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Water and soil sample analysis: data report

W12 Milestone 5 report
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Cover Photo

Hydraulic fracturing operations in Central Queensland (photograph by GISERA).

Contents

Abbreviations/Acronyms.....	3
Acknowledgements.....	4
Executive summary	5
1 Introduction.....	6
2 Sample collection.....	6
3 Analytical methods	8
3.1 Inorganic analysis.....	8
3.1.1 Sample collection and preservation	8
3.1.2 Conductivity and pH	8
3.1.3 Alkalinity	8
3.1.4 Total suspended solids	9
3.1.5 Organic carbon	9
3.1.6 Total and dissolved metals in waters	9
3.1.7 Total mercury in waters.....	9
3.1.8 Anions.....	10
3.1.9 Radionuclides.....	10
3.1.10 Total recoverable metals in soils	10
3.2 Organic analysis	10
3.2.1 Sample collection and preservation	10
3.2.2 Organic chemicals.....	11
4 Results.....	12
5 Conclusions.....	12
6 References.....	12
Appendix A: Details of samples collected.....	13
Appendix B: Inorganics data	19
Appendix C: Soils data	19
Appendix D: Organics data	19

List of Tables

Table 1. Summary of the water and soil sampling program.....	7
Table A1. Log of water samples collected during the field study.....	13
Table A2. Log of soils samples collected.....	17

Abbreviations/Acronyms

Abbreviation / Acronym	Description
AEM	Dilute-acid extractable metal
AFS	Atomic fluorescence spectrometry
ANSTO	Australian Nuclear Science and Technology Organisation
CRM	Certified reference material
DO	Dissolved oxygen
DOC	Dissolved organic carbon
Dup	Duplicate
FEP	Fluorinated ethylene propylene
FLPE	Fluorinated high density polyethylene
GISERA	Gas Industry Social and Environmental Research Alliance
MilliQ	High purity deionised water
HF	Hydraulic fracturing
ICP-AES	Inductively coupled plasma atomic emission spectrometry
IPC-MS	Inductively coupled plasma mass spectrometry
LDPE	Low density polyethylene
LOD	Limit of detection
NMI	National Measurement Institute
QA/QC	Quality assurance/quality control
Rep	Replicate
Std. dev	Standard deviation
TOC	Total organic carbon
TRM	Total recoverable metal
TSS	Total suspended solids

Acknowledgements

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The authors thank Origin Energy, Queensland, for their logistical support throughout this project and access to their gas field operations in Central Queensland, and the field operations team at Origin Energy's Reedy Creek gas processing facility. We also gratefully acknowledge assistance from the frac teams at Origin and Schlumberger, Savanna rig crews, the team at the Reedy Creek Wastewater facility, SGS Chinchilla laboratory staff, HSE staff, and teams providing additional assistance with groundwater and soil sampling.

Executive summary

CSIRO is currently undertaking a detailed study of the impacts of hydraulic fracturing (HF) of coal seams on air, soil and water quality at two locations in the Surat Basin, QLD. The air component of the study is being conducted by CSIRO Oceans & Atmosphere and the water/soil component is being conducted by CSIRO Land & Water.

The aims of the water and soil study are as follows:

- (i) To quantify the impacts of HF operations on the concentrations of contaminants in nearby surface waters, groundwater and soils.
- (ii) To assess the concentrations of HF chemicals and geogenic contaminants in flowback and produced waters resulting from CSG HF operations.
- (iii) To assess contaminant concentrations in the collected water and soil samples with relevant Australian water and soil quality guideline values.
- (iv) To conduct a laboratory assessment of various spill scenarios involving spillage of HF fluid and produced waters onto various soils types representative of the Surat Basin.

During the planning phase of the study, two sites were selected in the Surat Basin at Condabri and Combabula. Both gas fields are operated by Origin Energy. A sampling and monitoring plan for waters and soils was subsequently developed (Apte et al., 2017) and a field sampling program executed from July 2017 to April 2018 during which a range of water and soil samples were collected (Apte et al 2018).

