# **UC**

# Low Background Screening Capability in the UK

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Low Radioactivity Techniques 2015 18-20 March, 2015





#### **Rare Event Search Experiment Requirements**

- Background from material radioactivity << signal</li>
   Comprehensive material screening campaign
- Accurate map of contaminants for high precision background model
   Multiple techniques for complete U/Th chains
   Sub-component activities for neutron yields
- 3) Material selection well before construction and installation
   ➤ High throughput, low activity assays, particularly U/Th

## 238U Chain



# 238U Chain

<sup>238</sup> U Early sub-chain	226Ra sub-chain	<sup>210</sup> Pb sub-chain
238U       4. Gyr         a (4.2 MeV)         234Th       24 d         B (0.19 MeV)         Y (63 keV, 3.5%)         Y (93 keV, 4%)         234mPa         1.2 min         B (2.3 MeV)         Y (770 keV, 0.3%)         Y (1000 keV, 0.6%)         234U         0.25 Myr         a (4.8 MeV)	From <sup>239</sup> Th 226Ra 1.6 kyr a (4.8 MeV) y (190 keV, 4% 222Rn 3.8 d a (5.5 MeV) 218Po 3.1 min a (6.1 MeV) 214Pb 28 6 min B (1.0 MeV) y (295 keV, 19 y (352 keV, 36	From <sup>214</sup> Po 210 Pb 22 )r y (47 keV, 4%) 210 Bi y (47 keV, 4%) 210 Bi z (1.2 MeV) 210 Po 138 d z (5.3 MeV) 206 Pb 206 Pb
$\begin{array}{c} \downarrow \qquad \gamma (53 \text{ keV}, 0.2\%) \\ \hline 230\text{Th} \qquad 75 \text{ kyr} \\ \downarrow \qquad \qquad \downarrow \qquad \qquad$	214Bi B (3.2 MeV) γ (609 keV, 47 γ (1.12 MeV, 1 γ (1.76 MeV, 1	Mass Spec., NAA 7%) U/G Ge
10Ra	$\begin{array}{c} 2^{14}\text{Po} & 160 \ \mu\text{s} \\ \downarrow &                                 $	Si PIN alpha spectroscopy



#### **Ultra-low Background Screening in the UK**

- Gamma Spectroscopy
  - Boulby underground laboratory
- Mass Spectrometry
  - Dedicated low-background facility at UCL
- Radon Detection
  - Trace emanation facility at MSSL (UCL)

Technique	Isotopes	Typical Sensitivity Limits	Sample Mass	Destructive/ Non- desctructive	Assay Duration	Notes
HPGe	<ul> <li><sup>238</sup>U, <sup>235</sup>U, <sup>232</sup>Th</li> <li>chains, <sup>40</sup>K, <sup>60</sup>Co,</li> <li><sup>137</sup>Cs (any γ emitter)</li> </ul>	50 ppt U, 100 ppt Th	kg	Non- destructive	Up to 2 weeks	Very versatile, not as sensitive as other techniques, large samples
ICP-MS	<sup>238</sup> U, <sup>235</sup> U and <sup>232</sup> Th (top of chain)	10 <sup>-12</sup> g/g	mg to g	Destructive	Days	Requires sample digestion, preparation critical
Rn Emanation	<sup>222</sup> Rn, <sup>220</sup> Rn	0.1 mBq	kg	Non- destructive	Days to weeks	Large samples, limited by size of emanation



#### **Boulby Underground Laboratory**

#### Long Dark Matter history

- ZEPLIN programme
- Directionality (DRIFT)

#### Major lab upgrade

 Dedicated Low Background Counting Facility





### **Boulby Underground Germanium Suite (BUGS)**

Four detectors in Class 10,000 clean room



- Operations
  - Automated LN2 fills
  - Remote control, env. monitoring
  - Emergency systems, UPS
- Interchangeable Pb+Cu castles
  - Interlocking retractable roof
  - N2 purge fed through Pb/Cu to cavity



