

STACK EMISSIONS MONITORING REPORT



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Operator & Address:

Qioptiq Ltd
Glascoed Road
St Asaph
Denbighshire
LL17 0LL

Permit:

EPR Permit: JP3536LS

Release Point:

A1 - BP Plant

Sampling Date(s):

18th November 2014

ESG Job Number:	LNO 12115
Report Date:	19th December 2014
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:	Mark Woodruff
MCERTS Number:	MM 03 164
Business Title:	MCERTS Level 2 - Business Manager
Technical Endorsements:	1, 2, 3 & 4
Signature:	



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EXECUTIVE SUMMARY

MONITORING OBJECTIVES

Qioptiq Ltd operates a boron phosphide manufacturing process at St Asaph which is subject to EPR Permit JP3536LS, under the Environmental Permitting Regulations 2010.

Environmental Scientifics Group Limited 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, JP3536LS.

Plant

A1 - BP Plant

Operator

Qioptiq Ltd
Glascoed Road
St Asaph
Denbighshire
LL17 0LL

EPR Permit: JP3536LS

Stack Emissions Monitoring Test House

Environmental Scientifics Group Limited - 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.

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EXECUTIVE SUMMARY

EMISSIONS SUMMARY					
Parameter	Units	Result	Calculated Uncertainty +/-	Limit	MCERTS accredited result
Phosphine	mg/m ³	<i>0.01</i>	0.02	-	x
Phosphine Emission Rate	g/hr	-	-	-	
Diborane	mg/m ³	<i>0.01</i>	0.02	-	x
Diborane Emission Rate	g/hr	-	-	-	
Stack Gas Temperature	°C	-	-	-	x
Stack Gas Velocity	m/s	-	-	-	
Gas Volumetric Flow Rate (Actual)	m ³ /hr	-	-	-	
Gas Volumetric Flow Rate (STP, Wet)	m ³ /hr	-	-	-	
Gas Volumetric Flow Rate (STP, Dry)	m ³ /hr	-	-	-	
Gas Volumetric Flow Rate at Reference Conditions	m ³ /hr	-	-	-	

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 2014	06:20 - 13:30	360 minutes
Diborane Run 1	18 November 2014	06:20 - 13:30	360 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 Environmental Scientifics Group Limited 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	132.7 %
Diborane	SRM - BS EN 13649	AE 118	1015	Yes	0.007 mg/m ³	133.3 %

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 13284-1
Lowest Gas Velocity	-	m/s	-	-	-
Highest Gas Velocity	-	m/s	-	-	-
Ratio of Gas Velocities	-	: 1	$< 3 : 1$	-	BS EN 13284-1
Mean Velocity	-	m/s	-	-	-
Maximum angle of flow with regard to duct axis	-	°	$< 15^\circ$	-	BS EN 13284-1
No local negative flow	-	-	-	-	BS EN 13284-1

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 (CEN Methods)	Isokinetic (ISO Methods)	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 = $>$ 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.

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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	LNO 13-01	Horiba PG-250 Analyser	-	Laboratory Balance	LNO 00-12/00-13
Box Thermocouples	LNO 03-01	FT-IR	-	Tape Measure	LNO 24-DJH
Meter In Thermocouple	LNO 03-01	FT-IR Oven Box	-	Stopwatch	LNO 17-DJH
Meter Out Thermocouple	LNO 03-01	Bernath 3006 FID	-	Protractor	-
Control Box Timer	LNO 17-01	Signal 3030 FID	-	Barometer	LNO 08-DJH
Oven Box	LNO 13-12	Servomex	-	Digital Micromanometer	LNO 01-DJH
Probe	LNO 11-25	JCT Heated Head Filter	-	Digital Temperature Meter	LNO 03-DJH
Probe Thermocouple	LNO 10-25	Thermo FID	-	Stack Thermocouple	-
Probe	-	Stackmaster	-	Mass Flow Controller	LNO 29-67/29-65
Probe Thermocouple	-	FTIR Heater Box for Heated Line	-	MFC Display module	LNO 29-08
S-Pitot	LNO 06-DJH	Anemometer	-	1m Heated Line (1)	-
L-Pitot	-	Ecophysics NOx Analyser	-	1m Heated Line (2)	-
Site Balance	LNO 14-DJH	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	LNO 31-DJH	Site temperature Logger	-	10m Heated Line (2)	-
Small DGM	-		-	15m Heated Line (1)	-
Heater Controller	-		-	20m Heated Line (1)	-
Inclinometer (Swirl Device)	LNO 23-DJH		-	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 +/- %
-	-	-	-	-	-

