Wicklow County Council
Arklow Dredging
Annual Environmental Report 2015

224515-00_2016.05.18_ Environmental Report

Issue | 18 May 2016

Job number 224515-00

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Document Verification

ARUP

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		Name			
		Signature			

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1 Introduction

The Dumping at Sea Permit S0002-01 was granted by the EPA in April 2011 for the dredging and disposal works at Arklow Harbour.

The requirement for the Annual Environmental Reporting is outlined in Condition 7 of this permit.

This report has been prepared to satisfy this condition in the DaS licence for dredging and disposal works undertaken at Arklow Harbour during 2015. The methodology for the development of the ELRA follows EPA guidance and it has been prepared by Arup, an independent and appropriately qualified consultant.

2 Register of Loading and Dumping Activities

2.1 Summary

No loading and dumping activities were undertaken during 2015.

3 OSPAR Dumping Report

Included in Appendix B

4 Report Incidents Summary

Date	Incident description (CAUSE)	Impact (EFFECT)	List of Authorities contacted	Corrective action	Success of the corrective action	Incident Closed (Y/N)
No reported incidents occurred in 2015						

5 Complaints Summary

Date of complaint	**Name of complaint**	**Nature of the complaint**	**Response made to the complainant**	**Complaint Closed (Y/N)**
No complaints were received in 2015				

6 Monitoring Summary

6.1 Overview

Environmental Monitoring was undertaken by Hydrographic Surveys Ltd in Arklow Harbour and at the dredging disposal site off the coast of Arklow, Co. Wicklow.

The monitoring involved a bathymetric survey, bottom sampling and side scan sonar survey. All were undertaken on the 1 September 2015.

The main objectives of the monitoring were to:

- Establish the profile of the disposal site and the surround area
- Locate, if any, possible obstructions on the seabed
- Determine the characteristics of the sediment in the area

The bathymetric and side scan surveys extended to 500m north and south and 250m east and west of the disposal site. The sediment sampling was undertaken at various locations:

- Harbour Upstream
- Harbour Dock
- Harbour Downstream
- Disposal Pit

6.2 Bathymetric Survey

A bathymetric survey was undertaken in dump site with all levels reduced to Chart Datum. Tidal variations were recorded every five minutes during the survey using a Valeport 740 model vented tide gauge. Seabed levels were recorded both digitally and in analogue format using the Navisount 210 dual frequency echo sounder. Line spacing for the survey was at approximately 25m intervals and were perpendicular to the coastline as per the requirements of the survey.

The results of the Bathymetric survey show the site is sloping towards the seaward side from a high of 3.9m in the North West corner to a depth of 12.2 in the south west corner as shown in Drawing HS 108-1/15. The disposal site can be seen in the centre of the surveyed area. The shallowest area of the pit is in the north east corner where the seabed rises to 4.6m CD. The Bathymetric survey results are similar to the 7/10 Day Monitoring survey.

The bathymetric surveys are included in Appendix A

6.3 Side Scan Survey

A side scan survey was undertaken to identify any obstructions which may be present on the seabed. Survey lines were undertaken at 50m intervals perpendicular to the shoreline. As per the requirements of the survey.

The side scan survey results were very similar to the 7/10 Day monitoring results as shown in Drawing HS 113/15. The majority of the site showed up light grey in colour indicating a smooth sea bed with some small dark areas showing the presence of small rocks and boulders.

The side scan survey is included in appendix A.

6.4 Sediment Sampling

Sediment sampling was undertaken on 13 samples from the river, harbour and disposal site. The samples were taken using a stainless steel Van Veen sampler.

6.4.1 Harbour River

Sediment sampling was undertaken on the 1 September 2015. A total of 5 samples were taken from the Harbour River. The sediment samples were recorded as being black clay and silt with a foul odour and with no visual form of life.

Table 1 shows the range of grain size distributions encountered as a percentage in the five samples.

Table 1: Grain size distribution Harbour River

Particle Size	**Range (%)**
>2mm %	0-51.75
<2mm >63um	36.77-81.81
<63um	2.96-60.01

The organic content of the five sediment samples varied from 0.48 % to 8.30 %.

The chemical analysis results show the sediment is chemical parameters are all below the Dumping at Sea limits and therefore it is assumed they pose no threat to the surrounding environment.

6.4.2 Harbour Dock

Sediment sampling was undertaken on the 1 September 2015. A total of 5 samples were taken from the Harbour Dock. The sediment samples were recorded as consisting of medium and course sands with no visual life forms present.

Table 2 shows the range of grain size distributions encountered as a percentage in the five samples.

Table 2: Grain size distribution Harbour Dock

Particle Size	**Range (%)**
>2mm %	0 - 33.23
<2mm >63um	55.04 - 99.99
<63um	0 – 11.26

The chemical analysis results show the sediment is chemical parameters are all below the Dumping at Sea limits and therefore it is assumed they pose no threat to the surrounding environment.

6.4.3 Disposal Pit

Sediment sampling was undertaken on the 1 September 2015. A total of 2 samples were taken from the Harbour Dock. The sediment samples were recorded as consisting of black clay and silt with a foul odour and no visual sign of life forms.

Table 3 shows the range of grain size distributions encountered as a percentage in the five samples.

Table 3: Grain size distribution Disposal Pit

Particle Size	**Range (%)**
>2mm %	0
<2mm >63um	37.16 – 42.35
<63um	56.23 – 61.69

The organic content of the five sediment samples varied from 4.64 % to 5.28 %.

The chemical analysis results show the sediment is chemical parameters are all below the Dumping at Sea limits and therefore it is assumed they pose no threat to the surrounding environment.

7

7.1 Annual Statement of Measures

7.1.1 Measures Taken to Prevent Environmental Damage and Remedial Actions

Environmental monitoring of the harbour and the disposal site are being undertaken annually to ensure no negative environmental impacts occur within the area. The monitoring includes a bathymetric survey, a sine scan survey and sediment sampling. The sediment sampling includes both physical and chemical sampling of a range of sediment samples from various locations in the harbour and disposal site.

Appendix A

Surveys

A1 Bathymetric Surveys

SITE:

Arklow Disposal Site

Sheet Title:

1 Month Monitoring Survey of Arklow Overview Site

CLIENT:

Wicklow Co Co

Consulting Engineers:

J P Byrne Consulting Engineers & ARUP

SURVEYED BY:

HYDROGRAPHIC SURVEYS LTD.
The Cobbles
Crosshaven
Co. Cork
tel: +353 21 4831184

JOB NUMBER:

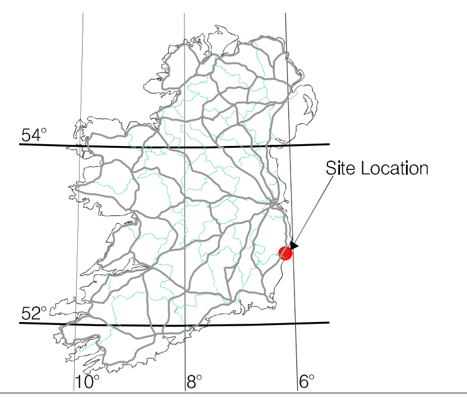
PH 15021

DRAWN BY: James Coade	DRAWING NUMBER: HS 108-1/15
SCALE: 1:2000 ON A1	APPROVED: J.B.J.
REVISION: D01	SURVEY DATE: 01/09/2015

Memoir

1. Constructed on Irish National Grid.
2. Horizontal control by D.G.P.S.
3. Soundings in metres and decimetres reduced to Chart Datum (C.D.)
4. WGS84 Latitude/Longitude lines shown.

Hydrographic Surveys Site



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Hydrographic Surveys Ltd.

SITE:

Arklow Disposal Site

Sheet Title:

1 Month Monitoring Survey of Arklow Disposal Site

CLIENT:

Wicklow Co Co

Consulting Engineers:

J P Byrne Consulting Engineers & ARUP

SURVEYED BY:

HYDROGRAPHIC SURVEYS LTD.
The Cobbles
Crosshaven
Co. Cork
tel: +353 21 4831184

JOB NUMBER:

PH 15021

DRAWN BY: James Coade	DRAWING NUMBER: HS 108-2/15
SCALE: 1:500 ON A1	APPROVED: J.B.J.
REVISION: D01	SURVEY DATE: 01/09/2015

Memoir

1. Constructed on Irish National Grid.
2. Horizontal control by D.G.P.S.
3. Soundings in metres and decimetres reduced to Chart Datum (C.D.)
4. WGS84 Latitude/Longitude lines shown.

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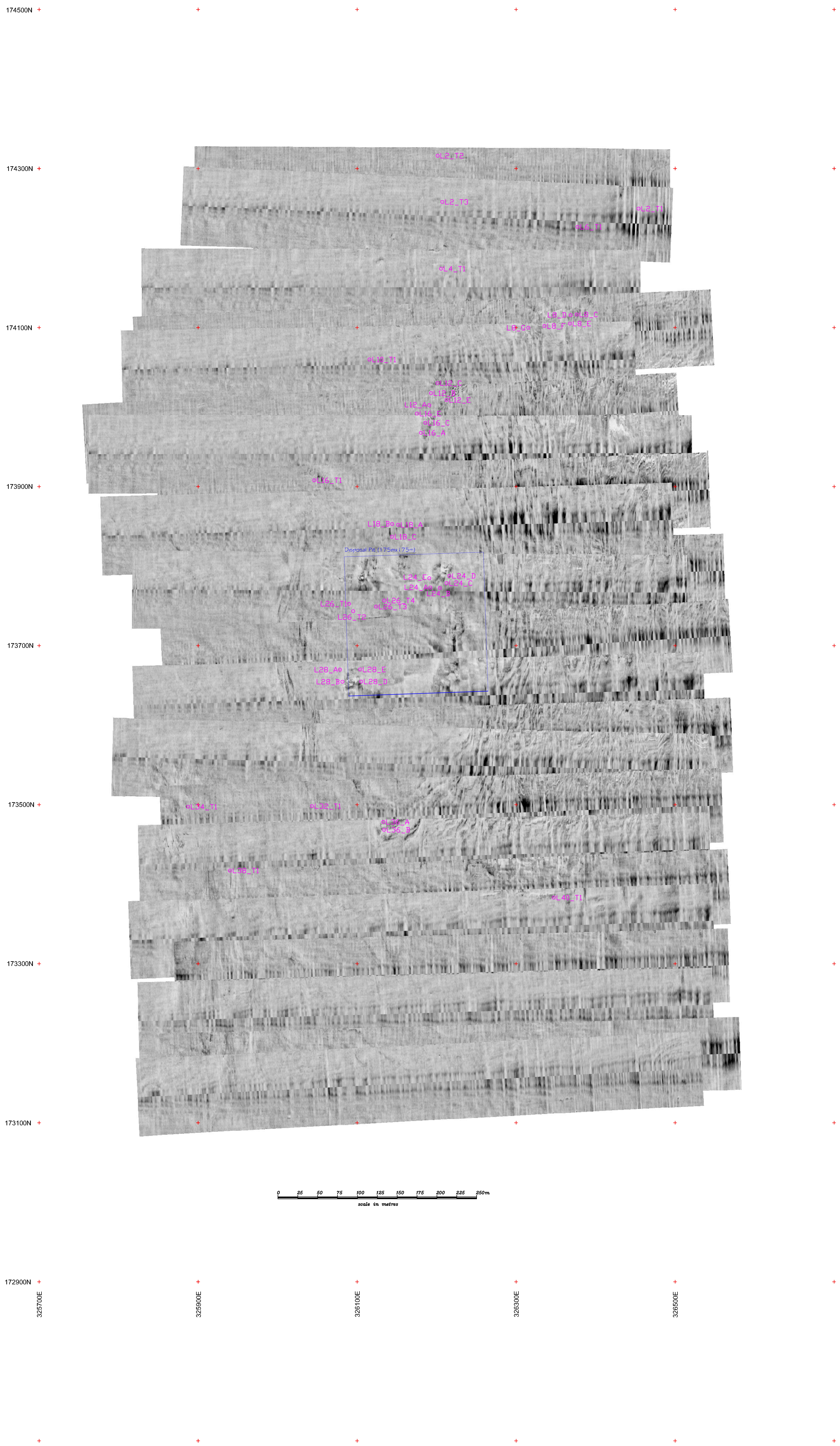
Hydrographic Surveys Ltd.

A2 Side Scan Sonar



SITE:

Arklow, Co. Wicklow

Sheet Title:

7/10 Day Monitoring
Side Scan Sonar Survey of
Arklow Dump Site

CLIENT:

Wicklow County Council

CONSULTING ENGINEERS:

ARUP Consulting Engineers
J P Byrne Consulting Engineers

SURVEYED BY:

HYDROGRAPHIC SURVEYS LTD
The Cobbles,
Crosshaven,
Co. Cork

tel: +353 21 4831184

JOB NUMBER:

PH15021A

DRAWING NUMBER:

HS 113/15

DRAWN BY:

James Coade

DATE:

01/09/2015

SCALE:	APPROVED:
1:2500 ON A1	J.B.J.

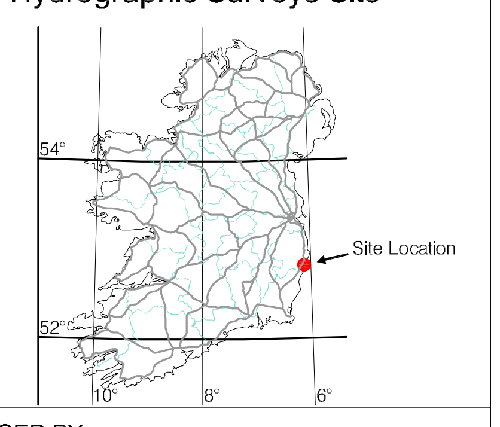
REVISION:

D01

Memoir

1. Constructed on Irish National Grid.
2. Horizontal control by D.G.P.S.
3. Soundings in metres and decimetres reduced to Chart Datum.
4. Interpreted side scan sonar Targets shown thus: ○ L1_T1
5. Side scan mosaic shown for presentation only, image may contain warping/stretching. Targets have been identified from processed unwarped data.
6. Results are based on interpretation of geophysical data and experience of author.

Hydrographic Surveys Site



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Appendix B

OSPAR Return

S0002 - 01 OSPAR RETURN 2014

		Additional comments
Permit No.	S0002-01	
Date of issue	21st April 2011	
Amount permitted (tonnes)	60,000 tonnes	
Capital or maintenance?	Maintenance	
Method of dredging	Backhoe	
Dumpsite	0.75km offshore from Arklow	

WASTES DUMPED

If permit relates to dredge sediments please complete section A. Otherwise, complete section B

A. DREDGED SEDIMENTS

(a)	Specification of waste (e.g. mud, silt, sand etc.)	N/A	
(b)	Derived from harbour, estuary or open water?	N/A	
(c)	Total quantity of wastes dumped (tonnes)?	0 tonnes	
(d)	Details of any chemical, toxicity and/or other testing carried out on these sediments. (Reports to be attached as separate documents. Include report references, date of analysis, contractors.)	N/A	
(e)	Please provide a map of the area dredged with chemical analysis sampling stations indicated. Please include details of quantities dredged from specific areas.	N/A	
(g)	What monitoring has taken place and by whom? Please attach report.	Monitoring undertaken by Hydrographic Surveys Ltd	
(f)	Other relevant information	N/A	

B. OTHER WASTES

(a)	Specification of waste	N/A	
(b)	Derived from harbour, estuary, open water or other (please specify)?	N/A	
(c)	Total quantity of wastes dumped (tonnes)	N/A	
(d)	Details of any chemical, toxicity and/or other testing carried out on this waste (reports to be attached as separate documents)	N/A	

(e)	Please provide a map of the area dredged with chemical analysis sampling stations indicated. Please provide details of specific areas and quantities dredged.	N/A	
(f)	What monitoring has taken place and by whom? Please attach report.	N/A	
	Other relevant information	N/A	

C. DUMPING AREA :
Note: EACH INDIVIDUAL DUMPSITE REQUIRES SEPARATE ENTRY

Dumpsite A1

(a)	Location		

Co-ordinates (minimum of 4):
Latitude (decimal degrees)

52.8001°	-6.1287°	
52.8001°	-6.1313°	
52.7994°	-6.1313°	
52.7994°	-6.1287°	

(b)	Quantity of material dumped here	47,712 tonnes in 2014	Using a density of 1,280kg/m3 and the recorded loads by volume ADA Doherty: 33,275m3 Jenny T: 4,000m3
(c)	Origin of material (eg berths, fairway etc)	Harbour, channel and turning basin	
(d)	Depth (metres)	-7.5m--9.5m CD	
(e)	Distance from coast (km)	0.75km	
(f)	Tidal flows		
	Direction (degrees)	26°N, 206°N	
	Maximum speed (m/s knots)	0.5m/s	
(g)	Method of dumping (if more than one vessel involved, give the range of loads and discharge conditions)		
(i)	Vessel(s) load (tonnes)	ADA Doherty: 192-422 tonnes Jenny T:102-166 tonnes	Using a density of 1,280kg/m3 and the recorded loads by volume ADA Doherty: 150 to 330m3 Jenny T: 80-130m3

	(ii)	Manner of discharge from vessel	ADA Doherty-Split hopper barge Jenny T-Bottom dumping barge	
	(iii)	Rate of discharge (te/day)	ADA Doherty: 192-3,949 tonnes Jenny T: 102-166 tonnes	Using a density of 1,280kg/m3 and the recorded loads by volume ADA Doherty: 150 to 3,085m3 Jenny T: 80 to130m3
	(iv)	Speed of vessel while dumping (m/s or knots)	Static	
(h)		Other relevant information (e.g. residual water movements):	N/A	

Dumpsite 2

(a)		Location		
		Co-ordinates (minimum of 4): Latitude (decimal degrees)		
(b)		Quantity of material dumped here		
(c)		Origin of material (eg berths, fairway etc)		
(d)		Depth (metres)		
(e)		Distance from coast (km)		
(f)		Tidal flows		
		Direction (degrees)		
		Maximum speed (m/s knots)		
(g)		Method of dumping (if more than one vessel involved, give the range of loads and discharge conditions)		
	(i)	Vessel(s) load (tonnes)		
	(ii)	Manner of discharge from vessel		

(iii) Rate of discharge (te/day)

(iv) Speed of vessel while dumping (m/s or knots)

(h) Other relevant information
(e.g. residual water movements):

Dumpsite 3

(a) Location

Co-ordinates (minimum of 4):

Latitude (decimal degrees)	Longitude (decimal degrees)

(b) Quantity of material dumped here

(c) Origin of material (eg berths, fairway etc)

(d) Depth (metres)

(e) Distance from coast (km)

(f) Tidal flows
Direction (degrees)

Maximum speed (m/s knots)

(g) Method of dumping (if more than one vessel involved, give the range of loads and discharge conditions)

(i) Vessel(s) load (tonnes)

(ii) Manner of discharge from vessel

(iii) Rate of discharge (te/day)

(iv) Speed of vessel while dumping (m/s or knots)

(h) Other relevant information

(e.g. residual water movements):

Appendix C

Monitoring Operations

C1 Survey Report

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ARKLOW DISPOSAL SITE

ONE MONTH BATHYMETRIC, BOTTOM SAMPLING AND SIDE SCAN SONAR SURVEY

HYDROGRAPHIC SURVEYS LTD.