Extensive laboratory analyses has been conducted on samples collected during the field campaign. The data have been collated and are presented in full in this report. Details of the analytical methods employed and quality control data are also provided. A comprehensive report which provides detailed interpretation of the field and laboratory data will be published by the end of 2018.

1 Introduction

CSIRO is currently undertaking a detailed study of the impacts of hydraulic fracturing (HF) of coal seams on air, soil and water quality at two locations in the Surat Basin, QLD. The air component of the study is being conducted by CSIRO Oceans & Atmosphere and the water/soil component is being conducted by CSIRO Land & Water.

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The spills study ((iv)) is being conducted as a parallel investigation and is the subject of a separate report (Kookana et al 2018).

2 Sample collection

Field activities, including sample collection, were focussed around two rural sites (Condabri and Combabula) in Central Queensland which were scheduled to undergo hydraulic fracturing operations in the second half of 2017. A summary of the water and soil samples collected is presented in Table 1. Further details of the sampling program and methods employed may be found in the report by Apte et al. (2018). The sample log detailing all water and soil samples collected may be found in Appendix A.

Table 1. Summary of the water and soil sampling program

Sample type	Samples collected	Number of samples taken	Notes
Dogwood Creek	Surface water	10	Samples collected upstream and downstream of the Condabri study area. Five sampling events: 3 during and 2 after HF operations.
Water bores	Groundwater	12	Three registered bores at the Combabula study site sampled on four occasions. The first two sampling events were during HF operations and the last two after operations had ceased.
Hydraulic fracturing	HF fluid samples	46	Frac zone samples (between 8 to 10 per well) used at 5 wells (typically between 8 and 10 zones) were obtained
Stimulation, flow back and production phases	Flushing, produced and flowback waters	76	Six wells were monitored over a period of six months commencing at the start of HF operations. Three wells at the Condabri site: CNN218, CON382, CNN204 and three at the Combabula site: COM313, COM337, COM359R. Well flushing, flowback and produced waters sampled.
Wastewater treatment facility (WTF)	Incoming water	4	Samples taken at the Reedy Creek WTF on 3 occasions over a four month period
Post-treatment	RO-treated water	3	Samples taken at the Reedy Creek WTF on 3 occasions over a four month period
Membrane rejects	Brine	3	Samples taken at the Reedy Creek WTF on 3 occasions over a four month period
TOTAL		154	
Soils	Soil samples from the well pad and adjacent areas	36	Soil cores (0-20, 20-40 and 40-60 cm depth) at Condabri site were collected at 6 points on six well pads after HF activities has ceased. Adjacent to each well pad., paired reference samples were collected. Additional soil samples were collected from each well pad and reference site and archived for potential later analysis (if contamination is detected).

3 Analytical methods

3.1 Inorganic analysis

3.1.1 Sample collection and preservation

Samples for total and dissolved metals were collected into LDPE bottles (Nalgene, Thermo Fisher), which had previously been acid washed by soaking in 10% v/v nitric acid (Merck, Tracepur) for at least 24 hours, followed by rinsing with copious amounts of Milli-Q water. Samples were filtered using acid washed Sartorius filter rigs fitted with 0.45 µm membrane filters. Samples for metals were preserved by the addition of nitric acid to a final concentration of 0.2% v/v.

Samples for total mercury were collected into either FEP or FLPE bottles that had previously been washed by sequential soaking in 2% Decon detergent, filtered and acidified (0.2% v/v nitric acid) seawater, 50% v/v nitric acid and 10% v/v hydrochloric acid. Bottles were rinsed with copious amounts of Milli-Q water between each stage. Bottles had 0.2 M bromine chloride (BrCl) preservative reagent added to achieve a final concentration of 0.5% v/v BrCl.

Samples for pH, conductivity, alkalinity and total suspended solids were collected in LDPE bottles and stored refrigerated at 4°C until analysis.

