

# A comparison of two liquid scintillation instruments for analysis of highly quenched samples

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## Acknowledgements

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- In particular: Ross Kleinschmidt, Megan Cook, Drew Watson and Michelle Warry



## Overview

- Motivation
- Measurements
- Case Study
- Conclusion

## Motivation – Leaching, why?

- AS 4439.3-1997 Wastes, sediments and contaminated soils, Part 3: preparation of leachates – Bottle leaching procedure
- Queensland's Radiation Safety Regulation 2010 for disposal of abrasive blasting material and mineral substances.
- The limit is 5 Bq/L - based on not exceeding 10 times the applicable concentration within the Australian Drinking Water Guidelines

Screening level of 0.5 Bq/L for both gross alpha and gross beta activity concentrations.

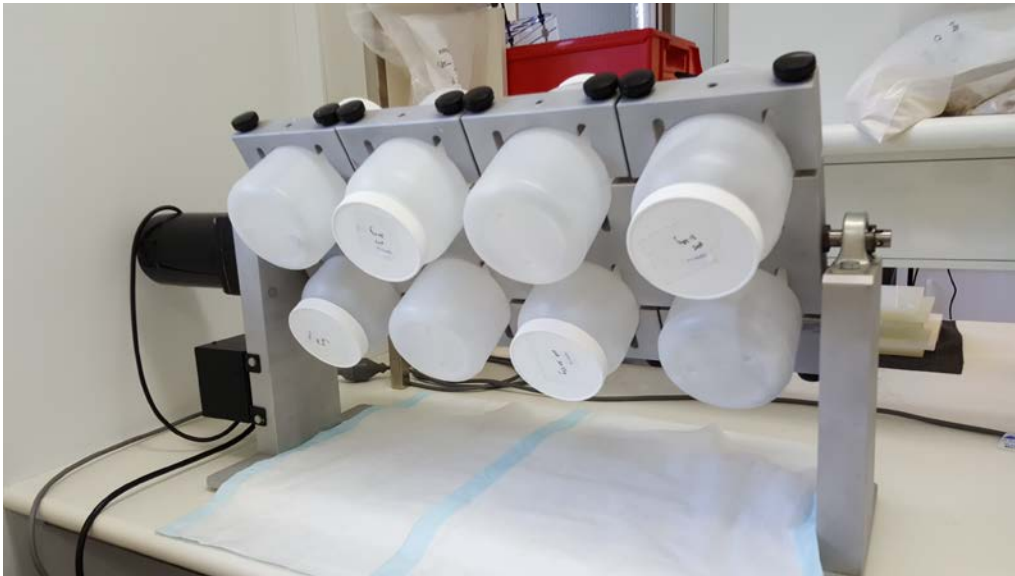


## Toxicity Characteristic Leaching Procedure (TCLP) of radionuclides

- Four leaching solutions
- Samples in duplicate, 10:1 leachate to soil
- Tracer recovery

20 $\mu$ L ~81Bq/L Am241

20 $\mu$ L ~80Bq/L Sr90/Y90



## Methods:

- Soil, leachate solution and tracer (if added), plus one blank per leachate will have rotation times of  $18 \pm 2$ hrs
- Settle overnight
- Direct addition 10mL leachate solution to 12mL Ultima Gold AB in 20mL plastic vials (to match existing routine lab methods)
- Count time 60mins



## Methods cont

- Gamma spec in aluminium jar geometry before leaching
- ICP-MS of each leachate to add to assessment of reliability of results based on what is affecting the quench and self absorption of the sample



## Instruments

- Wallac Quantulus 1220

Alpha/beta discrimination: Pulse shape analysis (PSA)

Quench Indicating Parameter (QIP): Spectral Quench

Parameter of External Standard (SQP(E))

- Hitachi Aloka Accuflex LSC LB7

Alpha/beta discrimination: Rise to Height Converter (RTC) \*This is automatic, can't be changed