REPORT PH15021

5th October 2015

Client:	Consulting Engineers:
Wicklow County Council County Buildings Whitegates Wicklow Town	ARUPS Consulting Engineer J P Byrne Consulting Engineers

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REPORT CONTROL SHEET

Client	Wicklow County Council					
Engineer Representative	J P Byrne Consulting Engineers ARUP Consulting Engineers					
Project Name	ARKLOW DISPOSAL SITE – ONE MONTH MONITORING – BATHYMETRIC, SAMPLING AND SIDE SCAN SONAR SURVEY					
Report Name	ARKLOW DISPOSAL SITE – ONE MONTH MONITORING – BATHYMETRIC, SAMPLING AND SIDE SCAN SONAR SURVEY					
Project Number	PH 15021A					
This Report Comprises of	TOC	Text	No. of Volumes	No. of Appendices	Drawings	Electronic data
	1	8	1	1	4	*.pdf, *.dwg

Revision	Status	Author(s)	Approved By	Issue Date
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APPENDICES

APPENDIX 1 Equipment List and Specifications

DRAWINGS

HS 108-1/15	Bathymetry Survey (CD) - *Full Survey Area*	Scale 1:2000
HS 108-2/15	Bathymetry Survey (CD) - *Disposal Pit Only*	Scale 1:500
HS 113/15	Side Scan Sonar Survey	Scale 1:2500
CSK-004	Sample Locations	Scale 1:10000

1. INTRODUCTION

1.1 SITE LOCATION & DESCRIPTION

Hydrographic Surveys Ltd. was instructed by ARUP Consulting Engineers on behalf of Wicklow County Council, to undertake a combined bathymetric survey, bottom sampling and side scan sonar survey at the dredging disposal site off the coast of Arklow, County Wicklow.

The bathymetric survey, bottom sampling and side scan sonar survey were undertaken on the 1st September 2015.

1.2 SURVEY OBJECTIVES AND EXTENTS

The main objectives of the survey were as follows:

- Establish seabed depth and profile in disposal pit and surrounding area
- Aid in the location of possible obstructions on the sea bed.
- Examine and determine sea bed sediments.

Based on the location of the original disposal pit, the bathymetric and side scan survey area extended to limits 500m north and south and 250m east and west of the disposal pit. The sampling survey was located both in the inner harbour and river as well as at the disposal pit.

2. METHODOLOGY

Horizontal control for the survey was provided by a Trimble DGPS receiver with broadcast differential signal corrections from monitoring stations. The navigation system was interfaced to Hypack survey software for logging and for online guidance. The unit has a sub-metre horizontal positioning accuracy. On-line transformation of WGS'84 Latitude and Longitude to Irish National Grid (ING) took place within the survey programme. All plotting of data was to Irish grid.

2.1 BATHYMETRIC SURVEY

2.1.1 Vertical Datum

All levels were reduced to Chart Datum. Tidal variations were recorded at Arklow Harbour using a Valeport 740 model vented tide gauge. Tidal height was recorded every 5 minutes for the duration of the survey. Tidal heights were recorded at an established TBM and levels reduced to the required datum, i.e. Chart Datum.

2.1.2 Depth Sounding

The Navisound 210 dual frequency echo sounder was used to record seabed levels in both digital and analogue format. The echo sounder has a resolution of 0.01m and was calibrated on site by the bar-check method. A bar check was undertaken prior to and on completion of each days survey. The sounder was interfaced with the dGPS via Hypack 2014 survey software thereby providing a digital record with related position fixes.

Line spacing for the bathymetric survey within the channel was at approximately 25m intervals, orientated perpendicular to the coastline, in accordance with the requirements of the survey.

The location and results of the bathymetry survey are shown in APPENDIX A: Drawing No. HS 108-1/15 and HS 108-1/15.

2.2 SIDE SCAN SURVEY SURVEY

The side scan sonar survey was undertaken to identify any potential obstructions located on the seabed surface. Sidescan sonar imaging was obtained using a C-Max digital, dual frequency 100/325kHz, sonar. Data was logged utilizing a Rugged CM2 Sonar transceiver connected to a PC running Maxview acquisition software. The DGPS signal was interfaced in Maxview and Hypack survey software.

Survey lines were undertaken at 50m line intervals, running perpendicular to the coastline, in accordance with the requirements set out in the tender document.

To obtain over 100% data overlap and achieve maximum data resolution, a 50m channel width was utilized. Processing was undertaken utilizing C Max View processing software and mosaics produced in AutoCAD charts using Hypack survey software

2.3 BOTTOM SAMPLING

HSL were instructed to collect 13 samples from the survey area. 6 no. samples were located in the river; 2 no. samples in the dock; and 5 no. were collected from the disposal pit.

The Samples were taken using a stainless steel Van Veen grab sampler. Once recovered the samples were sealed in specialised sample pots and labelled accordingly. The 5 no. samples which required benthic macroinvertebrates analysis were preserved using a mixture of formaldehyde (diluted) and borax (powder form). The 13 samples were sealed and dispatched by courier to the RPS Laboratories for analysis on 1st September 2015.

The benthic macro-invertebrate analysis results were released on the 18th September.

The granulometry and sediment chemistry analysis results were released on October 1st 2015.

3. RESULTS

The survey results and interpretations are discussed below.

3.1 BATHYMETRIC SURVEY RESULTS

The bathymetric survey results with contours were plotted to Chart Datum on drawing HS 108- 1/15. The sea bed indicates gradual gradient sloping seawards, from a high of 3.9m in the north west corner to a depth of 12.2m in the south west corner of the survey area. There is very little difference from the previous 7/10 Day Monitoring survey.

It is clear to see the disposal area in the centre of the survey area; the shallowest area of this pit is in the north east corner, where the seabed rises to 4.6m CD; this is the same as the 7/10 Day Monitoring survey.

In addition to the contractual drawing HS 108-1/15, an extra drawing (HS108-2/15) is included in this report. This displays the disposal pit at a scale of 1:500, and was produced in order to assist in the comparison of previous 1:500 scale drawings of the pit.

3.2 SIDE SCAN SONAR SURVEY RESULTS

The Side Scan Sonar results and mosaic were plotted on drawing HS 113/15. In general, the side scan results were very similar to the 7/10 Day survey. The side scan records were dominated by a light grey colour indicating a sandy smooth sea bed. There were some small dark spots on the records, indicating small rocks/boulders.

The numerous side-scan profile lines were processed and a selected number were used to create the final mosaic. Both the mosaic and identified targets were corrected for layback. Due to warping of the mosaic image when plotting, the identified target positions should be used for locating targets and positions not taken directly from the mosaic image.

3.3 BOTTOM SAMPLING RESULTS

All sample analysis reports have been supplied separate to this report. This includes;

1. The RPS Sample Analysis Report (*RPS Sampling Report 15-46846- Final.xlsx*).
2. The APEM Ltd. Marine Benthic Invertebrate Analysis Report (*Benthic_Macroinv_Report_414118_Arklow Sample_Analysis.pdf*).
3. Sample analysis results were also formatted into the EPA excel spreadsheet *(Arklow EPA DAS material analysis reporting form.xlsx).*

APPENDIX 1

Equipment List and Specifications

Equipment List

Navigation: Trimble AgDGPS 132 Receiver

This navigation unit provides sub-metre differential position accuracy in differential mode.

Bathymetry: Navisound 210 Series

Dual frequency echosounder with an accuracy of 0.10m.

Tidal Height: Valeport Model 740 Tide gauge

Vented gauge. Data logged to 1mm resolution with an accuracy equal to +/- 0.1% of the full scale.

Side Scan Sonar: CMax CM2

The CMax CM2 dual frequency side scan sonar has a full complement of display, recording, editing, marking, annotation, measurement, remote data entry and instant access replay facilities.

It has automatic, microprocessor control of the gain profile. All data is recorded digitally and can be processed through the CM2 Maxview programme and Hypack Hyscan Software. The range setting for this project was set at 50m per channel.

The towfish was deployed starboard side of the boat. Layback was accounted for in the presentation of data.

DRAWINGS

SITE:

Arklow Disposal Site

Sheet Title:

1 Month Monitoring Survey of Arklow Overview Site

CLIENT:

Wicklow Co Co

Consulting Engineers:

J P Byrne Consulting Engineers & ARUP

SURVEYED BY:

HYDROGRAPHIC SURVEYS LTD.
The Cobbles
Crosshaven
Co. Cork
tel: +353 21 4831184

JOB NUMBER:

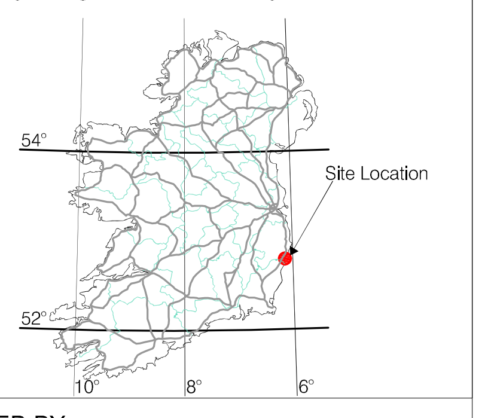
PH 15021

DRAWN BY: James Coade	DRAWING NUMBER: HS 108-1/15
SCALE: 1:2000 ON A1	APPROVED: J.B.J.
REVISION: D01	SURVEY DATE: 01/09/2015

Memoir

1. Constructed on Irish National Grid.
2. Horizontal control by D.G.P.S.
3. Soundings in metres and decimetres reduced to Chart Datum (C.D.)
4. WGS84 Latitude/Longitude lines shown.

Hydrographic Surveys Site



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Hydrographic Surveys Ltd.

SITE:

Arklow Disposal Site

Sheet Title:

1 Month Monitoring Survey of Arklow Disposal Site

CLIENT:

Wicklow Co Co

Consulting Engineers:

J P Byrne Consulting Engineers & ARUP

SURVEYED BY:

HYDROGRAPHIC SURVEYS LTD.
The Cobbles
Crosshaven
Co. Cork
tel: +353 21 4831184

JOB NUMBER:

PH 15021

DRAWN BY:	DRAWING NUMBER:
James Coade	HS 108-2/15
SCALE:	APPROVED:
1:500 ON A1	J.B.J.
REVISION:	SURVEY DATE:
D01	01/09/2015

Memoir

1. Constructed on Irish National Grid.
2. Horizontal control by D.G.P.S.
3. Soundings in metres and decimetres reduced to Chart Datum (C.D.)
4. WGS84 Latitude/Longitude lines shown.

PRODUCED BY:
HYDROGRAPHIC SURVEYS LTD.
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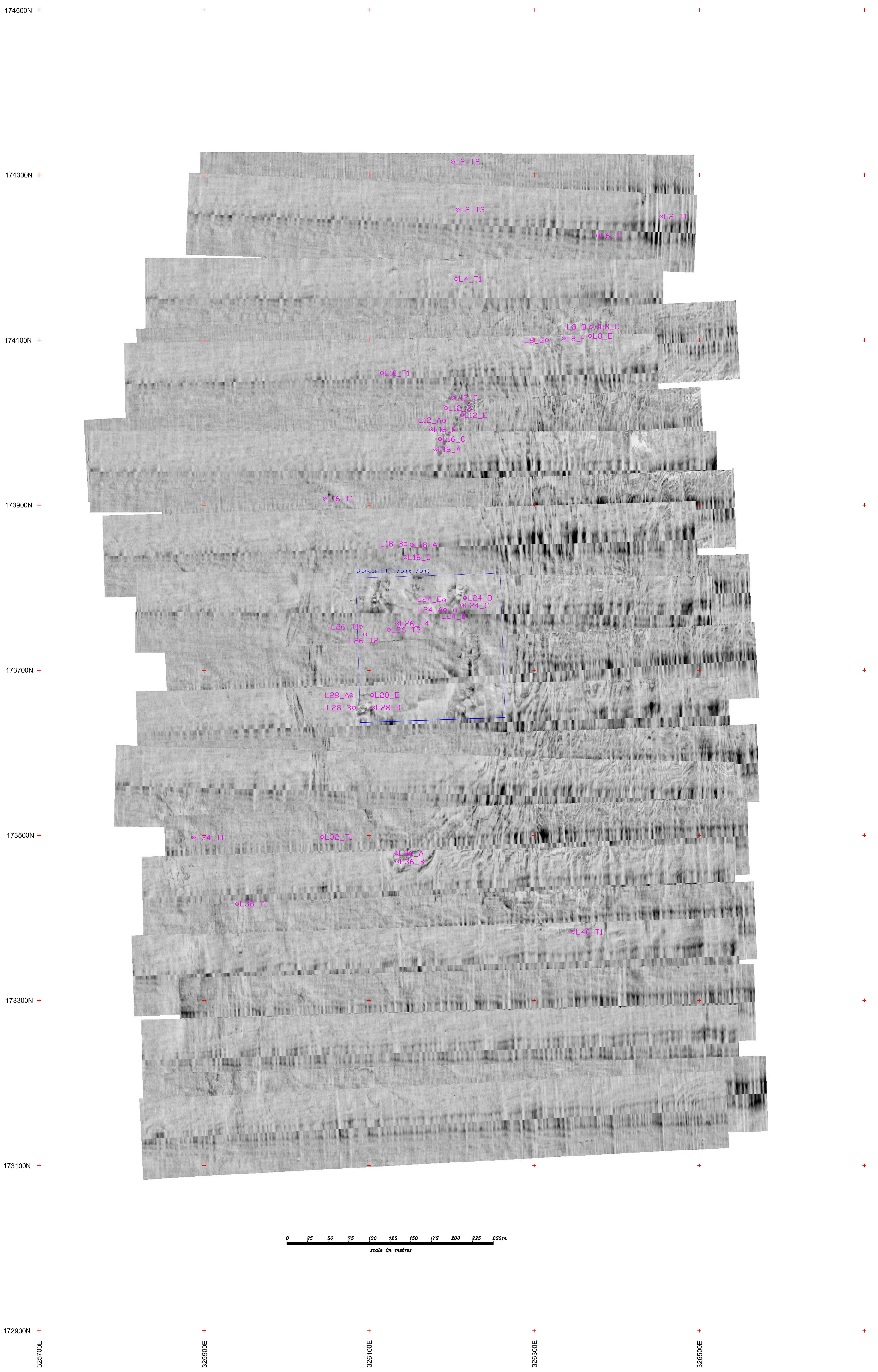


SITE:

Arklow, Co. Wicklow

Sheet Title:

7/10 Day Monitoring
Side Scan Sonar Survey of
Arklow Dump Site

CLIENT:

Wicklow County Council

CONSULTING ENGINEERS:

ARUP Consulting Engineers
J P Byrne Consulting Engineers

SURVEYED BY:

HYDROGRAPHIC SURVEYS LTD
The Cobbles,
Crosshaven,
Co. Cork

tel: +353 21 4831184

JOB NUMBER:

PH15021A

DRAWING NUMBER:

HS 113/15

DRAWN BY:

James Coade

DATE:

01/09/2015

SCALE:	APPROVED:
1:2500 ON A1	J.B.J.

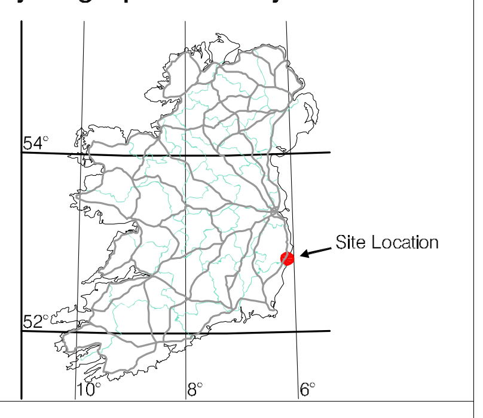
REVISION:

D01

Memoir

1. Constructed on Irish National Grid.
2. Horizontal control by D.G.P.S.
3. Soundings in metres and decimetres reduced to Chart Datum.
4. Interpreted side scan sonar Targets shown thus: ○ L1_T1
5. Side scan mosaic shown for presentation only, image may contain warping/stretching. Targets have been identified from processed unwarped data.
6. Results are based on interpretation of geophysical data and experience of author.

Hydrographic Surveys Site



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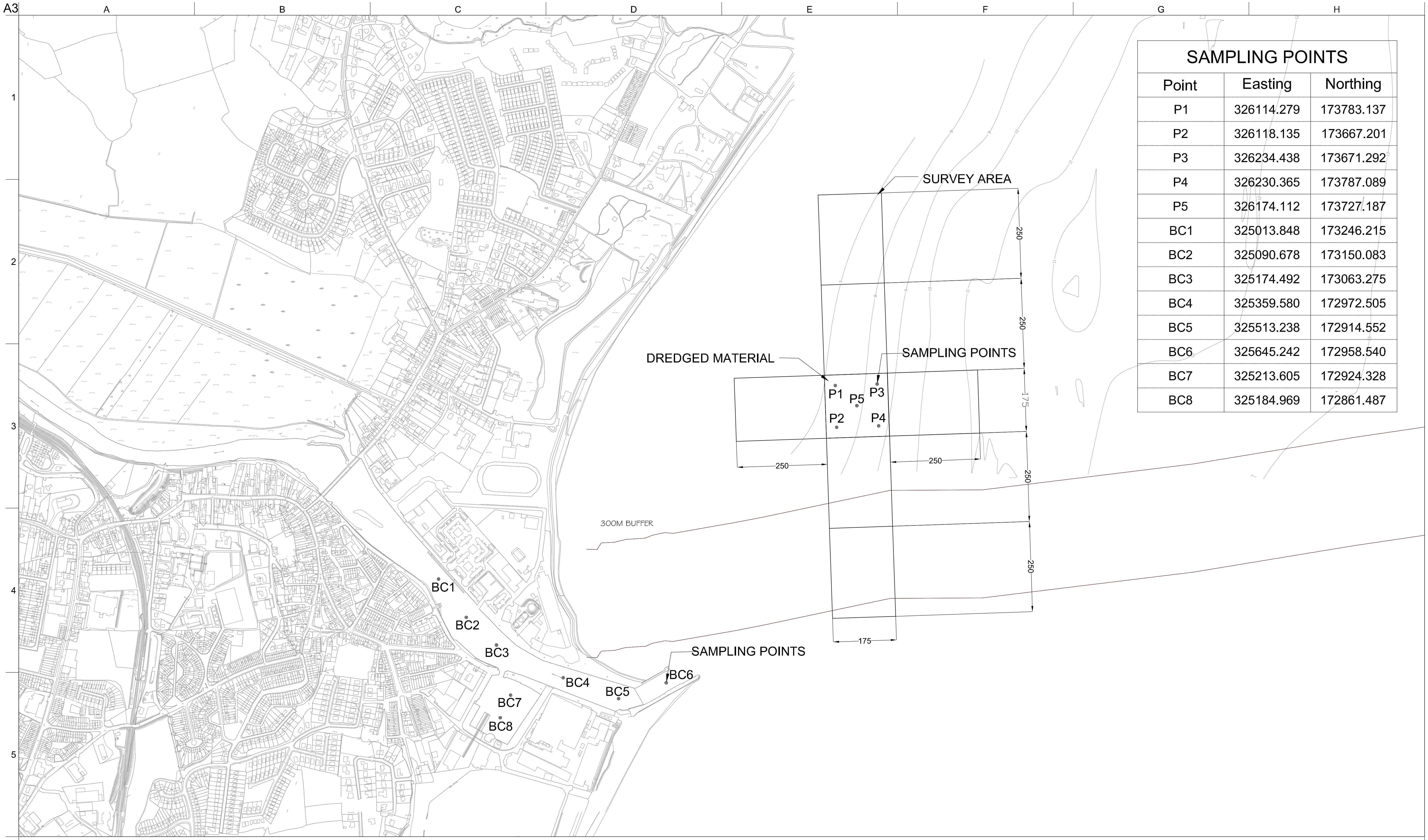

SAMPLING POINTS

Point	Easting	Northing
P1	326114.279	173783.137
P2	326118.135	173667.201
P3	326234.438	173671.292
P4	326230.365	173787.089
P5	326174.112	173727.187
BC1	325013.848	173246.215
BC2	325090.678	173150.083
BC3	325174.492	173063.275
BC4	325359.580	172972.505
BC5	325513.238	172914.552
BC6	325645.242	172958.540
BC7	325213.605	172924.328
BC8	325184.969	172861.487

Notes:
1. The exact location of the pit within the working area will be decided in consultation with the contractor
2. Constructed on Irish National Grid.
3. Soundings in metres and decimetres reduced to Chart Datum.