Total organic carbon samples were collected into pre-cleaned glass vials (Shimadzu) and stored refrigerated until analysis. A separate sub-sample was syringe filtered (0.45 µm) into another glass vial for dissolved organic carbon analysis.

Samples for chloride, sulfate, nitrate, nitrite and phosphate analysis were syringe filtered (0.45 µm) into LDPE bottles and stored frozen. Samples for ammonia analysis were syringe filtered (0.45 µm) into LDPE bottles containing a small quantity of sulfuric acid and stored refrigerated. These samples were shipped to the National Measurement Institute (NMI) for analysis.

Samples for radionuclide analysis were collected into HDPE 5L carboys then transported to the Australian Nuclear Science and Technology Organisation (ANSTO) for the analysis of uranium, thorium and radium, as well as gross alpha and beta activities.

3.1.2 Conductivity and pH

Electrical conductivity and pH were measured on unfiltered samples using an Orion Versa Star Pro meter, with Orion Ross Ultra pH probe and Orion Conductivity Cell (Thermo Fisher). The pH meter was calibrated using pH buffer solutions daily on use.

3.1.3 Alkalinity

Alkalinity was determined on unfiltered samples by titration with dilute sulfuric acid to pH 4.5 using a Hanna HI902 auto titrator, as per APHA method 2320 Alkalinity by titration (APHA 2017).

3.1.4 Total suspended solids

Total suspended solids (TSS) was determined gravimetrically using an in-house method based on the APHA method 2540. In brief, a known volume of well mixed water sample was filtered through a pre-weighed 0.45 µm filter that was then oven dried to constant weight.

3.1.5 Organic carbon

Total and dissolved (<0.45 µm) organic carbon was analysed using a Shimadzu TOC-LCSH Total Organic Carbon Analyser using the procedures recommended by the manufacturer. Prior to analysis, 300 µL of 6 M hydrochloric acid was added to each sample, followed by purging with oxygen gas for 20 minutes to remove inorganic carbon.

3.1.6 Total and dissolved metals in waters

Both filtered and unfiltered water samples underwent an aqua regia microwave digestion based on USEPA method 3015A (US EPA 2007). To a well-mixed, 25 mL volume of sample, 1.25 mL of nitric acid (Merck, Tracepur) and 1.25 mL of hydrochloric acid (Merck, Tracepur) was added. Samples were placed in a microwave (MARS Xpress) and heated to 80°C for 60 minutes, then made up to 30 mL final volume with ultra-pure water. Digested samples were then analysed by ICP-MS (Agilent 8800) and ICP-AES (Varian 730ES). Calibration standards for ICP-MS were prepared by serial dilution of a 68 element certified standard mix (Choice Analytical) into 0.42% v/v nitric acid and 0.42% v/v hydrochloric acid. ICP-AES standards were prepared from certified stocks (Accustandard, USA) into a final acid concentration of 2% v/v nitric acid. Quality control included analysis of certified reference materials, replicate analyses and spike recoveries.

3.1.7 Total mercury in waters

Total mercury in unfiltered water samples was determined by single-stage amalgamation followed by cold-vapour atomic fluorescence spectrometry (AFS) (Liang and Bloom, 1993). A sample volume of up to 80 mL was dispensed into a Pyrex-glass purging vessel and a 0.4 mL aliquot of bromine monochloride (BrCl) (0.2 M) in hydrochloric acid added to allow oxidation of any organic mercury present to inorganic mercury. The mixture was allowed to stand for a minimum of 90 minutes followed by the addition of 100 µL of hydroxylamine solution (3 M) to destroy any residual BrCl. The vessel was connected to a custom-built purge trap system and 0.5 mL stannous chloride (20% m/V) in 20% hydrochloric acid was added to reduce the inorganic mercury to elemental mercury. The elemental mercury was purged from solution in a nitrogen stream (20 minutes purge time) and trapped on a gold-coated glass bead trap. The trap was transferred to a thermal desorption unit interfaced to a Brooks Rand AFS. The trap was connected to a mercury-free helium gas stream and rapidly heated to 320°C. The released mercury was quantified by the AFS. Calibration standards were prepared by dilution of a certified stock (Choice Analytical).