QIP: ESCR – External Standard Channel Ratio





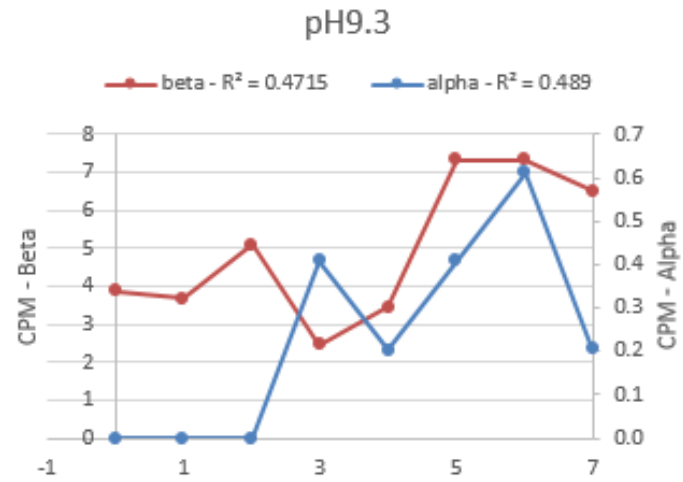
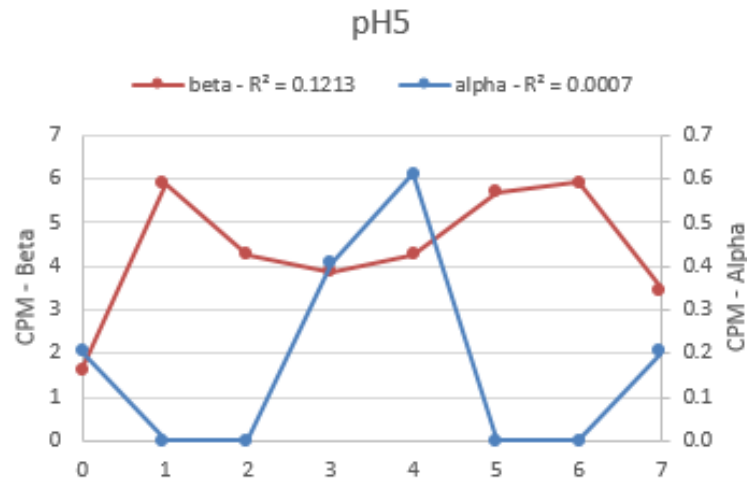
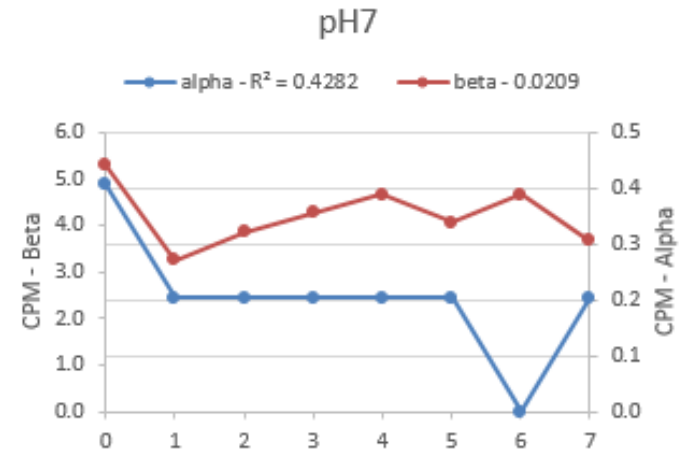
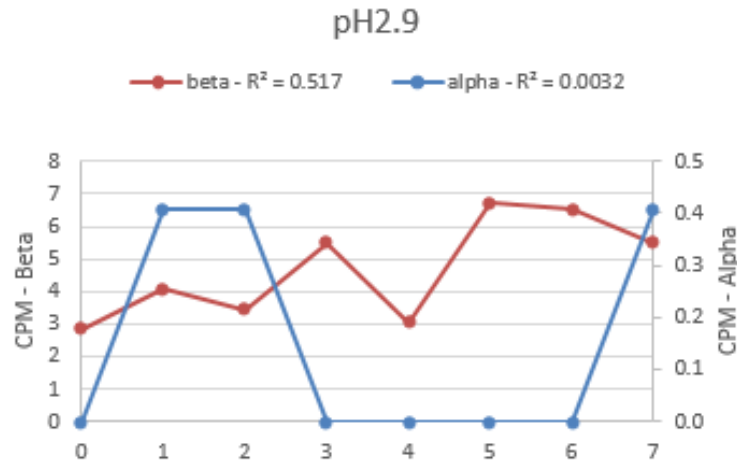
## Aims

- The present study was carried out to determine the correlation of these two QIPs with counting efficiencies

## Measurements

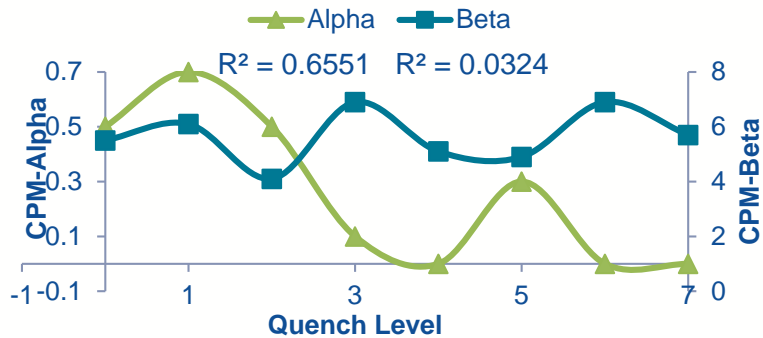
- Standard Instrument Quality parameters:
  - Background counts for each leachate pH on each instrument
  - Quench curves using nitromethane for each leachate pH
  - Alpha/beta discrimination and linearity for each leachate pH using Am241 and Sr90/Y90
- Experimental matrix specific parameters:
  - Samples were prepared in duplicate for each leachate
  - Experimental background, quench curves and linearity using Rhyolite for each leachate pH, spiked with Am241 and Sr90/Y90