Issue	Date	By	Chkd	Appd

ARUP

Arup, 50 Ringsend Road
Dublin 4
Tel +353(0)1 233 4455 Fax +353(0)1 668 3169
www.arup.ie

Job Title
Arklow Dredging Project:
Proposed Survey and Sampling

Client
Wicklow County Council

Site layout

Scale at A3 1:10000

Discipline Infrastructure

Job No
224515-01

Drawing Status
For Information

Drawing No
CSK-004

Issue
-

Do not scale

C2 Sampling

2 Shaftesbury Industrial Centre, Icknield Way, Letchworth Garden City, Hertfordshire, SG6 1HE
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Certificate of Analysis

Report No.: 15-46846

Issue No.:	**1**
Date of Issue	**1/10/2015**
Customer Details:	Hugh Power Priority Geotechnical Ltd Unit 12 Owenacurra Business Park Midleton Co Cork
Order No.:	Not given
Customer Reference:	Not given
Quotation Reference:	150522/09
Description:	13 sediment samples
Date Received:	3/9/2015
Test Methods:	Details available on request (refer to SOP code against relevant result/s)
Notes:	None
Approved By:	**Marco Lattughi, Operational Director**

This certificate is issued in accordance with the accreditation requirements of the United Kingdom Accreditation Service.
Observations and interpretations are outside of the scope of UKAS accreditation.
Results reported herein relate only to the items supplied to the laboratory for testing.

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Results Summary - Dry Weights, Moisture, Total Organic Carbon, TPH, Organotins & Density

Report No.: **15-46846**
Customer Reference: Not given
Order No: Not given

				Customer Sample No	**Certified Reference Material**		**BC1**	**BC2**	**BC3**	**BC4**	**BC5**	**BC6**	**BC7**	**BC8**	**PC1**	**PC2**	**PC3**	**PC4**	**PC5**
				Customer Sample ID															
				RPS Sample No			276585	276586	276587	276588	276589	276590	276591	276592	276593	276594	276595	276596	276597
				Sample Type	**SEDIMENT**		**SEDIMENT**	**SEDIMENT**	**SEDIMENT**	**SEDIMENT**	**SEDIMENT**	**SEDIMENT**	**SEDIMENT**	**SEDIMENT**	**SEDIMENT**	**SEDIMENT**	**SEDIMENT**	**SEDIMENT**	**SEDIMENT**
				Sample Location	**CRM-646**														
				Sample Depth (m)															
				Sampling Date			/ /	/ /	/ /	/ /	/ /	/ /	/ /	/ /	/ /	/ /	/ /	/ /	/ /
				Sampling Time															
Determinand	**CAS No**	**Codes**	**SOP**	**Units**	**Result**	**Recovery %**													
dry solids (at 105°C)			In house	%	n/a	n/a	70.3	63.8	18.2	62.5	44.4	51.6	26.7	28.5	77.7	78.2	76.1	68.5	78.6
carbonate % dry matter			In house	%	n/a	n/a	10.3	1.15	19.3	20.8	33.6	26.0	21.4	17.4	4.02	5.36	5.96	11.9	6.38
total organic carbon*		S		%	n/a	n/a	0.48	0.67	8.30	1.53	2.73	4.12	5.28	4.64	0.09	0.09	0.09	0.32	0.07
dibutyltin (DBT)	1002-53-5		In house	mg/kg	71.1	92.3	< 5.00	< 5.00	< 10.96	< 5.00	10.8	< 5.00	29.2	38.6	< 5.00	< 5.00	< 5.00	< 5.00	< 5.00
tributyltin (TBT)	56573-85-4		In house	mg/kg	50.5	105.2	< 2.00	< 2.00	< 10.96	< 2.00	12.2	51.4	176	404	< 2.00	< 2.00	< 2.00	< 2.00	< 2.00
density (on dry solid)			In house	g/cm3	n/a	n/a	1.7	1.7	4.4	1.6	2.0	1.9	2.6	3.2	1.9	1.9	1.8	1.6	1.9

Dibutyltin and tributyltin results have been dry weight corrected

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Results Summary - Metals

Report No.: **15-46846**
Customer Reference: Not given
Order No: Not given

Customer Sample No						**Standard Reference Material**		**BC1**	**BC2**	**BC3**	**BC4**	**BC5**	**BC6**	**BC7**	**BC8**	**PC1**	**PC2**	**PC3**	**PC4**	**PC5**
Customer Sample ID																				
RPS Sample No								276585	276586	276587	276588	276589	276590	276591	276592	276593	276594	276595	276596	276597
Sample Type						**SEDIMENT**		**SEDIMENT**	**SEDIMENT**	**SEDIMENT**	**SEDIMENT**	**SEDIMENT**	**SEDIMENT**	**SEDIMENT**	**SEDIMENT**	**SEDIMENT**	**SEDIMENT**	**SEDIMENT**	**SEDIMENT**	**SEDIMENT**
Sample Location						**SRM-2702**														
Sample Depth (m)																				
Sampling Date								/ /	/ /	/ /	/ /	/ /	/ /	/ /	/ /	/ /	/ /	/ /	/ /	/ /
Sampling Time																				
Determinand	**CAS No**	**Codes**	**SOP**	**Mass**	**Units**	**Result**	**Recovery %**													
aluminium*	7429-90-5	SI	ICP-MS	27	mg/kg	68100	81.1	55400	52800	40200	50900	37700	39500	57300	64400	24000	24200	10700	22000	13200
arsenic*	7440-38-2	SI	ICP-MS	75	mg/kg	43.3	95.6	26.1	22.3	24.5	22.7	18.7	17.3	31.9	31.1	9.52	9.07	7.32	9.44	6.13
cadmium*	7440-43-9	SI	ICP-MS	111	mg/kg	1.02	124.4	0.44	0.81	2.01	0.87	0.47	0.69	2.07	2.15	0.13	0.17	0.15	0.18	0.12
chromium*	7440-47-3	SI	ICP-MS	52	mg/kg	294.6	100.4	55.1	52.1	60.6	59	65.9	44.2	73.9	80.5	21.6	22.8	14.5	31.6	16.4
copper*	7440-50-8	SI	ICP-MS	65	mg/kg	Not certified	n/a	67.7	62.1	110	69	28.4	56.8	199	201	10.2	11.7	10.9	9.15	21.8
lead*	7439-92-1	SI	ICP-MS	208	mg/kg	140.3	97.4	101	113	230	112	66.9	76.1	231	209	21.9	24	11.5	16	12
lithium*	7439-93-2	SI	ICP-MS	7	mg/kg	Not certified	n/a	76.9	72.2	52	78.1	51.1	43.2	59.6	68	21	22.8	13.9	19.4	12
mercury*	7439-97-6	SI	AFS	202	mg/kg	0.46	102.2	0.03	0.01	0.08	0.03	0.04	0.03	0.12	0.1	< 0.01	< 0.01	< 0.01	0.01	< 0.01
nickel*	7440-02-0	SI	ICP-MS	60	mg/kg	66.0	87.5	22.8	20.9	26.9	24.8	29.1	18.3	30.2	33.4	9.66	9.6	5.21	9.97	4.38
zinc*	7440-66-6	SI	ICP-MS	65	mg/kg	437.8	90.2	216	251	439	272	148	188	597	588	54.7	47.5	33.9	45.6	25.1

Metals results have been dry weight corrected

RPS

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Results Summary - Polycyclic Aromatic Hydrocarbons (EPA 16 PAHs)

Report No.: **15-46846**
Customer Reference: Not given
Order No: Not given

					Customer Sample No	**Certified Reference Material**		**AQC spike**	**BC7**	**BC8**
					Customer Sample ID					
					RPS Sample No				276591	276592
					Sample Type	**SEDIMENT**		**SEDIMENT**	**SEDIMENT**	**SEDIMENT**
					Sample Location	**NIST-1944**		**Spike on clean sediment**		
					Sample Depth (m)					
					Sampling Date				/ /	/ /
					Sampling Time					
Determinand	**CAS No**	**Codes**	**SOP**	**Mass**	**Units**	**Result**	**Recovery %**	**Recovery %**		
naphthalene	91-20-3		304	128	ug/kg	Not Certified	N/A	91.14	40.4	44.9
acenaphthylene	208-96-8		304	152	ug/kg	Not Certified	N/A	85.00	31.1	27.4
acenaphthene	83-32-9		304	154	ug/kg	Not Certified	N/A	97.45	172	28.1
fluorene	86-73-7		304	166	ug/kg	Not Certified	88.09	95.90	102	60.4
phenanthrene	85-01-8		304	178	ug/kg	4399.4	83.48	100.50	265	80.7
anthracene	120-12-7		304	178	ug/kg	Not Certified	103.34	98.93	106	49.1
fluoranthene	206-44-0		304	202	ug/kg	6978.05	78.23	104.00	738	316
pyrene	129-00-0		304	202	ug/kg	7284.75	75.10	101.44	584	274
benzo(a)anthracene	56-55-3		304	228	ug/kg	3351.85	71.01	91.46	321	149
chrysene	218-01-9		304	228	ug/kg	4361.95	89.75	102.11	280	127
benzo(b)fluoranthene	205-99-2		304	252	ug/kg	5792.6	96.87	93.51	509	289
benzo(k)fluoranthene	207-08-9		304	252	ug/kg	2048	89.04	96.01	163	106
benzo(a)pyrene	50-32-8		304	252	ug/kg	3182.7	74.02	93.94	335	180
indeno(1,2,3-c,d)pyrene	193-39-5		304	276	ug/kg	922.7	84.76	93.67	220	131
dibenzo(a,h)anthracene	53-70-3		304	278	ug/kg	2356.4	121.57	91.57	87.3	57.6
benzo(g,h,i)perylene	191-24-2		304	276	ug/kg	2870.8	101.08	97.72	239	157

PAH results have been dry weight corrected

RPS

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Results Summary - PSA Results

Report No.: **15-46846**
Customer Reference: Not given
Order No: Not given

Customer Sample No					**BC1**	**BC2**	**BC3**	**BC4**	**BC5**	**BC6**	**BC7**	**BC8**	**PC1**	**PC2**	**PC3**	**PC4**	**PC5**
Customer Sample ID																	
RPS Sample No					276585	276586	276587	276588	276589	276590	276591	276592	276593	276594	276595	276596	276597
Sample Type					**SEDIMENT**	**SEDIMENT**	**SEDIMENT**	**SEDIMENT**	**SEDIMENT**	**SEDIMENT**	**SEDIMENT**	**SEDIMENT**	**SEDIMENT**	**SEDIMENT**	**SEDIMENT**	**SEDIMENT**	**SEDIMENT**
Sample Location																	
Sample Depth (m)																	
Sampling Date					/ /	/ /	/ /	/ /	/ /	/ /	/ /	/ /	/ /	/ /	/ /	/ /	/ /
Sampling Time																	
Determinand	**CAS No**	**Codes**	**SOP**	**Units**													
sample type*		S			Trimodal, Very Poorly Sorted	Polymodal, Very Poorly Sorted	Bimodal, Very Poorly Sorted	Bimodal, Very Poorly Sorted	Bimodal, Very Poorly Sorted	Unimodal, Poorly Sorted	Trimodal, Very Poorly Sorted	Bimodal, Very Poorly Sorted	Unimodal, Poorly Sorted	Unimodal, Moderately Sorted	Unimodal, Moderately Well Sorted	Polymodal, Very Poorly Sorted	Unimodal, Moderately Well Sorted
textural group (GRADISTAT)*		S			Sandy Gravel	Muddy Sandy Gravel	Muddy Sand	Muddy Sand	Sandy Mud	Muddy Sand	Sandy Mud	Sandy Mud	Gravelly Sand	Gravelly Sand	Slightly Gravelly Sand	Muddy Sandy Gravel	Sand
sediment name*		S			Sandy Fine Gravel	Very Coarse Silty Sandy Coarse Gravel	Very Coarse Silty Fine Sand	Coarse Silty Coarse Sand	Very Fine Sandy Coarse Silt	Very Coarse Silty Medium Sand	Very Fine Sandy Coarse Silt	Very Fine Sandy Coarse Silt	Very Fine Gravelly Medium Sand	Very Fine Gravelly Medium Sand	Slightly Very Fine Gravelly Medium Sand	Medium Silty Sandy Coarse Gravel	Moderately Well Sorted Medium Sand
arithmetic mean (method of moments)*		S		um	4090	10100	193	419	114	342	147	102	1800	810	530	7060	451
arithmetic sorting (method of moments)*		S		um	5760	12700	217	309	181	265	211	156	4820	1260	314	11600	162
arithmetic skewness (method of moments)*		S		um	1.72	1.14	1.61	0.038	2.49	0.535	1.89	2.54	4.49	6.11	4.16	1.67	0.795
arithmetic kurtosis (method of moments)*		S		um	4.63	3.08	4.91	1.61	8.88	2.21	5.79	9.79	22.5	46.4	36.4	4.61	3.24
geometic mean (method of moments)*		S		um	1540	1790	91.1	209	39.6	190	48.9	38.8	690	565	466	925	418
geometic sorting (method of moments)*		S		um	4.45	11.9	3.95	4.83	4.51	4.01	4.88	4.21	2.68	1.96	1.57	10.8	1.42
geometic skewness (method of moments)*		S		um	-0.305	-0.476	-0.472	-1.16	0.050	-1.29	0.021	0.074	2.15	1.55	0.588	-0.153	0.010
geometic kurtosis (method of moments)*		S		um	3.77	2.10	2.69	3.20	2.47	4.10	2.19	2.38	7.81	6.89	4.03	2.58	2.50
logarithmic mean (method of moments)*		S		phi	-0.625	-0.838	3.46	2.26	4.66	2.40	4.35	4.69	0.535	0.824	1.10	0.113	1.26
logarithmic sorting (method of moments)*		S		phi	2.15	3.57	1.98	2.27	2.17	2.00	2.29	2.07	1.42	0.973	0.652	3.44	0.510
logarithmic skewness (method of moments)*		S		phi	0.305	0.476	0.472	1.16	-0.050	1.29	-0.021	-0.074	-2.15	-1.55	-0.588	0.153	-0.010
logarithmic kurtosis (method of moments)*		S		phi	3.77	2.10	2.69	3.20	2.47	4.10	2.19	2.38	7.81	6.89	4.03	2.58	2.50
mean (Folk and Ward method - um)*		S		um	1750	1750	94.8	213	39.9	208	51.5	39.2	607	526	454	1260	418
sorting (Folk and Ward method - um)*		S		um	4.31	11.7	4.09	4.64	4.68	3.79	5.18	4.38	2.28	1.82	1.56	10.7	1.46
skewness (Folk and Ward method - um)*		S		um	0.314	-0.226	-0.142	-0.659	0.075	-0.458	0.125	0.097	0.447	0.180	0.032	0.305	0.002
kurtosis (Folk and Ward method - um)*		S		um	0.998	0.703	0.927	0.903	0.955	1.16	0.817	0.898	1.84	1.23	0.966	0.921	0.940
mean (Folk and Ward method - phi)*		S		phi	-0.806	-0.806	3.40	2.23	4.65	2.27	4.28	4.67	0.720	0.926	1.14	-0.331	1.26
sorting (Folk and Ward method - phi)*		S		phi	2.11	3.54	2.03	2.21	2.23	1.92	2.37	2.13	1.19	0.867	0.637	3.41	0.548
skewness (Folk and Ward method - phi)*		S		phi	-0.314	0.226	0.142	0.659	-0.075	0.458	-0.125	-0.097	-0.447	-0.180	-0.032	-0.305	-0.002
kurtosis (Folk and Ward method - phi)*		S		phi	0.998	0.703	0.927	0.903	0.955	1.16	0.817	0.898	1.84	1.23	0.966	0.921	0.940
mean description (Folk and Ward method)*		S			Very Coarse Sand	Very Coarse Sand	Very Fine Sand	Fine Sand	Very Coarse Silt	Fine Sand	Very Coarse Silt	Very Coarse Silt	Coarse Sand	Coarse Sand	Medium Sand	Very Coarse Sand	Medium Sand
sorting description (Folk and Ward method)*		S			Very Poorly Sorted	Very Poorly Sorted	Very Poorly Sorted	Very Poorly Sorted	Very Poorly Sorted	Poorly Sorted	Very Poorly Sorted	Very Poorly Sorted	Poorly Sorted	Moderately Sorted	Moderately Well Sorted	Very Poorly Sorted	Moderately Well Sorted
skewness description (Folk and Ward method)*		S			Very Coarse Skewed	Fine Skewed	Fine Skewed	Very Fine Skewed	Symmetrical	Very Fine Skewed	Coarse Skewed	Symmetrical	Very Coarse Skewed	Coarse Skewed	Symmetrical	Very Coarse Skewed	Symmetrical
kurtosis description (Folk and Ward method)*		S			Mesokurtic	Platykurtic	Mesokurtic	Mesokurtic	Mesokurtic	Leptokurtic	Platykurtic	Platykurtic	Very Leptokurtic	Leptokurtic	Mesokurtic	Mesokurtic	Mesokurtic