3.1.8 Anions

Dissolved chloride, sulfate, nitrate, nitrite and phosphate were analysed by the National Measurement Institute (NMI) in Sydney using their in-house analytical methods based on APHA methods (4110 B, 4500 NO3I and 4500-P G) NMI is a NATA accredited laboratory.

3.1.9 Radionuclides

Uranium, thorium and radium activities, as well as gross alpha and beta activities were analysed by the Australian Nuclear Science and Technology Organisation (ANSTO, Lucas Heights, Sydney) using their established methods. Gross alpha and beta radioactivities were determined using methods based on ISO 9696 and ISO9697. For uranium, thorium and radium analysis, samples were acid-digested on a hotplate, then processed for manganese dioxide co-precipitation, followed by ion-chromatography (IC) separation. Isolated fractions of uranium and thorium were co-precipitated with cerium fluoride, collected on fine resolution filter papers and then analysed by alpha spectrometry. The eluates from the IC separation were collected for radium isolation by lead sulphate and barium sulphate co-precipitations, collection of barium precipitates on fine resolution filter papers, followed by alpha and gamma spectrometry. ANSTO is the lead organisation in Australia for the measurement of environmental radioactivity.

3.1.10 Total recoverable metals in soils

Total recoverable metals were determined on dried and homogenised (mortar and pestle) soil samples using microwave assisted, reverse aqua regia digestion (based on USEPA 3051A). Homogenised portions (0.5 g) of sample were weighed into acid washed digest tubes, to which 9 mL nitric acid and 3 mL hydrochloric acid was added, then heated in a commercial microwave (MARS Xpress 6, CEM) to 175°C for 12 minutes. Digested samples were then analysed by a combination of ICP-MS and ICP-AES using matrix matched standards. Certified reference materials (ERM-CC018, European Reference Materials; OREAS-25a, Ore Research and Exploration Australia) were included in each digestion batch. Replicate analysis and spike recoveries were carried out on 10% of samples.

3.2 Organic analysis

3.2.1 Sample collection and preservation

Samples were collected in a number of different containers depending on the final analytical procedure. All samples were collected in amber glass containers that had been rinsed with Decon detergent and sequentially washed with deionised, reverse osmosis, MilliQ water, acetone and baked for 2 h at 400°C. Samples collected for analysis of volatile organic chemicals, including trihalomethanes (THMs), benzene, toluene, ethylbenzenes and xylenes (BTEX), total recoverable hydrocarbon (TRH) fraction C₆-C₉, were collected in 2x40 mL pre-acidified vials with a rubber septum lined lid and no headspace. Samples for non-volatile geogenic analytes, including polycyclic aromatic hydrocarbons (PAHs), phenols and TRH fractions >C₉ were collected in a 250 mL amber

glass bottle. Samples collected for analysis of hydraulic fracturing (HF) additives were collected in 500 mL amber glass bottles and acidified to pH <2 through the addition of 0.25 mL concentrated H₂SO₄ to each bottle. All samples were immediately transferred to an ice box containing frozen ice packs following collection and either sent to the NMI in the ice box or prepared for analysis in the laboratory within 48 h of collection.

3.2.2 Organic chemicals

Samples sent to the NMI were analysed for a range of geogenic chemicals, including PAHs, phenols, VOCs, BTEX and THMs (see Appendix C for full list), following modified versions of USEPA methods 8260, 8015 and 8270 (US EPA 1996, 2006, 2007a).

4 Results

The data arising from the chemical analyses may be found in Appendices B, C and D. Quality control data is included in each Appendix.

5 Conclusions

Extensive laboratory analysis has been conducted on samples collected during the field campaign. The data have been collated and are presented in full in this report. A comprehensive report which provides detailed interpretation of the field and laboratory data will be published by the end of 2018.