# Quantulus background response to increased quench - Nitromethane

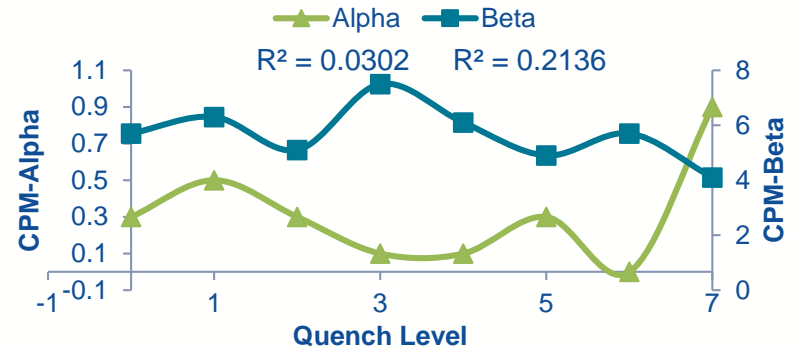


# Aloka background response to increased quench - Nitromethane

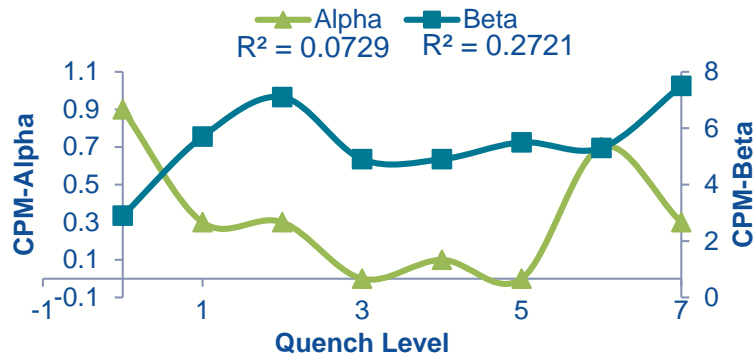
## pH2.9



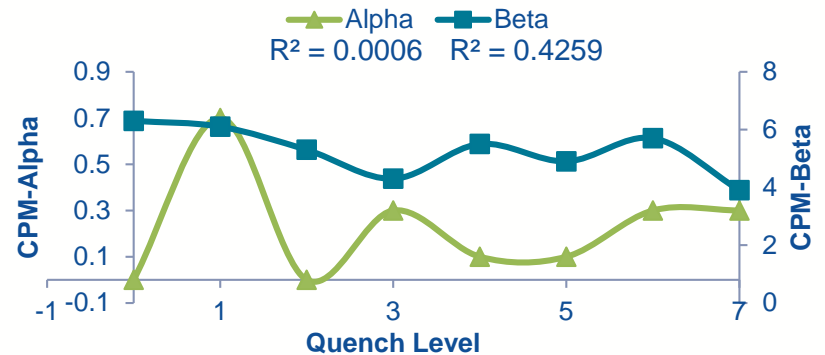