MODE 1 - um*		S		um	855	19200	108	605	18.9	428	18.9	18.9	428	428	428	19200	428
MODE 2 - um*		S		um	19200	9600	215	108	605		428	108				428	
MODE 3 - um*		S		um	6800	855					215					38300	
MODE 1 - phi*		S		phi	0.247	-4.24	3.24	0.747	5.75	1.25	5.75	5.75	1.25	1.25	1.25	-4.24	1.25
MODE 2 - phi*		S		phi	-4.24	-3.24	2.24	3.24	0.747		1.25	3.24				1.25	
MODE 3 - phi*		S		phi	-2.74	0.247					2.24					-5.24	
D10 - um*		S		um	411	51.5	13.1	15.3	6.0	20.2	6.8	6.4	297	273	264	38.9	261
D50 - um*		S		um	1100	2460	106	429	37.2	292	42.3	35.4	531	514	454	533	417
D90 - um*		S		um	14600	33300	520	850	352	737	476	296	2150	1200	843	21600	675
(D90/D10) - um*		S		um	35.4	646	39.6	55.6	58.6	36.4	70.2	46.3	7.23	4.41	3.19	555	2.59
(D90 - D10) - um*		S		um	14200	33300	507	835	346	716	469	290	1850	930	579	21600	414
(D75/D25) - um*		S		um	7.58	76.5	7.22	8.60	8.65	5.00	12.4	8.55	2.10	2.06	1.85	36.3	1.70
(D75 - D25) - um*		S		um	4300	18100	222	590	101	417	167	99.7	428	387	287	10100	223
D10 - phi*		S		phi	-3.87	-5.06	0.942	0.234	1.51	0.441	1.07	1.76	-1.1	-0.266	0.246	-4.43	0.568
D50 - phi*		S		phi	-0.141	-1.3	3.23	1.22	4.75	1.78	4.56	4.82	0.912	0.961	1.14	0.909	1.26
D90 - phi*		S		phi	1.28	4.28	6.25	6.03	7.38	5.63	7.21	7.29	1.75	1.87	1.92	4.68	1.94
(D90/D10) - phi*		S		phi	-0.332	-0.846	6.63	25.7	4.9	12.8	6.73	4.15	-1.59	-7.04	7.8	-1.06	3.42
(D90 - D10) - phi*		S		phi	5.15	9.34	5.31	5.8	5.87	5.19	6.13	5.53	2.86	2.14	1.68	9.12	1.37
(D75/D25) - phi*		S		phi	-0.266	-0.49	2.46	6.32	1.99	3.47	2.48	1.98	4.65	3.54	2.31	-0.538	1.87
(D75 - D25) - phi*		S		phi	2.92	6.26	2.85	3.1	3.11	2.32	3.64	3.1	1.07	1.04	0.887	5.18	0.765
% gravel*		S		%	36.5	51.8	0	0	0	0	0	0	10.5	5.23	0.56	33.2	0
% sand*		S		%	60.5	36.8	63.8	77.1	38.1	81.8	42.4	37.2	89.5	94.8	99.4	55.1	100
% mud*		S		%	2.96	11.4	36.2	22.9	61.9	18.2	57.6	62.8	0	0	0	11.7	0
% very coarse gravel (>32<64mm or <-5>-6phi)*		S		%	0	11.3	0	0	0	0	0	0	0	0	0	7.97	0
% coarse gravel (>16<32mm or <-4>-5phi)*		S		%	8.79	22.7	0	0	0	0	0	0	3.58	0	0	16	0
% medium gravel (>8<16mm or <-3>-4phi)*		S		%	9.03	6.24	0	0	0	0	0	0	1.22	1.24	0	4.39	0
% fine gravel (>4<8mm or <-2>-3phi)*		S		%	9.56	5.26	0	0	0	0	0	0	1.71	0.87	0.05	2.61	0
% very fine gravel (>2<4mm or <-1>-2phi)*		S		%	9.15	6.25	0	0	0	0	0	0	4.02	3.11	0.51	2.26	0
% very coarse sand (>1<2mm or <0>-1phi)*		S		%	16	6.12	0	0	0	0	0	0	7.95	6.94	3.64	3.22	0
% coarse sand (>0.5<1mm or <1>0phi)*		S		%	33.7	10.9	10.7	43.4	6.33	26.9	9.15	4.1	35.8	39.7	37	15.6	30.7
% medium sand (>0.25<0.5mm or <2>1phi)*		S		%	7.98	5.72	15	21.1	6.79	28.7	11.1	7.86	43.1	42.4	52.1	27.6	62.3
% fine sand (>0.125<0.25mm or <3>2phi)*		S		%	0.95	7.32	19.5	5.57	9.81	16.3	10.1	10.9	2.6	5.79	6.71	7.26	6.99
% very fine sand (>0.0625<0.125mm or <4>3phi)*		S		%	1.91	6.71	18.6	7.08	15.2	9.91	12	14.3	0	0	0	1.36	0
% very coarse silt (>0.03125<0.0625mm or <5>4phi*		S		%	1.16	4.71	13.7	6.3	15.9	5.34	14.3	15.8	0	0	0	2.53	0
% coarse silt (>0.015625<0.03125mm or <6>5phi)*		S		%	0.94	3.34	10.5	6.38	16.9	4.55	16.9	17.9	0	0	0	2.72	0
% medium silt (>0.007813<0.015625mm or <7>6phi)*		S		%	0.54	1.99	7.06	5.43	15.2	4.2	14.6	16.1	0	0	0	3.12	0
% fine silt (>0.003906<0.007813mm or <8>7phi)*		S		%	0.27	0.9	3.27	3.05	8.55	2.47	7.63	8.82	0	0	0	2.01	0
% very fine silt (>0.001953<0.003906mm or <9>8phi*		S		%	0.05	0.34	1.17	1.21	3.46	1.08	2.8	3.07	0	0	0	0.88	0
% clay (<0.001953mm or >9phi)*		S		%	0	0.11	0.52	0.5	1.9	0.54	1.5	1.26	0	0	0	0.48	0

RPS

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Results Summary - PSA Size Class & Statistics

Report No.: **15-46846**
Customer Reference: Not given
Order No: Not given

Customer Sample No				**BC1**	**BC2**	**BC3**	**BC4**	**BC5**	**BC6**	**BC7**	**BC8**	**PC1**	**PC2**	**PC3**	**PC4**	**PC5**
Customer Sample ID																
RPS Sample No				276585	276586	276587	276588	276589	276590	276591	276592	276593	276594	276595	276596	276597
Sample Type				**SEDIMENT**	**SEDIMENT**	**SEDIMENT**	**SEDIMENT**	**SEDIMENT**	**SEDIMENT**	**SEDIMENT**	**SEDIMENT**	**SEDIMENT**	**SEDIMENT**	**SEDIMENT**	**SEDIMENT**	**SEDIMENT**
Sample Location																
Sample Depth (m)																
Sampling Date				/ /	/ /	/ /	/ /	/ /	/ /	/ /	/ /	/ /	/ /	/ /	/ /	/ /
Sampling Time																
Sediment	**mm**	**phi ϕ**	**Units**													
Very coarse gravel	>32<64	<-5>-6	%	0.00	11.30	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	7.97	0.00
Coarse gravel	>16<32	<-4>-5	%	8.79	22.70	0.00	0.00	0.00	0.00	0.00	0.00	3.58	0.00	0.00	16.00	0.00
Medium gravel	>8<16	<-3>-4	%	9.03	6.24	0.00	0.00	0.00	0.00	0.00	0.00	1.22	1.24	0.00	4.39	0.00
Fine gravel	>4<8	<-2>-3	%	9.56	5.26	0.00	0.00	0.00	0.00	0.00	0.00	1.71	0.87	0.05	2.61	0.00
Very fine gravel	>2<4	<-1>-2	%	9.15	6.25	0.00	0.00	0.00	0.00	0.00	0.00	4.02	3.11	0.51	2.26	0.00
Very coarse sand	>1<2	<0>-1	%	16.00	6.12	0.00	0.00	0.00	0.00	0.00	0.00	7.95	6.94	3.64	3.22	0.00
Coarse sand	>0.5<1	<1>0	%	33.70	10.90	10.70	43.40	6.33	26.90	9.15	4.10	35.80	39.70	37.00	15.60	30.70
Medium sand	>0.25<0.5	<2>1	%	7.98	5.72	15.00	21.10	6.79	28.70	11.10	7.86	43.10	42.40	52.10	27.60	62.30
Fine sand	>0.125<0.25	<3>2	%	0.95	7.32	19.50	5.57	9.81	16.30	10.10	10.90	2.60	5.79	6.71	7.26	6.99
Very fine sand	>0.0625<0.125	<4>3	%	1.91	6.71	18.60	7.08	15.20	9.91	12.00	14.30	0.00	0.00	0.00	1.36	0.00
Very coarse silt	>0.03125<0.0625	<5>4	%	1.16	4.71	13.70	6.30	15.90	5.34	14.30	15.80	0.00	0.00	0.00	2.53	0.00
Coarse silt	>0.015625<0.03125	<6>5	%	0.94	3.34	10.50	6.38	16.90	4.55	16.90	17.90	0.00	0.00	0.00	2.72	0.00
Medium silt	>0.007813<0.015625	<7>6	%	0.54	1.99	7.06	5.43	15.20	4.20	14.60	16.10	0.00	0.00	0.00	3.12	0.00
Fine silt	>0.003906<0.007813	<8>7	%	0.27	0.90	3.27	3.05	8.55	2.47	7.63	8.82	0.00	0.00	0.00	2.01	0.00
Very fine silt	>0.001953<0.003906	<9>8	%	0.05	0.34	1.17	1.21	3.46	1.08	2.80	3.07	0.00	0.00	0.00	0.88	0.00
Clay	<0.001953	>9	%	0.00	0.11	0.52	0.50	1.90	0.54	1.50	1.26	0.00	0.00	0.00	0.48	0.00
Statistics*	Mean (phi)			-0.806	-0.806	3.4	2.23	4.65	2.27	4.28	4.67	0.72	0.926	1.14	-0.331	1.26
	Sorting			2.11	3.54	2.03	2.21	2.23	1.92	2.37	2.13	1.19	0.867	0.637	3.41	0.548
	Skewness			-0.314	0.226	0.142	0.659	-0.075	0.458	-0.125	-0.097	-0.447	-0.18	-0.032	-0.305	-0.002
	Kurtosis			0.998	0.703	0.927	0.903	0.955	1.16	0.817	0.898	1.84	1.23	0.966	0.921	0.94
	% Silt/Clay		%	2.96	11.39	36.22	22.87	61.91	18.18	57.73	62.95	0.00	0.00	0.00	11.74	0.00
	Textural Group**			Sandy Gravel	Muddy Sandy Gravel	Muddy Sand	Muddy Sand	Sandy Mud	Muddy Sand	Sandy Mud	Sandy Mud	Gravelly Sand	Gravelly Sand	htly Gravelly S	ddy Sandy Gra	Sand

* Folk & Ward

** GRADISTAT classification system (Blott, S. J. & Pye, K., 2001)

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Results Summary - PSA Wentworth Scale

Report No.: **15-46846**
Customer Reference: Not given
Order No: Not given

Customer Sample No		**BC1**	**BC2**	**BC3**	**BC4**	**BC5**	**BC6**	**BC7**	**BC8**	**PC1**	**PC2**	**PC3**	**PC4**	**PC5**
Customer Sample ID														
RPS Sample No		276585	276586	276587	276588	276589	276590	276591	276592	276593	276594	276595	276596	276597
Sample Type		**SEDIMENT**	**SEDIMENT**	**SEDIMENT**	**SEDIMENT**	**SEDIMENT**	**SEDIMENT**	**SEDIMENT**	**SEDIMENT**	**SEDIMENT**	**SEDIMENT**	**SEDIMENT**	**SEDIMENT**	**SEDIMENT**
Sample Location														
Sample Depth (m)														
Sampling Date		/ /	/ /	/ /	/ /	/ /	/ /	/ /	/ /	/ /	/ /	/ /	/ /	/ /
Sampling Time														
Parameter	**Units**													
Pebble	%	27.38	45.50	0.00	0.00	0.00	0.00	0.00	0.00	6.51	2.11	0.05	30.97	0.00
Granule	%	9.15	6.25	0.00	0.00	0.00	0.00	0.00	0.00	4.02	3.11	0.51	2.26	0.00
Very coarse sand	%	16.00	6.12	0.00	0.00	0.00	0.00	0.00	0.00	7.95	6.94	3.64	3.22	0.00
Coarse sand	%	33.70	10.90	10.70	43.40	6.33	26.90	9.15	4.10	35.80	39.70	37.00	15.60	30.70
Medium sand	%	7.98	5.72	15.00	21.10	6.79	28.70	11.10	7.86	43.10	42.40	52.10	27.60	62.30
Fine sand	%	0.95	7.32	19.50	5.57	9.81	16.30	10.10	10.90	2.60	5.79	6.71	7.26	6.99
Very fine sand	%	1.91	6.71	18.60	7.08	15.20	9.91	12.00	14.30	0.00	0.00	0.00	1.36	0.00
Silt Clay	%	2.96	11.39	36.22	22.87	61.91	18.18	57.73	62.95	0.00	0.00	0.00	11.74	0.00
Total	%	100.0	99.9	100.0	100.0	100.0	100.0	100.1	100.1	100.0	100.1	100.0	100.0	100.0

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Report No.:	**15-46846**
Customer Reference:	Not given
Order No:	Not given

Comments

Job	Description	Job Comments
15-46846	13 sediment samples in s	n/a

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Report Information

Results for soil/sediment samples are expressed on a dry weight basis unless otherwise stated.

Key to Report Codes

U	UKAS accredited test
S	Subcontracted to approved laboratory
SIU	Subcontracted to internal RPS Group Laboratory UKAS accredited test
I/S	Insufficient Sample
U/S	Unsuitable sample
S	Subcontracted to approved laboratory
SU	Subcontracted to approved laboratory UKAS Accredited for the test
SM	Subcontracted to approved laboratory MCERTS/UKAS Accredited for the test
I/S	Insufficient Sample
Waters	1 month from the issue date of this report
Other Liquids	2 months from the issue date of this report
Solids (including sediments)	2 months from the issue date of this report

*Sample retention may be subject to agreement with the customer for particular projects

Analytical Methods

PAH's and PCB's	GCMS analysis following extraction of the wet sediment with hexane:acetone by ultrasonic and equlibrium extraction. Extract cleaned-up with alumina and activated copper.
Total Hydrocarbons	GCFID analysis following extraction of the wet sediment with dichloromethane:methanol by ultrasonic extraction and subsequent partitioning with water. Extract cleaned-up with silica and activated copper.
Metals	ICP-MS analysis following microwave assisted digestion in hydrofluoric acid of the dried (<30°C) and ground sediment.
TOC	Combustion and infrared analysis following carbonate removal with hydrochloric acid.
Carbonate Content	Gravimetric analysis of a dri portion of the sediment following carbonate removal with hydrochloric acid.
PSA	Wet and dry sieving followed by laser diffraction analysis.
Density	Determination of density from the dry sediment by gravimetric analysis of a known volume of sediment.
Moisture content and dry solids at 105°C	A portion of the wet sediment is dried at 105°C to constant weight.
TBT and DBT	GCMS analysis following the extraction of the wet sediment and subsequent derivatisation.
Please note:	**All testing carried out using the <2mm fraction**

Laboratories

RPS Letchworth	UKAS Test House 1663
RPS Manchester	UKAS Test House 0605
ESG Scientifics (TOC)	UKAS Test House 0001
Thompson PSA only	

Profiency Testing (PT)

RPS Letchworth and Manchester Laboratories participate in the QUASIMEME Profiency Testing Scheme

EPA Dumping at Sea Permit Application - Material Analysis Reporting Form (Version 1.0)
Sheet 2. Project Info

1. General Information	Applicant (company name)	ARUP Consulting Engineers
	Location (port/harbour)	Arklow
	Dredge Quantity	-
	Permit Application Reg. No. (to be assigned by EPA)	S0002-01
2. Survey Information	Survey Company	Hydrographic Surveys Ltd.
	Sampling Date	01-Sep-15
	Analysing Laboratory	RPS Laboratories, Hertfordshire
	Sub Contract Lab	RPS Laboratories, Hertfordshire
	Analysis Date	01-Oct-15
3. Methods Information	Fraction analysed	All testing carried out using the <2mm fraction
	Water content of sample (reported as %)	A portion of the wet sediment is dried at 105°C to constant weight
	Are results reported as wet weight or dry weight?	Dry
	Granulometry method	Wet and dry sieving followed by laser diffraction analysis.
	TEH method	GCFID analysis following extraction of the wet sediment with dichloromethane:methanol by ultrasonic extraction and subsequent partitioning with water. Extract cleaned-up with silica and activated copper
	Organic carbon (OC) method	Combustion and infrared analysis following carbonate removal with hydrochloric acid
	Metals (incl. mercury & arsenic) extraction type	ICP-MS analysis following microwave assisted digestion in hydrofluoric acid of the dried (<30°C) and ground sediment.
	Methods of detection (metals, incl. mercury & arsenic)	ICP-MS analysis following microwave assisted digestion in hydrofluoric acid of the dried (<30°C) and ground sediment.
	Organics extraction types	Combustion and infrared analysis following carbonate removal with hydrochloric acid
	Methods of detection (TBT & DBT)	GCMS analysis following the extraction of the wet sediment and subsequent derivatisation
	Method of detection (PAH's and PCB's)	GCMS analysis following extraction of the wet sediment with hexaneacetone by ultrasonic and equlibrium extraction. Extract cleaned-up with alumina and activated copper.

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Marine Benthic Invertebrate Analysis Report

The analysis on adjacent tab(s) of this workbook has been carried out by APEM Ltd under method MINV-01.

Client Name:	RPS Mountainheath	
Address:	Unit 2 Shaftsbury Industrial Centre Icknield Way Letchworth Graden City Hertfordshire SG6 1HE	
Phone Number:	01462 480400	
P.O. Number:	S36214	
Prepared by:	Nicola Pennisi	Letchworth Laboratory Manager
Approved and issued by:	Chris Ashelby	Marine Technical Specialist
Issue Date:	18/09/2015	

If you have any comments or complaints regarding this or any other piece of work conducted by APEM Ltd, please contact D. Slattery (Quality Manager) d.slattery@apemltd.co.uk

APEM Report No. 414118-01

APEM Report No. 414118-01

Sample Number	Sample Date	Sample Method	Watercourse	Site Description	Analysis Type	Analysis Date	Analyst	QC Date	APEM location	Notes
55646	01/09/2015	0.045m2 Van Veen Grab 1mm	Irish Sea	PC_1	1.0mm mesh	10/09/2015	NP	11/09/15	Letchworth Lab	
55647	01/09/2015	0.045m2 Van Veen Grab 1mm	Irish Sea	PC_2	1.0mm mesh	10/09/2015	NP	11/09/15	Letchworth Lab	
55648	01/09/2015	0.045m2 Van Veen Grab 1mm	Irish Sea	PC_3	1.0mm mesh	10/09/2015	NP	11/09/15	Letchworth Lab	
55649	01/09/2015	0.045m2 Van Veen Grab 1mm	Irish Sea	PC_4	1.0mm mesh	10/09/2015	NP	11/09/15	Letchworth Lab	
55650	01/09/2015	0.045m2 Van Veen Grab 1mm	Irish Sea	PC_5	1.0mm mesh	10/09/2015	NP	11/09/15	Letchworth Lab	

APEM Report No. 414118-01

	Sample Number		55646	55647	55648	55649	55650
	Sample Date		01/09/2015	01/09/2015	01/09/2015	01/09/2015	01/09/2015
	Sample Method		0.045m2 Van Veen Grab 1mm	0.045m2 Van Veen Grab 1mm	0.045m2 Van Veen Grab 1mm	0.045m2 Van Veen Grab 1mm	0.045m2 Van Veen Grab 1mm
	Watercourse		Irish Sea	Irish Sea	Irish Sea	Irish Sea	Irish Sea
	Site Description		PC_1	PC_2	PC_3	PC_4	PC_5
	Analysis Type		1.0mm mesh	1.0mm mesh	1.0mm mesh	1.0mm mesh	1.0mm mesh
	Analysis Date		10/09/2015	10/09/2015	10/09/2015	10/09/2015	10/09/2015
	Analyst		NP	NP	NP	NP	NP
Code	Taxa ID	Qualifiers	55646	55647	55648	55649	55650
G0001	NEMERTEA		1		1		
P0109	Sthenelais limicola				1		
P0118	Eteone longa	aggregate		1		1	
P0141	Phyllodoce groenlandica				Frag.	1	
P0146	Phyllodoce rosea					3	
P0164	Eumida bahusiensis				1	1	
P0256	Glycera alba			1	1	Frag.	
P0494	Nephtys	juvenile	2	1		1	
P0498	Nephtys cirrosa			1			
P0502	Nephtys kersivalensis					1	
P0574	Lumbrineris aniara/cingulata			1			
P0672	Scoloplos armiger		1		1	2	
P0794	Spiophanes bombyx		4	20	10	9	1
P0834	Chaetozone christiei		1				
P0923	Notomastus		1	6	2	13	1
P1107	Lagis koreni					8	
R0077	Balanus crenatus			1		4	
S0213	Stenothoe marina						1
S0438	Ampelisca spinipes				Frag.		
S0452	Bathyporeia elegans			1			
S1445	Paguridae	juvenile	1				
W1336	Onchidoris proxima	?	1			1	
W1569	Nucula nitidosa						1
W1696	Mytilus edulis	juvenile	1	1	1	1	
W1977	Spisula solida	juvenile	1				
W2004	Pharus legumen	juvenile		1		2	
W2059	Abra alba					13	
W2152	Sphenia binghami					1	
W2166	Hiatella arctica					1	
Y0079	Alcyonidium mamillatum			P			
ZD0002	ASCIDIACEA	juvenile	1			3	
ZD0146	Molgula					1	

APEM Report No. 414118-01

Code	Taxa ID	Qualifiers	Notes
W1696	Mytilus edulis	juvenile	Commercially important as adults

Code	Taxa ID	Qualifiers	Notes
W1696	Mytilus edulis	juvenile	Commercially important as adults

EUROPEAN COMMISSION
JOINT RESEARCH CENTRE

Institute for Reference Materials and Measurements (Geel)

CERTIFIED REFERENCE MATERIAL
BCR® – 646

CERTIFICATE OF ANALYSIS

FRESH WATER SEDIMENT

	Mass fraction based on dry mass	
	Certified value 1) [µg/kg]	Uncertainty 2) [µg/kg]
TBT: $Sn(C_4H_9)_3^{+}$	480	80
DBT: $Sn(C_4H_9)_2^{2+}$	770	90
MBT: $Sn(C_4H_9)^{3+}$	610	120
TPhT: $Sn(C_6H_5)_3^{+}$	29	11
DPhT: $Sn(C_6H_5)_2^{2+}$	36	8
MPhT: $Sn(C_6H_5)^{3+}$	69	18

1) Unweighted mean value of the means of 6-14 accepted sets of data. The certified value is valid for the cation indicated. Unweighted mean of accepted mean values, independently obtained by 6 - 14 laboratories. The value is traceable to the International System of Units (SI).