6 References

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US EPA (2007a) Non-halogenated organics by gas chromatography. Method 8015C Revision 3.

US EPA (1998) Semivolatile organic compounds by gas chromatography/mass spectrometry (GC/MS). Method 8270D Revision 4.

Appendix A: Details of samples collected

Table A1. Log of water samples collected during the field study

Sample Description	Sampling Date
<i>Surface waters</i>	
Dogwood Creek Upstream	27/07/2017
Dogwood Creek Upstream 11 am	3/08/2017
Dogwood creek upstream	18/08/2017
Dogwood Creek Upstream	13/09/2017
Dogwood Creek Upstream	1/11/2017
Dogwood Creek Downstream	27/07/2017
Dogwood Creek Downstream 11:30 am	3/08/2017
Dogwood creek downstream	18/08/2017
Dogwood Creek Downstream	13/09/2017
Dogwood Creek Downstream	1/11/2017
<i>Groundwater</i>	
GW1 (Western Supply Bore)	17/08/2017
GW1 (taken from holding pond)	19/10/2017
GW1	14/12/2017
GW1 duplicate	14/12/2017
GW1	13/02/2018
GW2	17/08/2017
GW2 (COM-LB037 Carisbrooke House Bore) 9:00 am	19/10/2017
GW2 (COM-LB037) 9:45am	14/12/2017
GW2	13/02/2018
GW3 (tank originally thought to be Eastern Supply Bore)	17/08/2017
GW3 9:30 am	19/10/2017
Pine Dam Bore Feb 2018	13/02/2018
<i>Wastewater treatment plant</i>	
WTF In	9/11/2017
WTF In	11/01/2018
WTF In duplicate	11/01/2018
WTF In	7/3/2018
WTF Out	9/11/2017
WTF Out	11/01/2018
WTF Out	7/3/2018
WTF Brine	9/11/2017
WTF Brine	11/01/2018
WTF Brine	7/3/2018

Sample Description	Sampling Date
<i>Hydraulic fracturing fluids</i>	
CNN218 Zones 1 to 8	19/07/2017
CNN204 Zones 1 to 10	12/08/2017
CON382 Zones 1 to 10	26/07/2017
COM313 Zones 1 to 10	10/10/2017
COM337 Zones 1 to 8	12/10/2017
<i>Flowback and Produced Waters</i>	
CNN218a-1 (flowback)	19/07/2017
CNN218a-2 (flowback)	20/07/2017
CNN218a-3 (flowback)	20/07/2017
CNN218a-4 (flowback)	21/07/2017
CNN218 (flowback)	22/07/2017
CNN218 (produced)	1/08/2017
CNN218 (produced)	3/08/2017
CNN218 (produced)	4/08/2017
CNN218a-8 (produced)	8/08/2017
CNN218 (produced)	16/08/2017
CNN218 (produced)	22/08/2017
CNN218 (produced)	14/09/2017
CNN218 (produced)	10/10/2017
CNN218 (produced)	12/12/2017
CNN218 (produced)	9/01/2018
CON382 flush 11am (Flush-1)	26/07/2017
CON382 end of flush 1330 (Flush-2)	26/07/2017
CON382	29/07/2017
CON382	30/07/2017
CON382	31/07/2017
CON382	1/08/2017
CON382	3/08/2017
CON382	4/08/2017
CON382	8/08/2017
CON382	16/08/2017
CON382	22/08/2017
CON382 duplicate	22/08/2017
CON382	14/09/2017
CON382	10/10/2017
CNN204 clean (Flush-1)	12/08/2017
CNN204 dirty (Flush-2)	12/08/2017
CNN204	14/08/2017
CNN204	15/08/2017
CNN204	16/08/2017