## pH5



## pH7

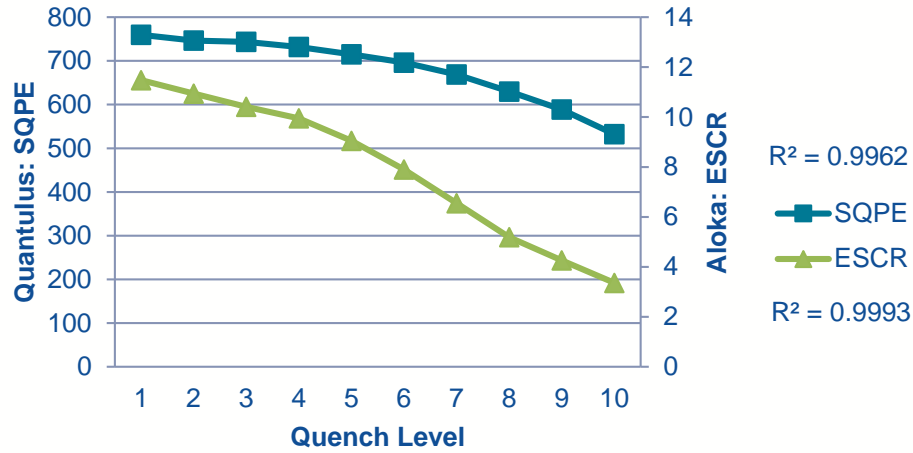


## pH9.3

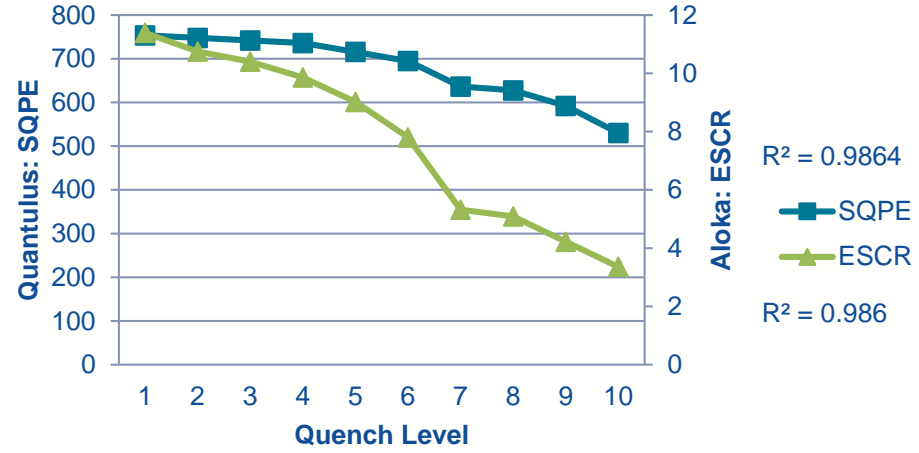


# QIP comparison

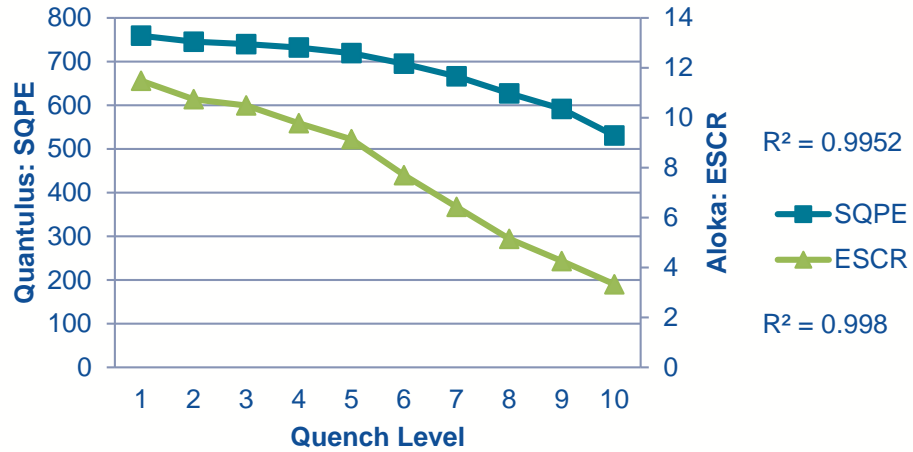
pH2.9



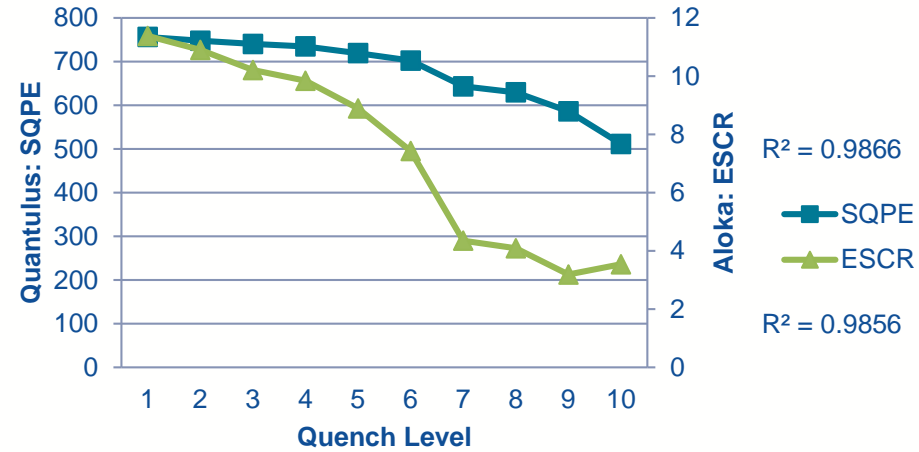
pH7



pH5

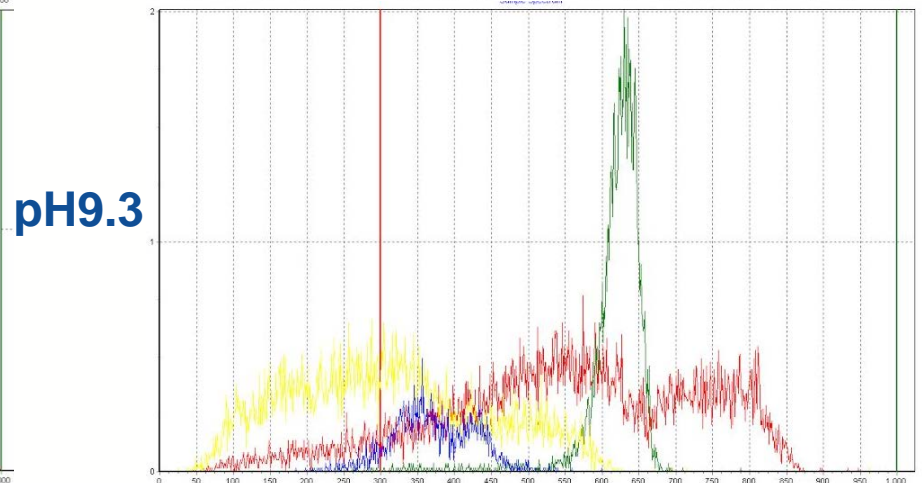
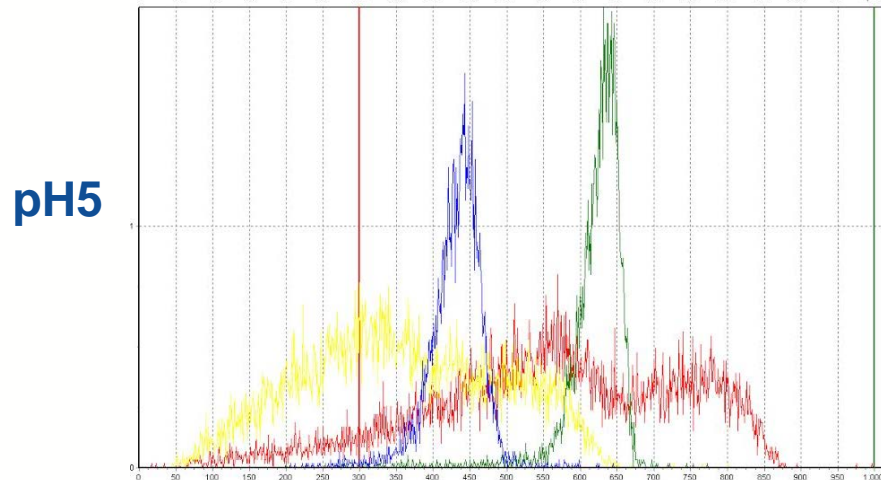