2) The certified uncertainty is the expanded uncertainty with a coverage factor k = 2, corresponding to a level of confidence of about 95 %, comprising uncertainties from the characterisation and inhomogeneity studies .

This certificate is valid for one year after purchase.

Sales date:

The minimum amount of sample to be used is 600 mg.

NOTE

This material has been certified by BCR (Community Bureau of Reference, the former reference materials programme of the European Commission). The certificate has been revised under the responsibility of IRMM.

Brussels, December 2000
Latest revision: April 2015

Signed: ____________________

Prof. Dr. Hendrik Emons
European Commission
Joint Research Centre
Institute for Reference Materials and Measurements
Retieseweg 111
B-2440 Geel, Belgium

DESCRIPTION OF THE SAMPLE

The material consists of a dried and ground sediment sample with a particle size < 90 micrometer stored in an amber glass bottle. The bottle contains about 40 g of powder. Additional information on the preparation and the certified values is given in the certification report.

ANALYTICAL METHOD USED FOR CERTIFICATION

- Gas chromatography-quartz furnace atomic absorption spectrometry, (GC-QTAAS)
- Gas chromatography-flame photometric detection, (GC-FPD)
- Gas chromatography-mass spectrometry, (GC-MS)
- Gas chromatography-microwave induced plasma atomic emission spectrometry, (GC-MIP-AES)
- Gas chromatography-inductively coupled plasma mass spectrometry, GC-ICP-MS)
- High performance liquid chromatography-inductively coupled plasma mass spectrometry, (HPLC-ICP-MS)
- High performance liquid chromatography-fluorescence spectrometry, (HPLC-FLD)
- Polarography

PARTICIPANTS

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- ENEA, Casaccia, Divisione di Chimica Ambientale, Roma (IT)
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- Rijkswaterstaat, RIKZ, Haren (NL)
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- Universidad de Santiago, Depto. de Química Analítica, Santiago de Compostela (ES)
- Vrije Universiteit Amsterdam, Instituut voor Milieuvraagstukken, Amsterdam (NL)
- European Commission, Joint Research Centre, Institute for Reference Materials and Measurements, Geel (BE)

SAFETY INFORMATION

The usual laboratory safety precautions apply.

INSTRUCTIONS FOR USE

The material is intended for analytical purposes. The sample can be used as is from the bottle. Before a bottle is opened, it should be shaken manually for 5 min so that the material is thoroughly re-mixed. The correction to dry mass should be made on a separate portion of 100 mg which should be dried in an oven at 105 ºC for 3-4 h until constant mass is attained. The reuse of the material after opening of the bottle is under the responsibility of the user, i.e. the certified values are not guaranteed in bottles that have been opened and further stored. Moreover, taking into account the potential instability of the tin compounds, it is recommended that no bottles be used if the history of the storage conditions in the laboratory is not known in detail. Care has been taken to ensure that the certified value represents the "true" value at the time of arrival at the customer as closely as possible. When the reference material is used to assess the performance of a procedure, the user should refer to the recommendations of the certification report.

STORAGE

The tightly closed bottles should be kept at - 30 ºC in the dark for long term storage periods. Before closing the bottle after use, it is advisable to flush the bottle with a dry, inert gas. However, the European Commission cannot be held responsible for changes that happen during storage of the material at the customer's premises, especially of opened samples.

LEGAL NOTICE

NOTE

A technical report on the production of BCR-646 is available on the internet (http://www.irmm.jrc.be). A paper copy can be obtained from IRMM on request.

Certificate of Analysis

Standard Reference Material® 1944

New York/New Jersey Waterway Sediment

This Standard Reference Material (SRM) is a mixture of marine sediment collected near urban areas in New York and New Jersey. SRM 1944 is intended for use in evaluating analytical methods for the determination of selected polycyclic aromatic hydrocarbons (PAHs), polychlorinated biphenyl (PCB) congeners, chlorinated pesticides, and trace elements in marine sediment and similar matrices. Reference values are also provided for selected dibenzo-*p*-dioxin and dibenzofuran congeners, total organic carbon, total extractable material, and particle-size characteristics. All of the constituents for which certified, reference, and information values are provided in SRM 1944 were naturally present in the sediment material before processing. A unit of SRM 1944 consists of a bottle containing 50 g of radiation sterilized, freeze-dried sediment material.

Certified Concentration Values: Certified values for concentrations, expressed as mass fractions, for 24 PAHs, 35 PCB congeners (some in combination), four chlorinated pesticides, and nine trace elements are provided in Tables 1-4. A NIST certified value is a value for which NIST has the highest confidence in its accuracy in that all known or suspected sources of bias have been investigated or accounted for by NIST. The certified values for the PAHs, PCB congeners, and chlorinated pesticides are based on the agreement of results obtained at NIST from two or more chemically independent analytical techniques. The certified values for the trace elements are based on NIST measurements by one technique and additional results from several collaborating laboratories.

Reference Concentration Values: Reference values for concentrations, expressed as mass fractions, are provided for 32 additional PAHs (some in combination) in Table 5, seven additional chlorinated pesticides in Table 6, and 19 additional inorganic constituents in Tables 7 and 8. Reference values are provided in Table 9 for the 17 2,3,7,8-substituted polychlorinated dibenzo-*p*-dioxin and dibenzofuran congeners and total tetra-, penta-, hexa-, and hepta-congeners of polychlorinated dibenzo-*p*-dioxin and dibenzofuran. Reference values for particle-size characteristics are provided in Table 10. Reference values for total organic carbon and percent extractable mass are provided in Table 11. Reference values are noncertified values that are the best estimate of the true value; however, the values do not meet the NIST criteria for certification and are provided with associated uncertainties that may reflect only measurement precision, may not include all sources of uncertainty, or may reflect a lack of sufficient statistical agreement among multiple analytical methods. Explanations in support of each reference value are given as notes in Tables 5-11.

Information Concentration Values: Information values for concentrations, expressed as mass fractions, are provided in Table 12 for eight additional trace elements. An information value is considered to be a value that will be of interest and use to the SRM user, but insufficient information is available to assess the uncertainty associated with the value or only a limited number of analyses were performed.

Expiration of Certification: The certification of **SRM 1944** is valid, within the measurement uncertainty specified, until **31 March 2019**, provided the SRM is handled in accordance with instructions given in this certificate (see “Instructions for Use”). The certification is nullified if the SRM is damaged, contaminated, or otherwise modified.

Maintenance of SRM Certification: NIST will monitor this SRM over the period of its certification. If substantive technical changes occur that affect the certification before the expiration of this certificate, NIST will notify the purchaser. Registration (see attached sheet) will facilitate notification.

The coordination of the technical measurements leading to the certification was under the leadership of S.A. Wise of the NIST Analytical Chemistry Division.

Stephen A. Wise, Chief
Analytical Chemistry Division

Gaithersburg, MD 20899
Certificate Issue Date: 22 December 2008
See Certificate Revision History on Page17

Robert L. Watters, Jr., Chief
Measurement Services Division

Consultation on the statistical design of the experimental work and evaluation of the data were provided by M.G. Vangel and M.S. Levenson of the NIST Statistical Engineering Division.

Support aspects involved in the issuance of this SRM were coordinated through the NIST Measurement Services Division.

The sediment material was collected with the assistance of the New York District of the U.S. Army Corp of Engineers (ACENYD), who provided the expertise in the site selection, the ship, sampling equipment, and personnel. L. Rosman of ACENYD and R. Parris (NIST) coordinated the collection of this sediment material. Collection and preparation of SRM 1944 were performed by R. Parris, M. Cronise, and C. Fales (NIST); L. Rosman and P. Higgins (ACENYD); and the crew of the *Gelberman* from the ACE Caven Point facility in Caven Point, NJ.

Analytical measurements for the certification of SRM 1944 were performed at NIST by E.S. Beary, D.A. Becker, R. Demiralp, R.R. Greenberg, M. Lopez de Alda, K.E. Murphy, B.J. Porter, D.L. Poster, L.C. Sander, M.M. Schantz, and L. Walton of the Analytical Chemistry Division. Measurements for percent total organic carbon measurements were provided by three commercial laboratories and T.L. Wade of the Geochemical and Environmental Research Group, Texas A&M University (College Station, TX). The particle-size distribution data were provided by Honeywell, Inc. (Clearwater, FL).

Analytical measurements for the polychlorinated dibenzo-*p*-dioxins and dibenzofurans were the results of an interlaboratory comparison study among 14 laboratories (see Appendix A) coordinated by S.A. Wise of the NIST Analytical Chemistry Division and R. Turle and C. Chiu of Environment Canada, Environmental Technology Centre, Analysis and Air Quality Division (Ottawa, Ontario, Canada). Analytical measurements for selected trace elements were provided by the International Atomic Energy Agency (IAEA, Seibersdorf, Austria) by M. Makarewicz and R. Zeisler. Results were also used from seven laboratories (see Appendix B) that participated in an intercomparison exercise coordinated by S. Willie of the Institute for National Measurement Standards, National Research Council Canada (NRCC, Ottawa, Ontario, Canada).

NOTICE AND WARNING TO USERS

Storage: SRM 1944 must be stored in its original bottle at temperatures less than 30 °C away from direct sunlight.

Handling: This material is naturally occurring marine sediment from an urban area and may contain constituents of unknown toxicities; therefore, caution and care should be exercised during its handling and use.

INSTRUCTIONS FOR USE

Prior to removal of subsamples for analysis, the contents of the bottle should be mixed. The concentrations of constituents in SRM 1944 are reported on a dry-mass basis. The SRM, as received, contains approximately 1.3 % moisture. The sediment sample should be dried to a constant mass before weighing for analysis, or if the constituents of interest are volatile, a separate subsample of the sediment should be removed from the bottle at the time of analysis and dried to determine the concentration on a dry-mass basis.

PREPARATION AND ANALYSIS[1]

Sample Collection and Preparation: The sediment used to prepare this SRM was collected from six sites in the vicinity of New York Bay and Newark Bay in October 1994. Site selection was based on contaminant levels measured in previous samples from these sites and was intended to provide relatively high concentrations for a variety of chemical classes of contaminants. The sediment was collected using an epoxy-coated modified Van Veen-type grab sampler designed to sample the sediment to a depth of 10 cm. A total of approximately 2100 kg of wet sediment was collected from the six sites. The sediment was freeze-dried, sieved (nominally 250 µm to 61 µm), homogenized in a cone blender, radiation sterilized (^{60}Co), and then packaged in screw-capped amber glass bottles.

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[1] Certain commercial equipment, instruments or materials are identified in this certificate to adequately specify the experimental procedure. Such identification does not imply recommendation or endorsement by the National Institute of Standards and Technology, nor does it imply that the materials or equipment identified are necessarily the best available for the purpose.

Conversion to Dry-Mass Basis: The results for the constituents in SRM 1944 are reported on a dry-mass basis; however, the material “as received” contains residual moisture. The amount of moisture in SRM 1944 was determined by measuring the mass loss after freeze-drying subsamples of 1.6 g to 2.5 g for five days at 1 Pa with a -10 °C shelf temperature and a -50 °C condenser temperature. The moisture content in SRM 1944 at the time of the certification analyses was 1.25 % ± 0.03 % (95 % confidence level).

Polycyclic Aromatic Hydrocarbons: The general approach used for the value assignment of the PAHs in SRM 1944 was similar to that reported for the recent certification of several environmental matrix SRMs [1-5] and consisted of combining results from analyses using various combinations of different extraction techniques and solvents, cleanup/isolation procedures, and chromatographic separation and detection techniques. This approach consisted of Soxhlet extraction and pressurized fluid extraction (PFE) using dichloromethane (DCM) or a hexane/acetone mixture, cleanup of the extracts using solid phase extraction (SPE) or normal-phase liquid chromatography (LC), followed by analysis using the following techniques: (1) reversed-phase liquid chromatography with fluorescence detection (LC-FL) for analysis of the total PAH fraction, (2) reversed-phase LC-FL analysis of isomeric PAH fractions isolated by normal-phase LC (i.e., multidimensional LC), (3) gas chromatography/mass spectrometric (GC/MS) analysis of the PAH fraction on three stationary phases of different selectivity, i.e., a 5 % (mole fraction) phenyl-substituted methylpolysiloxane phase, a 50 % (mole fraction) phenyl-substituted methylpolysiloxane phase, and a smectic liquid crystalline stationary phase.

Six sets of GC/MS results, designated as GC/MS (I), GC/MS (II), GC/MS (III), GC/MS (IV), GC/MS (V), and GC/MS (Sm), were obtained using three columns with different selectivities for the separation of PAHs. For GC/MS (I) analyses, duplicate subsamples of 1 g from eight bottles of SRM 1944 were Soxhlet extracted for 24 h with DCM. Copper powder was added to the extract to remove elemental sulfur. The concentrated extract was passed through a silica SPE cartridge and eluted with 2 % DCM in hexane. The processed extract was then analyzed by GC/MS using a 0.25 mm i.d. × 60 m fused silica capillary column with a 5 % phenyl-substituted methylpolysiloxane phase (0.25 µm film thickness) (DB-5 MS, J&W Scientific, Folsom, CA). The GC/MS (II) analyses were performed using 1 g to 2 g subsamples from three bottles of SRM 1944 and 2 g to 3 g subsamples from three bottles of SRM 1944 that had been mixed with a similar amount of water (i.e., a wetted sediment). These samples were Soxhlet extracted with DCM and processed through the silica SPE as described above; however, the extract was further fractionated using normal-phase LC on a semi-preparative aminopropylsilane column to isolate the PAH fraction [6-9]. The PAH fraction was then analyzed using the same column as described above for GC/MS (I); however, the subsamples were extracted, processed and analyzed as part of three different sample sets at different times using different calibrations for each set. For the GC/MS (III), 1 g to 2 g subsamples from six bottles of SRM 1944 were Soxhlet extracted for 18 h with 250 mL of a mixture of 50 % hexane/50 % acetone (volume fractions). The extracts were then processed and analyzed as described for GC/MS (II). For GC/MS (IV) analyses, 1 g to 2 g subsamples from six bottles of SRM 1944 were extracted using PFE with a mixture of 50 % hexane/50 % acetone as described by Schantz et al. [10], and the extracts were processed as described above for GC/MS (II). The GC/MS (V) results were obtained by analyzing three of the same PAH fractions that were analyzed in GC/MS (III) and three of the PAH fractions that were analyzed in GC/MS (IV) using a 50 % phenyl-substituted methylpolysiloxane stationary phase (0.25 mm i.d. × 60 m, 0.25 µm film thickness) (DB-17MS, J&W Scientific, Folsom, CA). For GC/MS (Sm) 1 g to 2 g subsamples from six bottles of SRM 1944 were Soxhlet extracted for 24 h with 250 mL of DCM. The extracts were processed as described above for GC/MS (I) using an aminopropylsilane SPE cartridge followed by GC/MS analysis using 0.2 mm i.d. × 25 m (0.15 µm film thickness) smectic liquid crystalline phase (SB-Smectic, Dionex, Lee Scientific Division, Salt Lake City, UT).

Two sets of LC-FL results, designated as LC-FL (Total) and LC-FL (Fraction), were used in the certification process. Subsamples of approximately 1 g from six bottles of SRM 1944 were Soxhlet extracted for 20 h using 200 mL of 50 % hexane/50 % acetone (volume fractions). The extracts were concentrated and then processed through two aminopropylsilane solid phase extraction (SPE) cartridges connected in series to obtain the total PAH fraction. A second 1 g subsample from the six bottles was Soxhlet extracted and processed as described above; the PAH fraction was then fractionated further on a semi-preparative aminopropylsilane column (µBondapak NH_2, 9 mm i.d. × 30 cm, Waters Associates, Milford, MA) to isolate isomeric PAH fractions as described previously [6-9]. The total PAH fraction and the isomeric PAH fractions were analyzed using a 5-µm particle-size polymeric octadecylsilane (C_{18}) column (4.6 mm i.d. × 25 cm, Hypersil-PAH, Keystone Scientific, Inc., Bellefonte, PA) with wavelength programmed fluorescence detection [7,8]. For all of the GC/MS and LC-FL measurements described above, selected perdeuterated PAHs were added to the sediment prior to solvent extraction for use as internal standards for quantification purposes.

Homogeneity Assessment for PAHs: The homogeneity of SRM 1944 was assessed by analyzing duplicate samples of 1 g from eight bottles selected by stratified random sampling. Samples were extracted, processed, and analyzed as described above for GC/MS (I). No statistically significant differences among bottles were observed for the PAHs at the 1 g sample size.

PCBs and Chlorinated Pesticides: The general approach used for the determination of PCBs and chlorinated pesticides in SRM 1944 was similar to that reported for the recent certification of several environmental matrix SRMs [2,4,11,12,13], and consisted of combining results from analyses using various combinations of different extraction techniques and solvents, cleanup/isolation procedures, and chromatographic separation and detection techniques. This approach consisted of Soxhlet extraction and PFE using DCM or a hexane/acetone mixture, cleanup/isolation using SPE or LC, followed by analysis using GC/MS and gas chromatography with electron capture detection (GC-ECD) on two columns with different selectivity.

Eight sets of results were obtained designated as GC-ECD (I) A and B, GC-ECD (II) A and B, GC/MS (I), GC/MS (II), GC/MS (III), and QA Exercise. For the GC-ECD (I) analyses, 1 g subsamples from four bottles of SRM 1944 were Soxhlet extracted with DCM for 18 h. Copper powder was added to the extract to remove elemental sulfur. The concentrated extract was passed through a silica SPE cartridge and eluted with 10 % DCM in hexane. The concentrated eluant was then fractionated on a semi-preparative aminopropylsilane column to isolate two fractions containing: (1) the PCBs and lower polarity pesticides, and (2) the more polar pesticides. GC-ECD analyses of the two fractions were performed on two columns of different selectivities for PCB separations: 0.25 mm × 60 m fused silica capillary column with a 5 % phenyl-substituted methylpolysiloxane phase (0.25 µm film thickness) (DB-5, J&W Scientific, Folsom, CA) and a 0.32 mm × 100 m fused silica capillary column with a 50 % (mole fraction) octadecyl (C-18) methylpolysiloxane phase (0.1 µm film thickness) (CPSil 5 C18 CB, Chrompack International, Middelburg, The Netherlands). The results from the 5 % phenyl phase are designated as GC-ECD (IA) and the results from the C-18 phase are designated as GC-ECD (IB). A second set of samples was also analyzed by GC-ECD (i.e., GC-ECD IIA and IIB). Subsamples of 1 g to 2 g from three bottles of SRM 1944 and 2 g to 3 g subsamples from three bottles of SRM 1944 that had been mixed with a similar amount of water (i.e., a wetted sediment) were extracted, processed, and analyzed as described above for GC-ECD (I); however, the subsamples were extracted, processed and analyzed as part of three different sample sets at different times using different calibrations for each set.

