampled Gas Temp K	Sampled Gas Pressure kPa	Sampled Gas Humidity % by volume	Oxygen Content % by volume	Limit of Detection % by mass	Leak %	Uncollected Mass mg
MU required	≤ 2%	≤ 2%	≤ 1%	≤ 1%	≤ 10%	≤ 5% of ELV	≤ 2%	≤ 10% of ELV
Run 1	0.000004	2	0.5	1	N/A	0.0005	-	-
as a %	0.0058	0.6849	0.5010	1.0000	N/A	N/A	1.0000	N/A
compliant?	Yes	Yes	Yes	Yes	N/A	N/A	Yes	N/A

Run	Volume (STP) m ³	Mass of Phosphine mg	O ₂ Correction -	Leak mg/m ³	Uncollected Mass mg	Lab Uncertainty mg	Combined uncertainty
Run 1	0.064	0.001	1.000	0.0001	0.0003	-	-
MU as mg/m ³	0.004	0.007	-	0.0001	0.004	0.001	0.009
MU as %	24.725	50.000	-	0.577	28.868	10.000	-

R1 - Uncertainty expressed at a 95% confidence level (where k = 2)	0.019	mg/m³	127	%
---	--------------	-------------------------	------------	----------

Developed for the STA by R Robinson, NPL

APPENDIX 3 - Measurement Uncertainty Budget Calculations

MEASUREMENT UNCERTAINTY BUDGET - DIBORANE

Run	Sampled Volume m ³	Sampled Gas Temp K	Sampled Gas Pressure kPa	Sampled Gas Humidity % by volume	Oxygen Content % by volume	Limit of Detection % by mass	Leak %	Uncollected Mass mg
MU required	≤ 2%	≤ 2%	≤ 1%	≤ 1%	≤ 10%	≤ 5% of ELV	≤ 2%	≤ 10% of ELV
Run 1	0.000004	2	0.5	1	N/A	0.001	-	-
as a %	0.0058	0.6849	0.5010	1.0000	N/A	N/A	1.5000	N/A
compliant?	Yes	Yes	Yes	Yes	N/A	N/A	Yes	N/A

Run	Volume (STP) m ³	Mass of Diborane mg	O ₂ Correction -	Leak mg/m ³	Uncollected Mass mg	Lab Uncertainty mg	Combined uncertainty
Run 1	0.064	0.002	1.000	0.000	0.001	-	-
MU as mg/m ³	0.002	0.014	-	0.000	0.008	0.003	0.017
MU as %	6.307	50.000	-	0.866	28.868	10.000	-

R1 - Uncertainty expressed at a 95% confidence level (where k = 2)	0.034	mg/m³	118	%
---	--------------	-------------------------	------------	----------

Developed for the STA by R Robinson, NPL

APPENDIX 3 - Measurement Uncertainty Budget Calculations

END OF REPORT