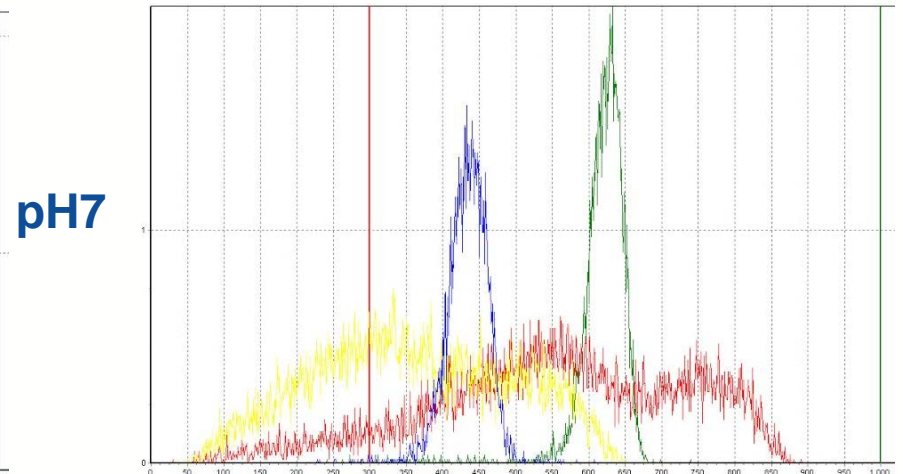
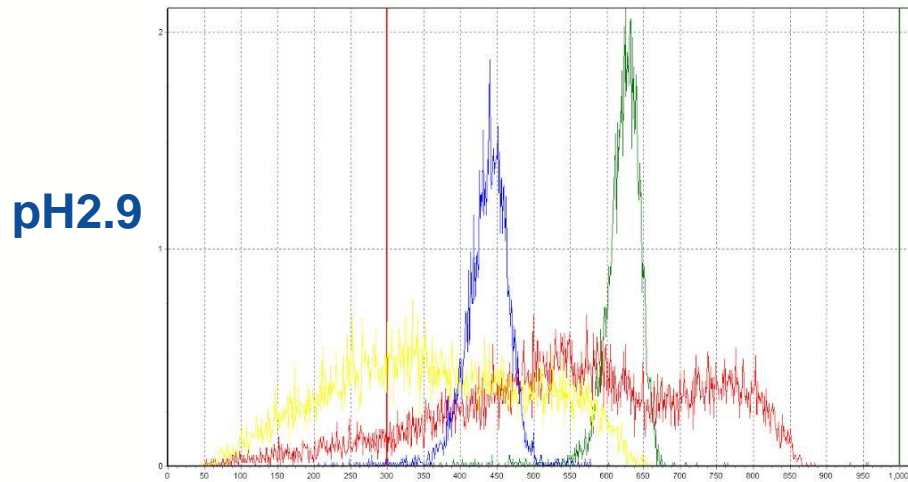


pH9.3



## Spectrum Quench

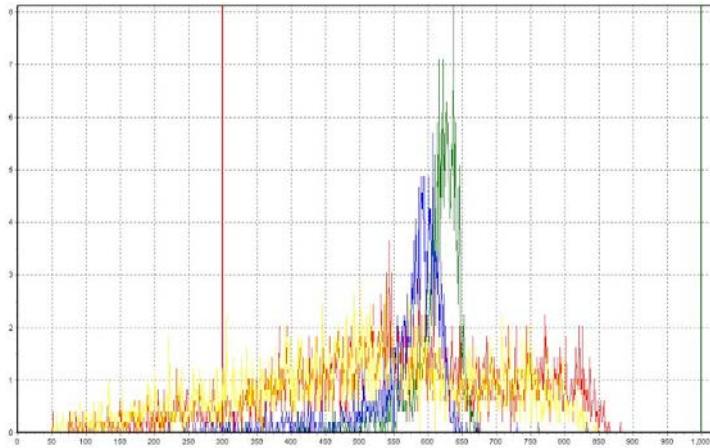
- pH 2.9, 5, 7, 9.3 at 0 $\mu$ L and 230 $\mu$ L Nitromethane, 20 $\mu$ L Am241 ~81Bq/L and 20 $\mu$ L Sr/Y ~Bq/L