Three sets of results were obtained by GC/MS. For GC/MS (I), 1 g to 2 g subsamples from six bottles were Soxhlet extracted with a mixture of 50 % hexane/50 % acetone. Copper powder was added to the extract to remove elemental sulfur. The concentrated extract was passed through a silica SPE cartridge and eluted with 10 % DCM in hexane. The extract was then analyzed by GC/MS using a 0.25 mm × 60 m fused silica capillary column with a 5 % phenyl-substituted methylpolysiloxane phase (0.25 µm film thickness). The GC/MS (II) results were obtained in the same manner as the GC/MS (I) analyses except that the six subsamples were extracted using PFE as described by Schantz et al. [10]. The GC/MS (III) analyses were performed on the same extract fractions analyzed in GC-ECD (II) using the 5 % phenyl-substituted methylpolysiloxane phase describe above for GC/MS (I). For both the GC-ECD and GC/MS analyses, two PCB congeners that are not significantly present in the sediment extract (PCB 103 and PCB 198 [14,15]), and 4,4'-DDT-d_8 were added to the sediment prior to extraction for use as internal standards for quantification purposes.

In addition to the analyses performed at NIST, SRM 1944 was used in an interlaboratory comparison exercise in 1995 as part of the NIST Intercomparison Exercise Program for Organic Contaminants in the Marine Environment [16]. Results from 19 laboratories that participated in this exercise were used as the eighth data set in the determination of the certified values for PCB congeners and chlorinated pesticides in SRM 1944. The laboratories participating in this exercise used the analytical procedures routinely used in their laboratories to measure PCB congeners and chlorinated pesticides.

Polychlorinated Dibenzo-*p*-dioxins and Dibenzofurans: Value assignment of the concentrations of the 17 2,3,7,8-substituted polychlorinated dibenzo-*p*-dioxin and dibenzofuran congeners and the total tetra- through hepta-substituted polychlorinated dibenzo-*p*-dioxins and dibenzofurans was accomplished by combining results from the analysis of SRM 1944 by 14 laboratories that participated in an interlaboratory comparison study (see Appendix A). Each laboratory analyzed three subsamples (typically 1 g) of SRM 1944 using their routine analytical procedures and gas chromatography with high resolution mass spectrometric detection (GC-HRMS).

The analytical procedures used by all of the laboratories included spiking with ^{13}C-labeled surrogates (internal standards); Soxhlet extraction with toluene; sample extract cleanup with acid/base silica, alumina, and carbon columns; and finally analysis of the cleaned up extract with GC-HRMS. Most of the laboratories used a 5 % phenyl-substituted methylpolysiloxane phase capillary column (DB-5), and about half of the laboratories confirmed 2,3,7,8-tetrachlorodibenzofuran using a 50 % (mole fraction) cyanopropylphenyl-substituted methylpolysiloxane capillary column (DB-225, J&W Scientific, Folsom, CA).

Analytical Approach for Inorganic Constituents: Value assignment for the concentrations of selected trace elements was accomplished by combining results of the analyses of SRM 1944 from NIST, NRCC, IAEA, and seven selected laboratories that participated in an interlaboratory comparison exercise coordinated by the NRCC [17] (see Appendix B). A similar approach was recently used to provide certified and reference concentration values for trace elements in two mussel tissue materials [18-20]. The analytical methods used for the determination of each element are summarized in Table 13. For the certified concentration values listed in Table 4, results were combined from: (1) analyses at NIST using isotope dilution inductively coupled plasma mass spectrometry (ID-ICPMS) or instrumental neutron activation analysis (INAA), (2) analyses at NRCC using ID-ICPMS, graphite furnace atomic absorption spectrometry (GFAAS), and/or inductively coupled plasma atomic emission spectroscopy (ICPAES), (3) analyses at IAEA using INAA, and (4) the mean of the results from seven selected laboratories that participated in the NRC interlaboratory comparison exercise. The reference concentration values in Table 7 were determined by combining results from (1) analyses performed at NIST using INAA; (2) analyses at NRCC using ID-ICPMS, GFAAS, ICPAES, and/or cold vapor atomic absorption spectroscopy (CVAAS); (3) analyses at IAEA using INAA; and (4) the mean of the results from five to seven laboratories that participated in the NRCC interlaboratory comparison exercise. The information concentration values in Table 12 were determined by INAA at NIST and IAEA.

NIST Analyses using ID-ICPMS: Lead, cadmium, and nickel were determined by ID-ICPMS [21]. Subsamples (0.4 g to 0.5 g) from six bottles of the SRM were spiked with ^{206}Pb, ^{111}Cd, and ^{62}Ni and wet ashed using a combination of nitric, hydrochloric, hydrofluoric, and perchloric acids. Lead and cadmium were determined in the same sample; nickel was determined in a second sample set. A small amount of crystalline material remained after the acid dissolution. Lithium metaborate fusion was performed on this residue to confirm that the residue contained insignificant amounts of the analytes. Cadmium and nickel were separated from the matrix material to eliminate the possibility of spectral interferences, and concentrations were determined from the measurement of the ^{112}Cd/^{111}Cd and ^{62}Ni/^{60}Ni ratios, respectively. The ^{208}Pb/^{206}Pb ratios were measured directly because interferences at these masses are negligible.

NIST Analyses using INAA: Analyses were performed in two steps [22]. Elements with short-lived irradiation products (Al, Ca, Cl, K, Mg, Mn, Na, Ti, and V) were determined by measuring duplicate 300 mg samples from each of 10 bottles of SRM 1944. The samples, standards, and controls were packaged in clean polyethylene bags and were individually irradiated for 15 s in the NIST Reactor Pneumatic Facility RT-4. Reactor power was 20 megawatts which corresponds to a neutron fluence rate of about 8×10^{13} cm^{-2}·s^{-1}. After irradiation, the samples, controls, and standards were repackaged in clean polyethylene bags and counted (gamma-ray spectrometry) three times at different decay intervals. A sample to detector distance (counting geometry) of 20 cm was used. Elements with long-lived irradiation products (Ag, As, Br, Co, Cr, Cs, Fe, Rb, Sb, Sc, Se, Th, and Zn) were determined by measuring one 300 mg sample from each of nine bottles of SRM 1944. The samples, standards, controls, and blank polyethylene bags were irradiated together for a total of 1 h at a reactor power of 20 megawatts. Approximately four days after irradiation, the polyethylene bags were removed, and each sample, standard, control, and blank was counted at 20 cm from the detector. The samples were then recounted at 10 cm from another detector. After an additional decay time of about one month, the samples, standards, controls, and blanks were counted a third time (at 10 cm) from the second detector.

Particle-Size Information: Dry particle-size distribution measurements for SRM 1944 were obtained as part of a collaborative effort with Honeywell's Particle and Components Measurements Laboratory (Clearwater, FL). A Microtrac particle analyzer, which makes use of light-scattering techniques, was used to measure the particle-size distribution of SRM 1944. Briefly, a reference beam is used to penetrate a field of particles and the light that scatters in the forward direction from the field is measured and the particle size as a volume distribution is derived via a computer-assisted analysis. From these data, the total volume, average size, and a characteristic width of the particle-size distribution are calculated. The system has a working range from 0.7 µm to 700 µm.

Total Organic Carbon and Percent Extractable Mass: Four laboratories provided results for Total Organic Carbon (TOC) using similar procedures. Briefly, subsamples of approximately 200 mg were reacted with 6 N hydrochloric acid and rinsed with deionized water prior to combustion in a gas fusion furnace. The carbon monoxide and carbon dioxide produced were measured and compared to a blank for calculation of the percent TOC. Each laboratory analyzed subsamples from six bottles of SRM 1944. For the determination of percent extractable mass, six subsamples of approximately 1 g to 2 g of SRM 1944 were extracted using Soxhlet extraction for 18 h with DCM. The extraction thimbles were allowed to air dry. After reaching constant mass, the difference in the mass before and after extraction was determined.

Table 1. Certified Concentrations for Selected PAHs in SRM 1944

PAHs	Mass Fractions in mg/kg (dry-mass basis)(a,b)		
Naphthalene(c,d,e,f,g)	1.65	±	0.31
Phenanthrene(c,d,e,f,g)	5.27	±	0.22
Anthracene(c,d,e,f,g)	1.77	±	0.33
Fluoranthene(c,d,e,f,g)	8.92	±	0.32
Pyrene(c,d,e,f,g)	9.70	±	0.42
Benzo[*c*]phenathrene(c,d,e,f,h)	0.76	±	0.10
Benz[*a*]anthracene(c,d,e,f,g,h)	4.72	±	0.11
Chrysene(h,k)	4.86	±	0.10[i]
Triphenylene(h,k)	1.04	±	0.27
Benzo[*b*]fluoranthene(g,h,j)	3.87	±	0.42
Benzo[*j*]fluoranthene(h,j)	2.09	±	0.44
Benzo[*k*]fluoranthene(c,d,e,f,g,h,j)	2.30	±	0.20
Benzo[*a*]fluoranthene(c,d,e,f,h,,j)	0.78	±	0.12
Benzo[*e*]pyrene(c,d,e,f,h,j)	3.28	±	0.11
Benzo[*a*]pyrene(c,d,e,f,g,h,j)	4.30	±	0.13
Perylene(c,d,e,f,g,h,j)	1.17	±	0.24
Benzo[*ghi*]perylene(c,d,e,f,j,k)	2.84	±	0.10
Indeno[1,2,3-*cd*]pyrene(c,d,e,f,j,k)	2.78	±	0.10
Dibenz[*a,j*]anthracene(c,d,e,f,j,k)	0.500	±	0.044
Dibenz[*a,c*]anthracene(j,k)	0.335	±	0.013
Dibenz[*a,h*]anthracene(j,k)	0.424	±	0.069
Pentaphene(c,d,e,f,j,k)	0.288	±	0.026
Benzo[*b*]chrysene(c,d,e,f,j,k,h)	0.63	±	0.10
Picene(c,d,e,f,j,k)	0.518	±	0.093

(a) Concentrations reported on dry-mass basis; material as received contains approximately 1.3 % moisture.

(b) The results are expressed as the certified value ± the expanded uncertainty. Each certified value is a mean of the means from two or more analytical methods, weighted as described in Paule and Mandel [23]. Each uncertainty, computed according to the CIPM approach as described in the ISO and NIST Guides [24], is an expanded uncertainty at the 95 % level of confidence, which includes random sources of uncertainty within each analytical method as well as uncertainty due to the drying study. The expanded uncertainty defines a range of values within which the true value is believed to lie, at a level of confidence of approximately 95 %.

(c) GC/MS (I) on 5 % phenyl-substituted methylpolysiloxane phase after Soxhlet extraction with DCM.

(d) GC/MS (II) on 5 % phenyl-substituted methylpolysiloxane phase after Soxhlet extraction with DCM.

(e) GC/MS (III) on 5 % phenyl-substituted methylpolysiloxane phase after Soxhlet extraction with 50 % hexane/50 % acetone.

(f) GC/MS (IV) on 5 % phenyl-substituted methylpolysiloxane phase after PFE with 50 % hexane/50 % acetone.

(g) LC-FL of total PAH fraction after Soxhlet extraction with 50 % hexane/50 % acetone.

(h) GC/MS (Sm) using a smectic liquid crystalline phase after Soxhlet extraction with DCM.

(i) The uncertainty interval for chrysene was widened based on expert consideration of the analytical methods and analysis of the data for all PAHs, which suggests that the half-widths of the expanded uncertainties should not be less than 2 %.

(j) GC/MS (V) on 50 % phenyl-substituted methylpolysiloxane phase of extracts from GC/MS (III) and GC/MS (IV).

(k) LC-FL of isomeric PAH fractions after Soxhlet extraction with 50 % hexane/50 % acetone.

Table 2. Certified Concentrations for Selected PCB Congeners(a) in SRM 1944

PCB Congeners		Mass Fractions in µg/kg (dry-mass basis) (b,c)		
PCB 8	(2,4'-Dichlorobiphenyl)(d,e,f,g,h,i,j,k)	22.3	±	2.3
PCB 18	(2,2',5-Trichlorobiphenyl)(d,e,f,g,h,i,j,k)	51.0	±	2.6
PCB 28	(2,4,4'-Trichlorobiphenyl) (d,e,f,g,j,k)	80.8	±	2.7
PCB 31	(2,4',5-Trichlorobiphenyl) (d,e,f,g,j)	78.7	±	1.6[l]
PCB 44	(2,2',3,5'-Tetrachlorobiphenyl) (d,e,f,g,h,i,j,k)	60.2	±	2.0
PCB 49	(2,2',4,5'-Tetrachlorobiphenyl) (d,e,f,g,h,i,j,k)	53.0	±	1.7
PCB 52	(2,2',5,5'-Tetrachlorobiphenyl) (d,e,f,g,h,i,j,k)	79.4	±	2.0
PCB 66	(2,3',4,4'-Tetrachlorobiphenyl) (e,g,h,i,j)	71.9	±	4.3
PCB 87	(2,2',3,4,5'-Pentachlorobiphenyl) (d,e,f,g,h,i,j)	29.9	±	4.3
PCB 95	(2,2',3,5',6-Pentachlorobiphenyl) (e,g,h,i,j)	65.0	±	8.9
PCB 99	(2,2',4,4',5-Pentachlorobiphenyl) (d,e,f,g,h,i,j,k)	37.5	±	2.4
PCB 101	(2,2',4,5,5'-Pentachlorobiphenyl) (d,e,f,g,h,i,j,k)	73.4	±	2.5
90	(2,2',3,4',5-Pentachlorobiphenyl)			
PCB 105	(2,3,3',4,4'-Pentachlorobiphenyl) (e,f,g,h,i,j,k)	24.5	±	1.1
PCB 110	(2,3,3',4',6-Pentachlorobiphenyl) (g,h,i,j)	63.5	±	4.7
PCB 118	(2,3',4,4',5-Pentachlorobiphenyl) (d,e,f,g,h,i,j,k)	58.0	±	4.3
PCB 128	(2,2',3,3',4,4'-Hexachlorobiphenyl) (d,e,f,g,h,i,j,k)	8.47	±	0.28
PCB 138	(2,2',3,4,4',5'-Hexachlorobiphenyl) (d,e,f,g,h,i,j,k)	62.1	±	3.0
163	(2,3,3',4',5,6-Hexachlorobiphenyl)			
164	(2,3,3',4',5',6-Hexachlorobiphenyl)			
PCB 149	(2,2',3,4',5',6-Hexachlorobiphenyl) (d,e,f,g,h,i,j,k)	49.7	±	1.2
PCB 151	(2,2',3,5,5',6-Hexachlorobiphenyl) (d,e,f,g,h,i,j,k)	16.93	±	0.36
PCB 153	(2,2',4,4',5,5'-Hexachlorobiphenyl) (d,e,f,g,h,i,j,k)	74.0	±	2.9
PCB 156	(2,3,3',4,4',5-Hexachlorobiphenyl) (d,e,f,g,h,i,j)	6.52	±	0.66
PCB 170	(2,2',3,3',4,4',5-Heptachlorobiphenyl) (d,e,f,g,h,i,j,k)	22.6	±	1.4
190	(2,3,3',4,4',5,5'-Heptachlorobiphenyl)			
PCB 180	(2,2',3,4,4',5,5'-Heptachlorobiphenyl) (d,e,f,g,h,i,j,k)	44.3	±	1.2
PCB 183	(2,2',3,4,4',5',6-Heptachlorobiphenyl) (d,e,f,g,h,i,j)	12.19	±	0.57
PCB 187	(2,2',3,4',5,5',6-Heptachlorobiphenyl) (d,e,f,g,h,i,j,k)	25.1	±	1.0
159	(2,3,3',4,5,5'-Hexachlorobiphenyl)			
182	(2,2',3',4,4',5,6'-Heptachlorobiphenyl)			
PCB 194	(2,2',3,3',4,4',5,5'-Octachlorobiphenyl) (d,e,f,g,h,i,j)	11.2	±	1.4
PCB 195	(2,2',3,3',4,4',5,6-Octachlorbiphenyl) (d,e,f,g,h,i,j,k)	3.75	±	0.39
PCB 206	(2,2',3,3',4,4',5,5',6-Nonachlorobiphenyl) (d,e,f,g,h,i,j,k)	9.21	±	0.51
PCB 209	Decachlorobiphenyl (d,e,f,g,h,i,j,k)	6.81	±	0.33

(a) PCB congeners are numbered according to the scheme proposed by Ballschmiter and Zell [14] and later revised by Schulte and Malisch [15] to conform with IUPAC rules; for the specific congeners mentioned in this SRM, the Ballschmiter-Zell numbers correspond to those of Schulte and Malisch. When two or more congeners are known to coelute under the conditions used, the congener listed first is the major component; additional congeners may be present as minor components.

(b) Concentrations reported on dry-mass basis; material as received contains approximately 1.3 % moisture.

(c) The results are expressed as the certified value ± the expanded uncertainty. Each certified value is a mean of the means from two or more analytical methods, weighted as described in Paule and Mandel [23]. Each uncertainty, computed according to the CIPM approach as described in the ISO and NIST Guides [24], is an expanded uncertainty at the 95 % level of confidence, which includes random sources of uncertainty within each analytical method as well as uncertainty due to the drying study. The expanded uncertainty defines a range of values within which the true value is believed to lie, at a level of confidence of approximately 95 %.

(d) GC-ECD (IA) on 5 % phenyl-substituted methylpolysiloxane phase after Soxhlet extraction with DCM.

(e) GC-ECD (IB) on the 50 % C-18 dimethylpolysiloxane phase; same extracts analyzed as in GC-ECD (IA).

(f) GC-ECD (IIA) on 5 % phenyl-substituted methylpolysiloxane phase after Soxhlet extraction with DCM.

(g) GC-ECD (IIB) on the 50 % octadecyl (C-18) methylpolysiloxane phase; same extracts analyzed as in GC-ECD (IIA).

(h) GC/MS (I) on 5 % phenyl-substituted methylpolysiloxane phase after Soxhlet extraction with 50 % hexane/50 % acetone.

(i) GC/MS (II) on 5 % phenyl-substituted methylpolysiloxane phase after PFE extraction with 50 % hexane/50 % acetone.

(j) GC/MS (III) on 5 % phenyl-substituted methylpolysiloxane phase; same extracts analyzed as in GC-ECD (IIA).

(k) Results from 19 laboratories participating in an interlaboratory comparison exercise.

(l) The uncertainty interval for PCB 31 was widened based on expert consideration of the analytical methods and analysis of the data for all PCB congeners, which suggests that the half-widths of the expanded uncertainties should not be less than 2 %.

Table 3. Certified Concentrations for Selected Chlorinated Pesticides in SRM 1944

Chlorinated Pesticides	Mass Fractions in µg/kg (dry-mass basis)(a,b)
Hexachlorobenzene(e,f,g,h,i,j)	6.03 ± 0.35
cis-Chlordane (α-Chlordane) (c,d,e,f,g,h,i,j)	16.51 ± 0.83
trans-Nonachlor(c,d,e,f,g,h,i,j)	8.20 ± 0.51
4,4'-DDT(c,d,e,f,g,h,i,j)	119 ± 11

(a) Concentrations reported on dry-mass basis; material as received contains approximately 1.3 % moisture.

(b) The results are expressed as the certified value ± the expanded uncertainty. Each certified value is a mean of the means from two or more analytical methods, weighted as described in Paule and Mandel [23]. Each uncertainty, computed according to the CIPM approach as described in the ISO and NIST Guides [24], is an expanded uncertainty at the 95 % level of confidence, which includes random sources of uncertainty within each analytical method as well as uncertainty due to the drying study. The expanded uncertainty defines a range of values within which the true value is believed to lie, at a level of confidence of approximately 95 %.

(c) GC-ECD (IA) on 5 % phenyl-substituted methylpolysiloxane phase after Soxhlet extraction with DCM.

(d) GC-ECD (IB) on the 50 % octadecyl (C-18) methylpolysiloxane phase; same extracts analyzed as in GC-ECD (IA).

(e) GC-ECD (IIA) on 5 % phenyl-substituted methylpolysiloxane phase after Soxhlet extraction with DCM.