APPENDIX 1 - Monitoring Schedule, Calibration Checklist & Monitoring Team

STACK EMISSIONS MONITORING TEAM

Team Leader

Dominic Houghton
MCERTS Level 2, Technical Endorsements 1, 2, 3 & 4
MM 04 529
MCERTS Expiry Date - Dec 2016
H&S Expiry Date - Sep 2014

Technician

Mark Derbyshire
MCERTS Level 1, Technical Endorsement 1
MM 07 824
MCERTS Expiry Date - Dec 2017
H&S Expiry Date - Apr 2017

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	06:20 - 13:30 18 November 2014	0.014	0.007	-	-
Field Blank	-	0.007	-	-	-

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	0.002	0.002	0.004	Yes

	Type of tube	Max. Tube Temperature °C	Max. Storage / Transit Temp. °C
Run 1	Mercuric Cyanide Treated Silica	12	18

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	ND	ND	100	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	06:20 - 13:30 18 November 2014	0.014	0.007	-	-
Field Blank	-	0.007	-	-	-

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	Pre sampling leak rate	Post sampling leak rate	Acceptable leak rate	Leak Tests Acceptable?
	l/min	l/min	l/min	l/min	
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	Treated Silica	12	18

Tube sampling temperature should be < 40°C

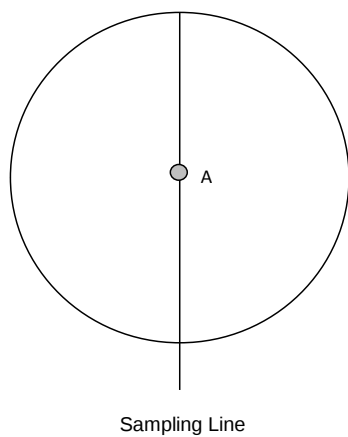
DIBORANE ADSORPTION EFFICIENCY

Parameter	Total ug	Back ug	Adsorption Efficiency %	Acceptable Adsorption Efficiency %	Adsorption Efficiency Acceptable ?
Run 1	ND	ND	100	95	N/A - No ELV

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

STACK DIAGRAM

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



- Isokinetic sampling point
- Isokinetic sampling points not used
- Non Isokinetic/Gases sampling point

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

[illegible]

SAMPLING LOCATION



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 %	0.0056	0.7018	0.4998	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	0.068	0.001	1.000	0.000	0.000	-	-
MU as mg/m ³	0.004	0.007	-	0.000	0.004	0.001	0.009
MU as %	31.118	50.000	-	0.577	28.868	10.000	-

R1 - Uncertainty expressed at a 95% confidence level (where k = 2)	0.019	mg/m³	132.69	%
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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	0.0005	-	-
as a %	0.0056	0.7018	0.4998	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 Diborane mg	O ₂ Correction -	Leak mg/m ³	Uncollected Mass mg	Lab Uncertainty mg	Combined uncertainty
Run 1	0.068	0.001	1.000	0.000	0.000	-	-
MU as mg/m ³	0.004	0.007	-	0.000	0.004	0.001	0.009
MU as %	31.749	50.000	-	0.577	28.868	10.000	-

R1 - Uncertainty expressed at a 95% confidence level (where k = 2)	0.019	mg/m³	133.29	%
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Developed for the STA by R Robinson, NPL

END OF REPORT