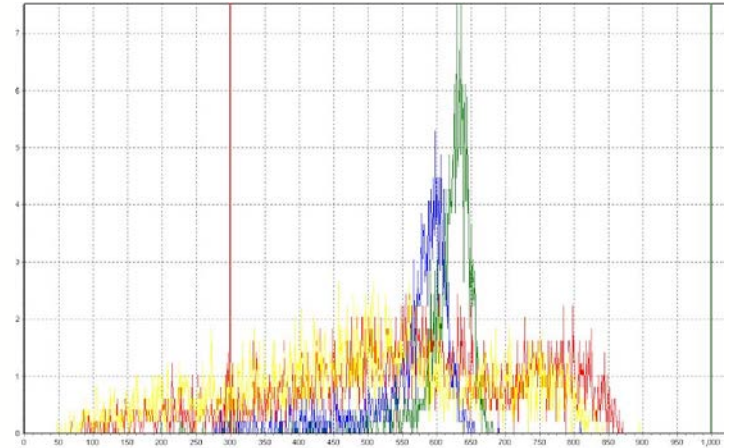
## Experimental Spectrum Quench

- pH 2.9, 5, 7, 9.3 at 0ug and 230 $\mu$ g Rhyolite, 60 $\mu$ L Am241 ~81Bq/L and 60 $\mu$ L Sr/Y ~Bq/L