(f) GC-ECD (IIB) on the 50 % octadecyl (C-18) methylpolysiloxane phase; same extracts analyzed as in GC-ECD (IIA).

(g) GC/MS (I) on 5 % phenyl-substituted methylpolysiloxane phase after Soxhlet extraction with 50 % hexane/50 % acetone.

(h) GC/MS (II) on 5 % phenyl-substituted methylpolysiloxane phase after PFE extraction with 50 % hexane/50 % acetone.

(i) GC/MS (III) on 5 % phenyl-substituted methylpolysiloxane phase; same extracts analyzed as in GC-ECD (IIA).

(j) Results from 19 laboratories participating in an interlaboratory comparison exercise.

Table 4. Certified Concentrations for Selected Inorganic Constituents in SRM 1944

Elements	Degrees of Freedom	Mass Fractions in percent (dry-mass basis)(a,b)
Aluminum(c,d,e)	4	5.33 ± 0.49
Iron(c,d,e)	6	3.53 ± 0.16
		Mass Fractions in mg/kg (dry-mass basis)(a,b)
Arsenic(c,d,e,f,g)	10	18.9 ± 2.8
Cadmium(c,f,h,i)	6	8.8 ± 1.4
Chromium(c,d,f,g,i)	9	266 ± 24
Lead(c,h,i)	5	330 ± 48
Manganese(c,d,e)	8	505 ± 25
Nickel(c,g,h,i)	6	76.1 ± 5.6
Zinc(c,d,e,g,i)	9	656 ± 75

(a) The results are expressed as the certified value ± the expanded uncertainty. The certified value is the mean of four results: (1) the mean of NIST INAA or ID-ICPMS analyses, (2) the mean of two methods performed at NRCC, and (3) the mean of results from seven selected laboratories participating in the NRCC intercomparison exercise, and (4) the mean results from INAA analyses at IAEA. The expanded uncertainty in the certified value is equal to $U = ku_c$, where u_c is the combined standard uncertainty and k is the coverage factor, both calculated according to the ISO and NIST Guides [24]. The value of u_c is intended to represent at the level of one standard deviation the combined effect of all the uncertainties in the certified value. Here u_c accounts for both possible method biases, within-method variation, and material inhomogeneity. The coverage factor, k, is the Student's t-value for a 95 % prediction interval with the corresponding degrees of freedom. Because of the material inhomogeneity, the variability among the measurements of multiple samples can be expected to be greater than that due to measurement variability alone.

(b) Concentrations reported on dry-mass basis; material as received contains approximately 1.3 % moisture.

(c) Results from five to seven laboratories participating in the NRCC interlaboratory comparison exercise.

(d) Measured at NIST using INAA.

(e) Measured at NRCC using ICPAES.

(f) Measured at NRCC using GFAAS.

(g) Measured at IAEA using INAA.

(h) Measured at NIST using ID-ICPMS.

(i) Measured at NRCC using ID-ICPMS.

Table 5. Reference Concentrations for Selected PAHs in SRM 1944

NOTE: These concentrations are provided as reference values because either the results have not been confirmed by an independent analytical technique as required for certification or the agreement among results from multiple methods was insufficient for certification. Although bias has not been evaluated for the procedures used, the reference values should be useful for comparison with results obtained using similar procedures.

PAHs	Mass Fractions in mg/kg (dry-mass basis)(a,b)		
1-Methylnaphthalene(c,d,e,f)	0.52	±	0.03
2-Methylnaphthalene(c,d,e,f)	0.95	±	0.05
Biphenyl(c,d,e,f)	0.32	±	0.07
Acenaphthene(c,d,e,f)	0.57	±	0.03
Fluorene(c,d,e,f)	0.85	±	0.03
Dibenzothiophene(d,e,f)	0.62	±	0.01(g)
1-Methylphenanthrene(c,d,e,f)	1.7	±	0.1
2-Methylphenanthrene(c,d,e,f)	1.90	±	0.06
3-Methylphenanthrene(c,d,e,f)	2.1	±	0.1
4-Methylphenanthrene and 9-Methylphenanthrene(c,d,e,f)	1.6	±	0.2
2-Methylanthracene(c,d,e,f)	0.58	±	0.04
3,5-Dimethylphenanthrene(c)	1.31	±	0.04
2,6-Dimethylphenanthrene(c)	0.79	±	0.02(g)
2,7-Dimethylphenanthrene(c)	0.67	±	0.02(g)
3,9-Dimethylphenanthrene(c)	2.42	±	0.05(g)
1,6-, 2,9-, and 2,5-Dimethylphenanthrene(c)	1.67	±	0.03(g)
1,7-Dimethylphenanthrene(c)	0.62	±	0.02(g)
1,9- and 4,9-Dimethylphenanthrene(c)	1.20	±	0.03(g)
1,8-Dimethylphenanthrene(c)	0.24	±	0.01(g)
1,2-Dimethylphenanthrene(c)	0.28	±	0.01(g)
8-Methylfluoranthene(c)	0.86	±	0.02(g)
7-Methylfluoranthene(c)	0.69	±	0.02
1-Methylfluoranthene(c)	0.66	±	0.02(g)
3-Methylfluoranthene(c)	2.46	±	0.07
2-Methylpyrene(c)	1.81	±	0.04(g)
4-Methylpyrene(c)	1.44	±	0.03(g)
1-Methylpyrene(c)	1.29	±	0.03
Anthanthrene(h)	0.9	±	0.1

(a) Concentrations reported on dry-mass basis; material as received contains approximately 1.3 % moisture.

(b) The reference value for each analyte is the equally-weighted mean of the means from two or more analytical methods or the mean from one analytical technique. The uncertainty in the reference value defines a range of values that is intended to function as an interval that contains the true value at a level of confidence of 95 %. This uncertainty includes sources of uncertainty within each analytical method, among methods, and from the drying study.

(c) GC/MS (I) on 5 % phenyl-substituted methylpolysiloxane phase after Soxhlet extraction with DCM.

(d) GC/MS (II) on 5 % phenyl-substituted methylpolysiloxane phase after Soxhlet extraction with DCM.

(e) GC/MS (III) on 5 % phenyl-substituted methylpolysiloxane phase after Soxhlet extraction with 50 % hexane/50 % acetone.

(f) GC/MS (IV) on 5 % phenyl-substituted methylpolysiloxane phase after PFE with 50 % hexane/50 % acetone.

(g) The uncertainty interval for this compound was widened in accordance with expert consideration of the analytical procedures, along with the analysis of the data as a whole, which suggests that the half-widths of the expanded uncertainties should not be less than 2 %.

(h) LC-FL of isomeric PAH fractions after Soxhlet extraction with 50 % hexane/50 % acetone.

Table 6. Reference Concentrations for Selected Chlorinated Pesticides in SRM 1944

NOTE: These concentrations are provided as reference values because either the results have not been confirmed by an independent analytical technique as required for certification or the agreement among results from multiple methods was insufficient for certification. Although bias has not been evaluated for the procedures used, the reference values should be useful for comparison with results obtained using similar procedures.

Chlorinated Pesticides	Mass Fractions in µg/kg (dry-mass basis)(a,b)
α-HCH(c,d,e,f)	2.0 ± 0.3
trans-Chlordane (γ-Chlordane) (c,d,e,f,g,h,i,j)	8 ± 2
cis-Nonachlor (d,e,f,i,j)	3.7 ± 0.7
2,4'-DDE (c,d,e,f,g,h,i,j)	19 ± 3
2,4'-DDD (e,f,g,h,i,j)	38 ± 8
4,4'-DDE (c,d,e,f,g,h,i,j)	86 ± 12
4,4'-DDD (c,d,e,f,g,h,i,j)	108 ± 16

(a) Concentrations reported on dry-mass basis; material as received contains approximately 1.3 % moisture.

(b) The reference value for each analyte is the equally-weighted mean of the means from two or more analytical methods or the mean from one analytical technique. The uncertainty in the reference value defines a range of values that is intended to function as an interval that contains the true value at a level of confidence of 95 %. This uncertainty includes sources of uncertainty within each analytical method, among methods, and from the drying study.

(c) GC-ECD (IA) on 5 % phenyl-substituted methylpolysiloxane phase after Soxhlet extraction with DCM.

(d) GC-ECD (IB) on the 50 % octadecyl (C-18) methylpolysiloxane phase; same extracts analyzed as in GC-ECD (IA).

(e) GC-ECD (IIA) on 5 % phenyl-substituted methylpolysiloxane phase after Soxhlet extraction with DCM.

(f) GC-ECD (IIB) on the 50 % octadecyl (C-18) methylpolysiloxane phase; same extracts analyzed as in GC-ECD (IIA).

(g) GC/MS (I) on 5 % phenyl-substituted methylpolysiloxane phase after Soxhlet extraction with 50 % hexane/50 % acetone.

(h) GC/MS (II) on 5 % phenyl-substituted methylpolysiloxane phase after PFE extraction with 50 % hexane/50 % acetone.

(i) GC/MS (III) on 5 % phenyl-substituted methylpolysiloxane phase; same extracts anlayzed as in GC-ECD (IIA).

(j) Results from 19 laboratories participating in an interlaboratory comparison exercise.

Table 7. Reference Concentrations for Selected Inorganic Constituents in SRM 1944 as Determined by Multiple Laboratories

NOTE: These concentrations are provided as reference values because either the results have not been confirmed by an independent analytical technique as required for certification, the agreement among results from multiple methods was insufficient for certification, or insufficient analyses have been performed at NIST to confirm the results of the outside laboratories.

Elements	Degrees of Freedom	Mass Fraction in percent (dry-mass basis)(a,b)
Silicon(c,d)	81	31 ± 3
		Mass Fraction in mg/kg (dry-mass basis)(a,b)
Beryllium(c,h)	17	1.6 ± 0.3
Copper(c,d,f)	101	380 ± 40
Mercury(c,i)	18	3.4 ± 0.5
Selenium(c,e,f)	24	1.4 ± 0.2
Silver(c,d,e,g)	8	6.4 ± 1.7
Thallium(c,f)	12	0.59 ± 0.1
Tin(c,f)	22	42 ± 6

(a) The results are expressed as the reference value ± the expanded uncertainty. The reference value is the equally weighted mean of available results from: (1) NIST INAA analyses, (2) two methods performed at NRCC, (3) results from seven selected laboratories participating in the NRCC intercomparison exercise, and (4) results from INAA analyses at IAEA. The expanded uncertainty in the reference value is equal to $U = ku_c$ where u_c is the combined standard uncertainty and k is the coverage factor, both calculated according to the ISO and NIST Guides [24]. The value of u_c is intended to represent at the level of one standard deviation, the uncertainty in the value. Here u_c accounts for both possible method differences, within-method variation, and material inhomogeneity. The coverage factor, k, is the Student's t-value for a 95 % prediction interval with the corresponding degrees of freedom. Because of material inhomogeneity, the variability among the measurements of multiple samples can be expected to be greater than that due to measurement variability alone.

(b) Concentrations reported on dry-mass basis; material as received contains approximately 1.3 % moisture.

(c) Results from five to seven laboratories participating in the NRCC interlaboratory comparison exercise.

(d) Measured at NRCC using GFAAS.

(e) Measured at NIST using INAA.

(f) Measured at NRCC using ID-ICPMS.

(g) Measured at IAEA using INAA.

(h) Measured at NRCC using ICPAES.

(i) Measured at NRCC using CVAAS.

Table 8. Reference Concentrations for Selected Inorganic Constituents in SRM 1944 as Determined by INAA

NOTE: These concentrations are provided as reference values because the results have not been confirmed by an independent analytical technique as required for certification; therefore, unrecognized bias may exist for some analytes in this matrix.

Element	Effective Degrees of Freedom	Mass Fraction in percent (dry-mass basis)(a,b)		
Calcium	21	1.0	±	0.1
Chlorine	21	1.4	±	0.2
Potassium	21	1.6	±	0.2
Sodium	25	1.9	±	0.1
		Mass Fraction in mg/kg (dry-mass basis)(a,b)		
Bromine	10	86	±	10
Cesium	11	3.0	±	0.3
Cobalt	10	14	±	2
Rubidium	14	75	±	2
Scandium	37	10.2	±	0.2
Titanium	21	4300	±	300
Vanadium	21	100	±	9

(a) The results are expressed as the reference value ± the expanded uncertainty. The reference value is based on the results from an INAA study. The associated uncertainty accounts for both random and systematic effects, but because only one method was used, unrecognized bias may exist for some analytes in this matrix. The expanded uncertainty in the reference value is equal to $U = ku_c$, where u_c is the combined standard uncertainty and k is the coverage factor, both calculated according to the ISO and NIST Guides [24]. The value of u_c is intended to represent at the level of one standard deviation, the uncertainty in the value. Here u_c accounts for within-method variation and material inhomogeneity. The coverage factor, k, is the Student's t-value for a 95 % prediction interval with the corresponding degrees of freedom. Because of material inhomogeneity, the variability among the measurements of multiple samples can be expected to be greater than that due to measurement variability alone.

(b) Concentrations reported on dry-mass basis; material as received contains approximately 1.3 % moisture.

Table 9. Reference Concentrations for Selected Dibenzo-*p*-dioxin and Dibenzofuran Congeners in SRM 1944

NOTE: These concentrations are provided as reference values because the results have not been confirmed by an independent analytical technique as required for certification. Although bias has not been evaluated for the procedures used, the reference values should be useful for comparison with results obtained using similar procedures.

Dibenzo-*p*-dioxin and Dibenzofuran Congeners	Mass Fraction in µg/kg (dry-mass basis)(a,b)		
2,3,7,8-Tetrachlorodibenzo-*p*-dioxin	0.133	±	0.009
1,2,3,7,8-Pentachlorodibenzo-*p*-dioxin	0.019	±	0.002
1,2,3,4,7,8-Hexachlorodibenzo-*p*-dioxin	0.026	±	0.003
1,2,3,6,7,8-Hexachlorodibenzo-*p*-dioxin	0.056	±	0.006
1,2,3,7,8,9-Hexachlorodibenzo-*p*-dioxin	0.053	±	0.007
1,2,3,4,6,7,8-Heptachlorodibenzo-*p*-dioxin	0.80	±	0.07
Octachlorodibenzo-*p*-dioxin	5.8	±	0.7
2,3,7,8-Tetrachlorodibenzofuran(c)	0.039	±	0.015(d)
1,2,3,7,8-Pentachlorodibenzofuran	0.045	±	0.007
2,3,4,7,8-Pentachlorodibenzofuran	0.045	±	0.004
1,2,3,4,7,8-Hexachlorodibenzofuran	0.22	±	0.03
1,2,3,6,7,8-Hexachlorodibenzofuran	0.09	±	0.01
2,3,4,6,7,8-Hexachlorodibenzofuran	0.054	±	0.006(e)
1,2,3,7,8,9-Hexachlorodibenzofuran	0.019	±	0.018(f)
1,2,3,4,6,7,8-Heptachlorodibenzofuran	1.0	±	0.1
1,2,3,4,7,8,9-Heptachlorodibenzofuran	0.040	±	0.006(e)
Octachlorodibenzofuran	1.0	±	0.1
Total Toxic Equivalents (TEQ)(g)	0.25	±	0.01
Total Tetrachlorodibenzo-*p*-dioxins	0.25	±	0.05(e)
Total Pentachlorodibenzo-*p*-dioxins	0.19	±	0.06
Total Hexachlorodibenzo-*p*-dioxins	0.63	±	0.09
Total Heptachlorodibenzo-*p*-dioxins	1.8	±	0.2
Total Tetrachlorodibenzofurans	0.7	±	0.2
Total Pentachlorodibenzofurans	0.74	±	0.07
Total Hexachlorodibenzofurans	1.0	±	0.1
Total Heptachlorodibenzofurans	1.5	±	0.1
Total Dibenzo-*p*-dioxins(h)	8.7	±	0.9
Total Dibenzofurans(h)	5.0	±	0.5

(a) Each reference value is the mean of the results from up to 14 laboratories participating in an interlaboratory exercise. The expanded uncertainty in the reference value is equal to $U = ku_c$, where u_c is the combined standard uncertainty calculated according to the ISO and NIST Guides [24] and k is the coverage factor. The value of u_c is intended to represent at the level of one standard deviation, the combined effect of all the uncertainties in the reference value. Here u_c is the uncertainty in the mean arising from the variation among the laboratory results. The degrees of freedom is equal to the number of available results minus one (13 unless noted otherwise). The coverage factor, k, is the value from a student's t-distribution for a 95 % confidence interval.

(b) Concentrations reported on dry-mass basis; material as received contains approximately 1.3 % moisture.

(c) Confirmation results using a 50 % cyanopropyl phenyl polysiloxane or 90 % *bis*-cyanopropyl 10 % cyanopropylphenyl polysiloxane phase columns.

(d) Degrees of freedom = 7 for this compound.

(e) Degrees of freedom = 12 for this compound.

(f) Degrees of freedom = 9 for this compound.

(g) TEQ is the sum of the products of each of the 2,3,7,8-substituted congeners multiplied by their individual toxic equivalency factors (TEFs) recommended by the North Atlantic Treaty Organization (NATO) [25]. With regard to 2,3,7,8-tetrachlorodibenzofuran, the results of the confirmation column were used when available to calculate the TEQ.

(h) Total of tetra- through octachlorinated congeners.

Table 10. Reference Values for Particle-Size Characteristics for SRM 1944

NOTE: These results are provided as reference values because the results are method specific as defined by the procedures described in the Preparation and Analysis section. Although bias has not been evaluated for the procedures used, the reference values should be useful for comparison with results obtained using similar procedures.

Particle Measurement	Value[a]
Mean diameter (volume distribution, MV, µm)[b]	151.2 ± 0.4
Mean diameter (area distribution, µm)[c]	120.4 ± 0.1
Mean diameter (number distribution, µm)[d]	75.7 ± 0.3
Surface Area (m^2/cm^3)[e]	0.050 ± 0.013

[a] The reference value is the mean value of measurements from the analysis of subsamples from four bottles. Each uncertainty, computed according to the CIPM approach as described in the ISO and NIST Guides [24], is an expanded uncertainty at the 95 % level of confidence, which includes random sources of uncertainty. The expanded uncertainty defines a range of values for the reference value within which the true value is believed to lie, at a level of confidence of 95 %.

[b] The mean diameter of the volume distribution represents the center of gravity of the distribution and compensates for scattering efficiency and refractive index. This parameter is strongly influenced by coarse particles.

[c] The mean diameter of the area distribution, calculated from the volume distribution with less weighting by the presence of coarse particles than MV.

[d] The mean diameter of the number distribution, calculated using the volume distribution weighted to small particles.

[e] Calculated specific surface area assuming solid, spherical particles. This is a computation and should not be interchanged with an adsorption method of surface area determination as this value does not reflect porosity or topographical characteristics.

The following data show the percent of the volume that is smaller than the indicated size:

Percentile	Particle Diameter (µm)[a]
95	296 ± 5
90	247 ± 2
80	201 ± 1
70	174 ± 1
60	152 ± 1
50[b]	135 ± 1
40	120 ± 1
30	106 ± 1
20	91 ± 1
10	74 ± 1

[a] The reference value for particle diameter is the mean value of measurements from the analysis of subsamples from four bottles. Each uncertainty, computed according to the CIPM approach as described in the ISO and NIST Guides [24], is an expanded uncertainty at the 95 % level of confidence, which includes random sources of uncertainty. The expanded uncertainty defines a range of values for the reference value within which the true value is believed to lie, at a level of confidence of 95 %.

[b] Median diameter (50 % of the volume is less than 135 µm).

Table 11. Reference Values for Total Organic Carbon and Percent Extractable Mass in SRM 1944

NOTE: These results are provided as reference values because the results are method specific as defined by the procedures described in the Preparation and Analysis section. Although bias has not been evaluated for the procedures used, the reference values should be useful for comparison with results obtained using similar procedures.