Sample Description	Sampling Date
CNN204	22/08/2017
CNN204	29/08/2017
CNN204	5/09/2017
CNN204	13/09/2017
CNN204	10/10/2017
CNN204	15/11/2017
CNN204	12/12/2017
CNN204	9/01/2018
COM313 Flush-1	10/10/2017
COM313 Flush-2	10/10/2017
COM313 Produced Day 1	19/10/2017
COM313 Produced Day 2	20/10/2017
COM313 Produced Day 3	21/10/2017
COM313 Produced Water Week 1	26/10/2017
COM313 Produced Water	31/10/2017
COM313 Produced Water	8/11/2017
COM313 Week 4	14/11/2017
COM313	12/12/2017
COM313	9/01/2018
COM313	12/02/2018
COM313	12/3/2018
COM313 April 2018	4/4/2018
COM313 (duplicate) April 2018	4/4/2018
COM337 Flush-1	12/10/2017
COM337 Flush-2	12/10/2017
COM337 Produced Day 1	20/10/2017
COM337 Produced Day 2	21/10/2017
COM337 Produced Day 3	22/10/2017
COM359R Flush-1	19/10/2017
COM359R Flush-2	19/10/2017
COM359R Produced Water Day 1	24/10/2017
COM359R Produced Water Day 2	25/10/2017
COM359R Produced Water Day 3	26/10/2017
COM359R Produced Water	1/11/2017
COM359R Produced Water	8/11/2017
COM359R Week 4	12/12/2017
COM359	12/12/2017
COM359R	9/01/2018
COM359R	12/02/2018
COM359R March 2018	12/3/2018
COM359R April 2018	4/4/2018

Sample Description	Sampling Date
COM359R (duplicate) April 2018	4/4/2018

Table A2. Log of soils samples collected

	Well	Type	Depth (cm)	Date Collected
1	CNN204	Background	0-20	8/11/2017
2	CNN204	Background	20-40	8/11/2017
3	CNN204	Background	40-60	8/11/2017
4	CNN207	Background	0-20	8/11/2017
5	CNN207	Background	20-40	8/11/2017
6	CNN207	Background	40-60	8/11/2017
7	CNN209	Background	0-20	8/11/2017
8	CNN209	Background	20-40	8/11/2017
9	CNN209	Background	40-60	8/11/2017
10	CNN210	Background	0-20	8/11/2017
11	CNN210	Background	20-40	8/11/2017
12	CNN210	Background	40-60	8/11/2017
13	CNN218	Background	0-20	8/11/2017
14	CNN218	Background	20-40	8/11/2017
15	CNN218	Background	40-60	8/11/2017
16	CNN210	Hand Auger background	0-20	8/11/2017
17	CNN210	Hand Auger background	20-40	8/11/2017
18	CNN210	Hand Auger background	40-60	8/11/2017
19	CNN204	Drill lease	0-20	8/11/2017
20	CNN204	Drill lease	20-40	8/11/2017
21	CNN204	Drill lease	40-60	8/11/2017
22	CNN207	Drill lease	0-20	8/11/2017
23	CNN207	Drill lease	20-40	8/11/2017

	Well	Type	Depth (cm)	Date Collected
24	CNN207	Drill lease	40-60	8/11/2017
25	CNN209	Drill lease	0-20	8/11/2017
26	CNN209	Drill lease	20-40	8/11/2017
27	CNN209	Drill lease	40-60	8/11/2017
28	CNN210	Drill lease	0-20	8/11/2017
29	CNN210	Drill lease	20-40	8/11/2017
30	CNN210	Drill lease	40-60	8/11/2017
31	CNN218	Drill lease	0-20	8/11/2017
32	CNN218	Drill lease	20-40	8/11/2017
33	CNN218	Drill lease	40-60	8/11/2017
34	CON382	Drill lease	0-20	8/11/2017
35	CON382	Drill lease	20-40	8/11/2017
36	CON382	Drill lease	40-60	8/11/2017

Appendix B: Inorganics data

Inorganic analysis data

<https://doi.org/10.25919/5eb34069929d5>

Appendix C: Soils data

Soils data

<https://doi.org/10.25919/5eb34069929d5>

Appendix D: Organics data

Organics data

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