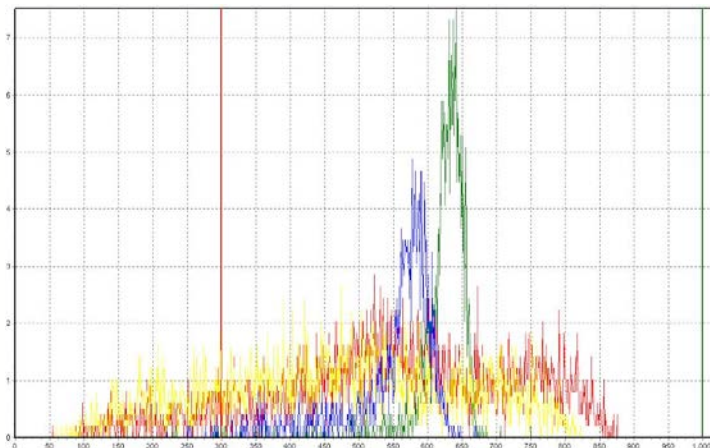
pH2.9



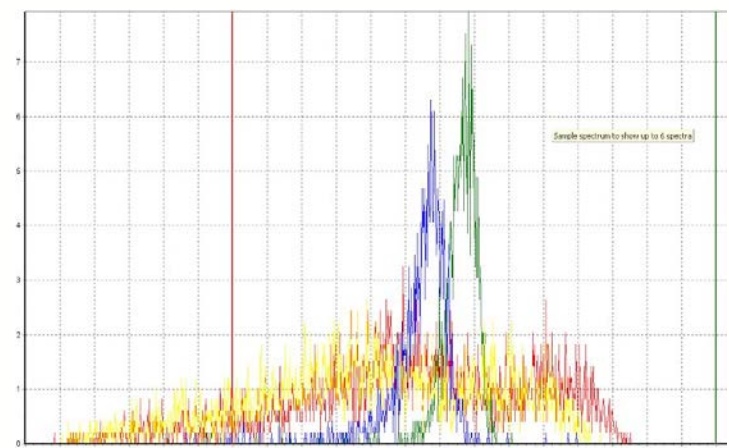
pH7



pH5



pH9.3



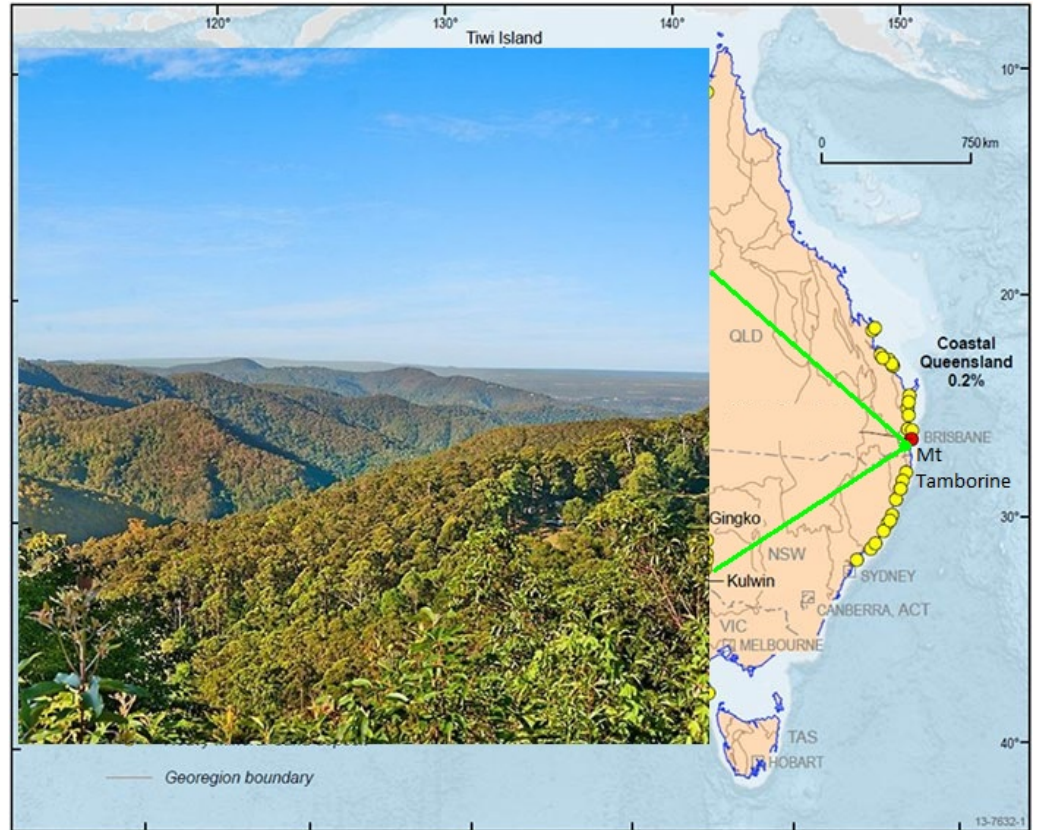
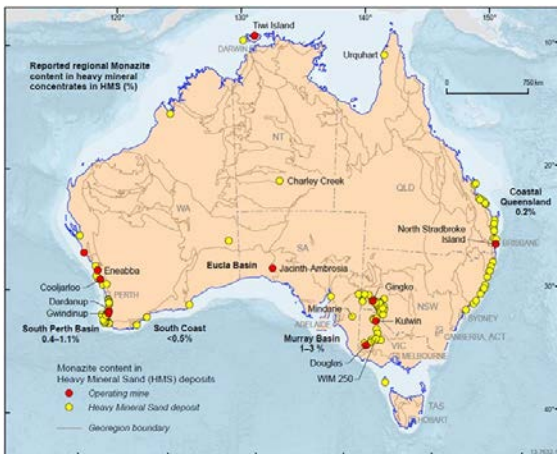
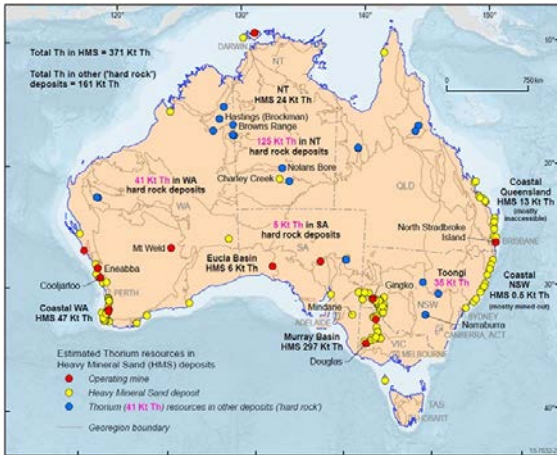
## Cast Study

- TCLP analysis was performed for samples sourced from Mount Tamborine in south east Queensland. Mt Tamborine is a plateau formed from lava after the volcanic eruption of Mount Warning 22 million years ago.
- At solutions of pH 2.9, 5, reagent water, 9.3