Total Organic Carbon (TOC)	4.4 % ± 0.3 % mass fraction[a,b]
Extractable Mass[c]	1.15 % ± 0.04 % mass fraction[a,d]

[a] Concentration is reported on a dry-mass basis; material as received contains approximately 1.3% moisture.

[b] The reference value for total organic carbon is an equally weighted mean value from routine measurements made by three laboratories. Each uncertainty, computed according to the CIPM approach as described in the ISO and NIST Guides [24], is an expanded uncertainty at the 95 % level of confidence, which includes random sources of uncertainty. The expanded uncertainty defines a range of values for the reference value within which the true value is believed to lie, at a level of confidence of 95 %.

[c] Extractable mass as determined from Soxhlet extraction using DCM.

[d] The reference value for extractable mass is the mean value of six measurements. Each uncertainty, computed according to the CIPM approach as described in the ISO and NIST Guides [24], is an expanded uncertainty at the 95 % level of confidence, which includes random sources of uncertainty. The expanded uncertainty defines a range of values for the reference value within which the true value is believed to lie, at a level of confidence of 95 %.

Table 12. Information Values for Concentrations for Selected Inorganic Constituents in SRM 1944 as Determined by INAA

NOTE: These results are provided as information values only because insufficient information is available to assess adequately the uncertainty associated with the value or only a limited number of analyses were performed.

Elements	Mass Fractions in percent (dry-mass basis)[a]
Magnesium[b]	1.0
	Mass Fractions in mg/kg (dry-mass basis)[a]
Antimony[b,c]	5
Cerium[c]	65
Europium[c]	1.3
Gold[c]	0.10
Lanthanum[c]	39
Thorium[c]	13
Uranium[c]	3.1

[a] Concentration is reported on a dry-mass basis; material as received contains approximately 1.3 % moisture.

[b] Measured at NIST using INAA.

[c] Measured at IAEA using INAA.

Table 13. Analytical Methods Used for the Analysis of SRM 1944 for Inorganic Constituents

Elements	**Analytical Methods**
Aluminum	FAAS, ICPAES, INAA, XRF
Antimony	GFAAS, HGAAS, ICP-MS, ID-ICPMS, INAA
Arsenic	GFAAS, HGAAS, ICPMS, INAA, XRF
Beryllium	GFAAS, ICP-AES, ICPMS
Bromine	INAA
Cadmium	FAAS, GFAAS, ICPMS, ID-ICPMS
Calcium	INAA
Cerium	INAA
Cesium	INAA
Chlorine	INAA
Chromium	FAAS, GFAAS, ICPMS, ID-ICPMS, INAA, XRF
Cobalt	INAA
Copper	FAAS, GFAAS, ICPAES, ICPMS, ID-ICPMS, XRF
Europium	INAA
Gold	INAA
Iron	FAAS, ICPAES, ICPMS, ID-ICPMS, INAA, XRF
Lanthanum	INAA
Lead	FAAS, GFAAS, ICPMS, ID-ICPMS, XRF
Magnesium	INAA
Manganese	FAAS, ICPAES, ICPMS, INAA, XRF
Mercury	CVAAS, ICPMS
Nickel	GFAAS, ICPAES, ICPMS, ID-ICPMS, INAA, XRF
Potassium	INAA
Rubidium	INAA
Scandium	INAA
Selenium	GFAAS, HGAAS, ICPMS, INAA
Silicon	FAAS, ICPAES, XRF
Silver	FAAS, GFAAS, ICPMS, INAA
Sodium	INAA
Thallium	GFAAS, ICPAES, ICPMS, ID-ICPMS
Thorium	INAA
Tin	GFAAS, ICPMS, ID-ICPMS
Titanium	INAA
Uranium	INAA
Vanadium	INAA
Zinc	FAAS, ICPAES, ICPMS, ID-ICPMS, XRF, INAA

Methods

CVAAS	Cold vapor atomic absorption spectrometry
FAAS	Flame atomic absorption spectrometry
GFAAS	Graphite furnace atomic absorption spectrometry
HGAAS	Hydride generation atomic absorption spectrometry
ICPAES	Inductively coupled plasma atomic emission spectrometry
ICPMS	Inductively coupled plasma mass spectrometry
ID-ICPMS	Isotope dilution inductively coupled plasma mass spectrometry
INAA	Instrumental neutron activation analysis
XRF	X-ray fluorescence spectrometry

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Certificate Revision History: 22 December 2008 (Extension of certification period); 14 May 1999 (Original certificate date).

Users of this SRM should ensure that the certificate in their possession is current. This can be accomplished by contacting the SRM Program at: telephone (301) 975-2200; fax (301) 926-4751; e-mail srminfo@nist.gov; or via the Internet at http://www.nist.gov/srm.

APPENDIX A

The analysts and laboratories listed below participated in the interlaboratory comparison exercise for the determination of polychlorinated dibenzo-*p*-dioxins and dibenzofurans in SRM 1944.

W.J. Luksemburg, Alta Analytical Laboratory, Inc., El Dorado Hills, CA
L. Phillips, Axys Analytical Services Ltd., Sidney, British Columbia, Canada
M.J. Armbruster, Battelle Columbus Laboratories, Columbus, OH
G. Reuel, Canviro Analytical Laboratories Ltd., Waterloo, Ontario, Canada
C. Brochu, Environment Québec, Laval, Québec, Canada
G. Poole, Environment Canada Environmental Technology Centre, Ottawa, Ontario, Canada
B. Henkelmann, GSF National Research Center for Environment and Health, Neuherberg, Germany
R. Anderson, Institute of Environmental Chemistry, Umeå University, Umeå, Sweden
C. Lastoria, Maxxam Analytics, Inc., Mississauga, Ontario, Canada
E. Reiner, Ontario Ministry of Environment and Energy, Etobicoke, Ontario, Canada
J. Macaulay, Research and Productivity Council, Fredericton, New Brunswick, Canada
T.L. Wade, GERG, Texas A&M University, College Station, TX
C. Tashiro, Wellington Laboratories, Guelph, Ontario, Canada
T.O. Tiernan, Wright State University, Dayton, OH

APPENDIX B

The analysts and laboratories listed below participated in the interlaboratory comparison exercise for the determination of trace elements in SRM 1944.

A. Abbgy, Applied Marine Research Laboratory, Old Dominion University, Norfolk, VA
A. Scott, Australian Government Analytical Laboratories, Pymble, Australia
H. Mawhinney, Animal Research Institute, Queensland Department of Primary Industries, Queensland, Australia
E. Crecelius, Battelle Pacific Northwest, Sequim, WA
M. Stephenson, California Department of Fish and Game, Moss Landing, CA
B. Presley, Department of Oceanography, Texas A&M University, College Station, TX
K. Elrick, U.S. Geological Survey, Atlanta, GA

DEPARTMENT OF COMMERCE • UNITED STATES OF AMERICA

National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 2702

Inorganics in Marine Sediment

This Standard Reference Material (SRM) is marine sediment collected at the mouth of the Baltimore Harbor, Baltimore, MD and is intended for use in evaluating analytical methods for the determination of selected elements in marine or fresh water sediment and similar matrices. All of the constituents in SRM 2702, for which certified, reference, and information values are provided, were naturally present in the sediment material before processing. A unit of SRM 2702 consists of a bottle containing 50 g of radiation-sterilized, freeze-dried sediment material.

Certified Concentration Values: Certified values for concentrations, expressed as mass fractions, for 25 elements are provided in Table 1. The certified values are based on the agreement of results from two or more chemically independent analytical techniques obtained at NIST and collaborating laboratories [1]. A NIST certified value is a value for which NIST has the highest confidence in its accuracy in that all known or suspected sources of bias have been investigated or taken into accounted.

Reference Concentration Values: Reference values for concentrations, expressed as mass fractions, are provided for eight additional elements in Table 2. Reference values are non-certified values that are the best estimate of the true value. However, the values do not meet the NIST criteria for certification and are provided with associated uncertainties that may reflect only measurement precision, may not include all sources of uncertainty, or may reflect a lack of sufficient statistical agreement among multiple analytical methods [1].

Information Concentration Values: Information values for concentrations, expressed as mass fractions, are provided in Table 3 for 11 elements. An information value is considered to be a value that will be of interest and use to the SRM user, but for which insufficient information is available to assess adequately the uncertainty associated with the value, or only a limited number of analyses were performed [1].

Expiration of Certification: The certification of **SRM 2702** is valid, within the measurement uncertainty specified, until **01 October 2022**, provided the SRM is handled and stored in accordance with the instructions given in this certificate (see "Instructions for Handling, Storage and Use"). However, the certification is nullified if the SRM is damaged, contaminated, or otherwise modified.

Maintenance of SRM Certification: NIST will monitor this SRM over the period of its certification. If substantive technical changes occur that affect the certification before the expiration of this certificate, NIST will notify the purchaser. Registration (see attached sheet) will facilitate notification.

The coordination of the technical measurements leading to the certification of this SRM was performed by R.L. Zeisler of the NIST Analytical Chemistry Division.

The preparation and collection of the sediment material was completed by M.P. Cronise and C.N. Fales of the NIST Measurement Services Division and B.J. Porter and M.M. Schantz of the NIST Analytical Chemistry Division. Assistance in collecting the sediment material was provided by G.G. Lauenstein, J. Collier, and J. Lewis of the National Oceanic and Atmospheric Administration (NOAA).

Statistical analysis was provided by M. Vangel and W.S. Liggett, Jr. formerly of the NIST Statistical Engineering Division.

Stephen A. Wise, Chief
Analytical Chemistry Division

Gaithersburg, MD 20899
Certificate Issue Date: 16 July 2012
Certificate Revision History on Last Page

Robert L. Watters, Jr., Chief
Measurement Services Division

Analytical measurements at NIST were performed by C.M. Beck II, T.A. Butler, R. Demiralp Oflaz, M.E. Howard, R.M. Lindstrom, S.E. Long, E.A. Mackey, J.L. Mann, K.E. Murphy, R.L. Paul, J.R. Sieber, R.D. Vocke, L.J. Wood, L.L. Yu, and R.L. Zeisler of the NIST Analytical Chemistry Division.

Certification of SRM 2702 was done in collaboration with S. Willie, J.W.H. Lam, C. Scriver, and L. Yang of the National Research Council Canada, Institute for National Measurement Standards, Chemical Metrology and S.A. Wilson, M. Adams, P.H. Briggs, P. Lamothe, D. Siems, and J. Taggart of the U.S. Geological Survey, Branch of Geochemistry, Denver, CO.

Support aspects involved in the issuance of this SRM were coordinated through the NIST Measurement Services Division.

INSTRUCTIONS FOR HANDLING, STORAGE AND USE

Handling: This material is a naturally occurring marine sediment from an urban area and may contain constituents of unknown toxicity; therefore, caution and care should be exercised during its handling and use.

Storage: SRM 2702 must be stored in its original bottle at temperatures less than 30 °C away from direct sunlight.

Use: Prior to removal of subsamples for analysis, the contents of the bottle should be mixed. The concentrations of constituents in SRM 2702 are reported on a dry-mass basis. The SRM, as received, contains approximately 2.3 % moisture. The sediment sample should be dried to a constant mass before weighing for analysis or a separate subsample of the sediment should be removed from the bottle at the time of analysis and dried to determine the concentration on a dry-mass basis. If the constituents of interest are volatile, then the moisture must be determined with a separate subsample. The drying procedures described below, or equivalent, are recommended; the temperature of 90 °C shall **NOT** be exceeded.

PREPARATION AND ANALYSIS(1)

Sample Collection and Preparation: The sediment used to prepare this SRM was collected from the Chesapeake Bay at the mouth of the Baltimore Harbor, Baltimore, MD near the Francis Scott Key Bridge (39°12.3'N and 76°31.4'W). This location is near the site where SRM 1941 and SRM 1941a were collected. The sediment was collected using a Kynar-coated modified Van Veen-type grab sampler. A total of approximately 3300 kg of wet sediment was collected from the site. The sediment was freeze-dried, sieved at 70 µm (100 % passing), homogenized in a cone blender, radiation sterilized at 33 kGy to 45 kGy (^{60}Co) dose, and then packaged in screw-capped amber glass bottles each containing approximately 50 g.

Conversion to Dry-Mass Basis: The results for the constituents in SRM 2702 are reported on a dry-mass basis; however, the material "as received" contains residual moisture. The amount of moisture in SRM 2702 was determined by measuring the mass loss after freeze drying subsamples of 1.1 g to 1.3 g for four days at 1 Pa with a –10 °C shelf temperature and a –50 °C condenser temperature. The moisture content in SRM 2702 was 2.39 % ± 0.08 % (95 % confidence level). Alternately, drying in a conventional oven was studied. One gram portions were dried at 90 °C for 12 h and 18 h; a stable weight was achieved after 18 h. The measured moisture content was 2.27 % ± 0.09 % (95 % confidence level).

Homogeneity Assessment: The homogeneity of SRM 2702 was assessed by analyzing duplicate samples of approximately 0.1 g from fifteen bottles selected by stratified random sampling. Results from direct determinations by instrumental neutron activation analysis (INAA) and energy-dispersive X-ray fluorescence analysis (ED-XRF) showed observed standard deviations for all reported elements within the precision of the analytical techniques. No statistically significant differences among bottles were observed for the elements at this sample size.

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(1) Certain commercial equipment, instruments or materials are identified in this certificate to adequately specify the experimental procedure. Such identification does not imply recommendation or endorsement by the National Institute of Standards and Technology, nor does it imply that the materials or equipment identified are necessarily the best available for the purpose.

Analytical Approach: NIST has a number of analytical techniques available for value assignment. The ones applied to this SRM include dissolution based techniques such as thermal or inductively coupled plasma isotope dilution mass spectrometry (TID-MS or ICP-IDMS) and direct analysis techniques such as INAA, neutron capture prompt gamma activation analysis (PGAA), and radiochemical neutron activation analysis (RNAA). Several of these techniques were also used by collaborating laboratories and complemented by additional dissolution techniques such as atomic absorption spectrometry (AAS), inductively coupled plasma atomic emission spectrometry (ICP-AES) and mass spectrometry (ICP-MS) and direct analysis techniques such as wavelength dispersive X-ray fluorescence (WD-XRF) and ED-XRF. Care has been taken to obtain results from both chemically destructive techniques and non-destructive or direct analysis techniques.

Certified Values and Uncertainties: Certified values, derived from the results of several analytical methods performed by NIST and collaborating laboratories and whose combinations differ from element to element, are provided in Table 1. A complete description of the modes of value assignment is given in reference 1. For consistency across elements, a Bayesian statistical model was chosen for computation of certified values and uncertainties [2]. The uncertainty listed with each value is an expanded uncertainty, with coverage factor 2 (approximately 95 % confidence). The reporting follows the ISO Guide [3].

For each element, there is a NIST result with an uncertainty that is complete in coverage of recognized sources of uncertainties, complemented by results from collaborating laboratories with similarly complete uncertainties, and usually several results without complete uncertainties. The uncertainties of the latter results were augmented on the basis of the differences among the results obtained by different methods [4]. The Bayesian methods combine results by different methods from different laboratories according to the results provided and the uncertainties provided with them. Thus, a consistent methodology was used for all elements.

Table 1. Certified Concentrations for Selected Elements

	Mass Fraction mg/kg (unless noted as %)				Mass Fraction mg/kg (unless noted as %)		
Al(a,b,c,d)	8.41 %	±	0.22 %	Ni(a,b,e,f,g,h)	75.4	±	1.5
As(a,b,d,e)	45.3	±	1.8	P(a,b,d,j)	0.1552 %	±	0.0066 %
Ba(a,f,g)	397.4	±	3.2	Pb(,a,b,f,g,h)	132.8	±	1.1
Ce(a,b,d,g)	123.4	±	5.8	Rb(a,d,g)	127.7	±	8.8
Cd(a,e,f,h,i)	0.817	±	0.011	Sb(a,d,g,h)	5.60	±	0.24
Co(a,b,d)	27.76	±	0.58	Sc(a,b,d)	25.9	±	1.1
Cr(a,b,d,g,h)	352	±	22	Sr(a,b,f,g)	119.7	±	3.0
Fe(a,b,c,d)	7.91 %	±	0.24 %	Th(a,b,d,g)	20.51	±	0.96
Hg(e,j,k)	0.4474	±	0.0069	Ti(a,b,c,d)	0.884 %	±	0.082 %
K(a,b,c,d)	2.054 %	±	0.072 %	Tl(f,h)	0.8267	±	0.0060
La(a,b,d,g)	73.5	±	4.2	V(a,b,d)	357.6	±	9.2
Mn(a,b,d)	1757	±	58	Zn(a,b,d,g,h)	485.3	±	4.2
Na(a,b,c,d)	0.681 %	±	0.020 %				

(a) Inductively coupled plasma mass spectrometry (ICP-MS)
(b) Inductively coupled plasma atomic emission spectrometry (ICP-AES)
(c) Wavelength dispersive X-ray fluorescence (WD-XRF)
(d) NIST method - Instrumental neutron activation analysis (INAA)
(e) Atomic absorption spectrometry including hydride generation and cold vapor techniques (AAS)
(f) NIST method - Inductively coupled plasma isotope dilution mass spectrometry (ICP-IDMS)
(g) Energy dispersive X-ray fluorescence (ED-XRF)
(h) Inductively coupled plasma isotope dilution mass spectrometry (ICP-IDMS)
(i) Prompt gamma activation analysis (PGAA)
(j) NIST method – Radiochemical neutron activation analysis (RNAA)
(k) NIST method - Cold vapor ICP-IDMS

Reference Values and Uncertainties: Reference values are based on results from one method carried out in several laboratories or from two or more analytical methods without NIST results. The Bayesian methods of combining the results by different methods from different laboratories were applied as above. These results do not fulfill the criteria for certification since a full estimate of method bias or results from NIST methods have not been available. The reporting follows the ISO Guide [3].

Table 2. Reference Values for Concentrations of Selected Elements

	Mass Fraction mg/kg (unless noted as %)				Mass Fraction mg/kg (unless noted as %)		
Ag(a,b)	0.622	±	0.078	Mg(c,d,e)	0.990 %	±	0.074 %
Ca(c,d,e)	0.343 %	±	0.024 %	Mo(c,d,f)	10.8	±	1.6
Cu(a,b,c,d,f)	117.7	±	5.6	Se(c,d,f)	4.95	±	0.46
Ga(c,d,f)	24.3	±	1.9	Sn(a,d,f)	31.6	±	2.4

(a) Inductively coupled plasma isotope dilution mass spectrometry (ICP-IDMS)
(b) Atomic absorption spectrometry including hydride generation and cold vapor techniques (AAS)
(c) Inductively coupled plasma mass spectrometry (ICP-MS)
(d) Inductively coupled plasma atomic emission spectrometry (ICP-AES)
(e) Wavelength dispersive X-ray fluorescence (WD-XRF)
(f) Energy dispersive X-ray fluorescence (ED-XRF)

Information Values: Information values are given in SRM 2702 to assist users in the assays of non-certified elements. Information values are based on results that did not allow complete assessment of all sources of uncertainty, hence, only estimated means without uncertainties are given.

Table 3. Information Values for Selected Elements

	Mass Fraction mg/kg (unless noted as %)		Mass Fraction mg/kg (unless noted as %)
Be(a,b)	3.0	Nb(b,e)	63
C (total) (c)	3.36 %	Nd(d)	56
C (organic)(c)	3.27 %	S(c)	1.5 %
Cs(a,d)	7.1	Sm(d)	10.8
Hf(d)	12.6	U(a,e)	10.4
Li(a,b)	78.2	W(d)	6.2

(a) Inductively coupled plasma mass spectrometry (ICP-MS)
(b) Inductively coupled plasma atomic emission spectrometry (ICP-AES)
(c) Combustion techniques
(d) NIST method - Instrumental neutron activation analysis (INAA)
(e) Energy dispersive X-ray fluorescence (ED-XRF)

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