# Mount Tambourine, Queensland, Australia

- Rhyolite – igneous rock with elevated uranium and thorium



# ICP-MS of interferences

pH2.9

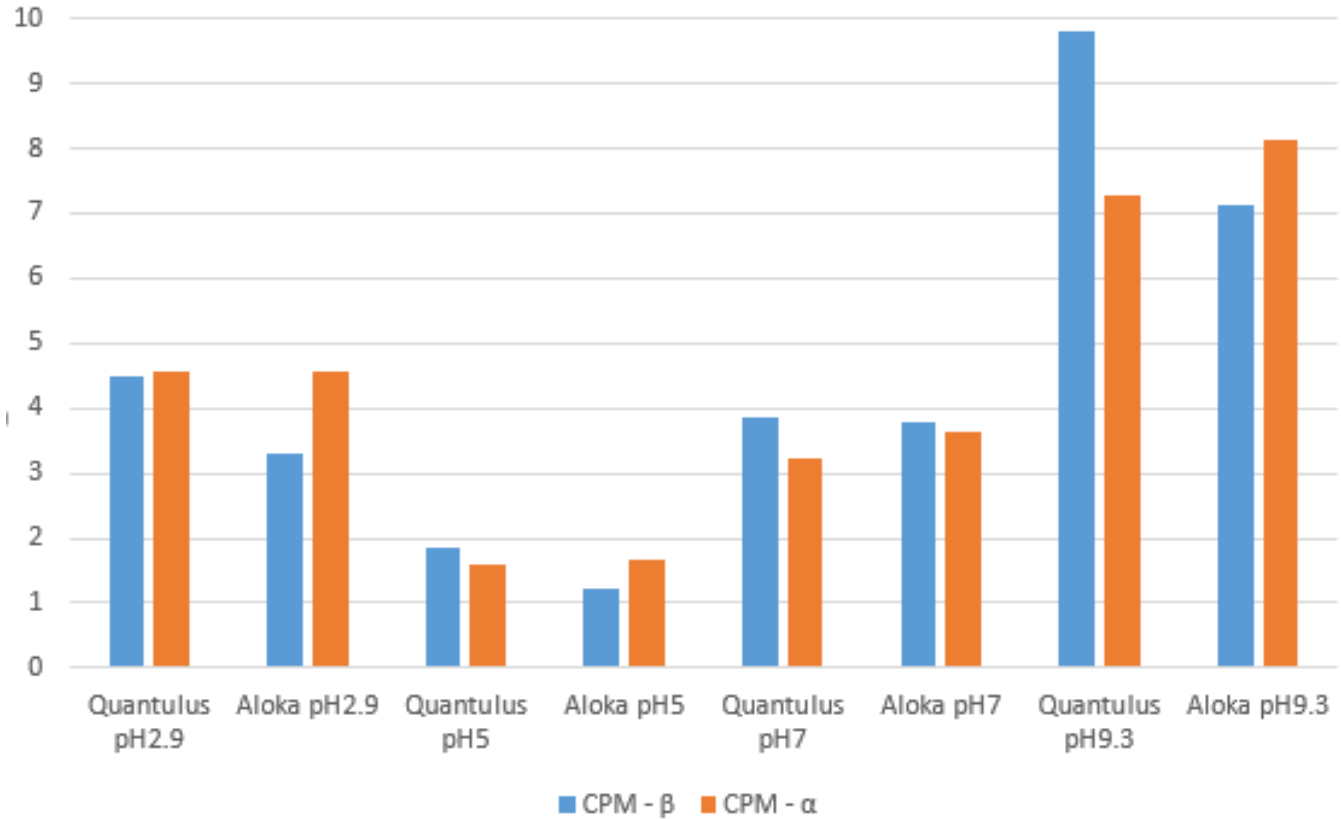
pH5

pH7

pH9.3

Metals by ICP-MS	Units	Reporting Limit	17NA4650	17NA4651	17NA4652	17NA4653
Aluminium	mg/L	0.003	< 0.003	< 0.003	< 0.003	< 0.003
Antimony	mg/L	0.0001	0.48	0.44	0.52	0.95
Arsenic	mg/L	0.0001	0.0009	< 0.0001	< 0.0001	< 0.0001
Barium	mg/L	0.0001	0.0006	< 0.0005	< 0.0005	< 0.0005
Beryllium	mg/L	0.0001	0.077	0.047	0.061	0.065
Cadmium	mg/L	0.0001	< 0.0001	< 0.0001	< 0.0001	< 0.0001
Chromium	mg/L	0.0001	0.0098	0.014	0.0053	0.0065
Cobalt	mg/L	0.0001	0.014	0.0024	0.015	0.026
Copper	mg/L	0.001	0.095	0.085	0.082	0.17
Iron	mg/L	0.005	0.63	0.46	0.51	0.78
Lead	mg/L	0.0001	< 0.0001	< 0.0001	< 0.0001	< 0.0001
Mercury	mg/L	0.0001	0.0034	0.0015	0.0052	0.049
Manganese	mg/L	0.0001	0.058	0.044	0.053	0.099
Molybdenum	mg/L	0.0001	0.0005	0.0004	0.0004	0.0014
Nickel	mg/L	0.0001	0.0002	< 0.0001	< 0.0001	< 0.0001
Selenium	mg/L	0.0001	< 0.0010	< 0.0010	< 0.0010	< 0.0010
Silver	mg/L	0.001	2.7	1.9	4.3	4.5
Strontium	mg/L	0.0001	0.0002	< 0.0001	< 0.0001	< 0.0001
Thallium	mg/L	0.0001	0.0069	0.0032	0.0055	0.011
Tin	mg/L	0.0001	0.0003	0.0002	0.0002	0.0003
Titanium	mg/L	0.001	0.96	0.13	1.0	1.9
Uranium	mg/L	0.0001	< 0.0001	< 0.0001	< 0.0001	< 0.0001
Vanadium	mg/L	0.0001	0.018	0.014	0.015	0.021
Zinc	mg/L	0.001	0.004	< 0.001	0.002	0.002

# GABW LSC Results



## Conclusions

- Quantulus has a lower background for pH2.9 beta, pH5 alpha and beta, pH 7 beta and pH9.3 alpha and beta
- Aloka has a lower background for pH2.9 alpha and pH 7 alpha
- Nitromethane not ideal for quench calibration at pH9.3, soil is better, however will have to investigate salt loading of sample further
- Continuing process of development

**How to calculate uncertainties for a changing matrix in different pH conditions where it is not really possible to calibrate a matrix blank?**

Thank you