**UC** 







#### 0.8 kg ULB BE5030 10 keV threshold

2 kg ULB Ortec GEM-XX-95







1.5 kg ULB SAGe 28 mm X 40 mm well ULB BE2825 0.4 kg pre-screener



# 

#### **Analysis Software**



Select All

#### Default Background Values: Chaloner

	Energy (keV)	Parent	Daughter	Branching Ratio	BackRate (mBq)	Error (mBq)	Background	Fit Sigma	Include Report
26	238.60	Th-232	Pb-212	43.6	0.092	0.009	0	5	
27	727.30	Th-232	Bi-212	6.74			0	5	
28	1620.7	Th-232	Bi-212	1.52			0	5	
29	583.20	Th-232	TI-208	85.1	0.033	0.005	0	5	
30	860.60	Th-232	TI-208	12.5			0	5	
31	2614.5	Th-232	TI-208	99.7	0.016	0.004	0	5	
32	1460.8	K-40	K-40	10.7	0.107	0.009	0	10	
33	1173.2	Co-60	Co-60	99.9	0.039	0.006	0	5	
34	1332.5	Co-60	Co-60	99.9	0.022	0.004	0	5	
35	143.80	U-235	U-235	11.0	0.084	0.008	0	5	
36	185.70	U-235	U-235	57.2			0	5	
37	661.70	Cs-137	Cs-137	85.0			0	5	
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					V Us	se B. Hatio	Subtract Ba	ackground	Greate Repor





#### Simulations

- Modeled detectors 'bottom-up' with engineering drawings
- Calibrated with detector scans, validated with sources
- GEANT4 based simulation integrated into analysis software



#### Efficiency

Isotope	Chaloner Bq/kg	Lunehead Bq/kg	IAEA Recommended
TI-208	9.87 ± 0.13	$10.24 \pm 0.16$	11.6 ± 2.1
Pb-210	39.39 ± 0.44	-	35.5 ± 3.9
Bi-212	33.87 ± 0.77	$34.57 \pm 0.92$	$34.2 \pm 4.0$
Bi-214	17.80 ± 0.40	18.49 ± 0.48	19.6 ± 1.6
Pb-214	20.84 ± 0.13	$21.05 \pm 0.19$	21.6 ± 1.2
Ac-228	29.45 ±0.37	$31.23 \pm 0.53$	31.5 ± 1.4
Th-234	30.73 ± 0.31	-	28.7 ± 5.9
K-40	563.07 ± 2.18	557.69 ± 2.45	611.0 ± 11.0
Cs-137	20.85 ± 0.11	$20.66 \pm 0.13$	21.74 ± 1.78
Isotope	Chaloner Bq/kg	Lunehead Bq/kg	Rhyolite Recommended
Th232	45.00 ± 0.25	43.07 ± 0.08	48.47 ± 1.88
U238 (e)	98.01 ± 2.72	$116.37 \pm 0.96$	117.22 ± 15.21
U238 (I)	97.00 ± 0.61	94.47 ± 0.10	103.68 ± 12.55
K40	870.16 ± 4.47	881.61 ± 0.79	904.94 ± 73.50



#### **Status of BUGS**

- Chaloner and Lunehead installed Sept 2014
- Approximately 50 ppt U/Th sensitivity achieved
- QR code sample tracking system in-place, results fed to LZ database
- LZ material screening initiated
  - Screening schedule integrated into project
  - Live-time requirements informed by Monte Carlo
- Lumpsey and Wilton on-line May/June 2015
- Neutron moderator, enclosures, scintillator installation mid-2015



#### **Mass Spectrometry at UCL**

- New Agilent 7900 ICP-MS mainframe procured exclusively for ultra-low background assays
- Installed and commissioned at UCL Aug 2014
- Standard detection sensitivity at ~ppt for U/Th
- Upgraded Feb 2015
  - HF capability (30%), microflow nebulizer, Pt skimmer/sampling cones
  - Reaction cell (H2) discrimination capability in addition to no gas, and He mode KED
- Presently in HEP lab at UCL, to be moved with sample preparation equipment to dedicated LZ ICP-MS lab (class 10K clean room)







#### **ICP-MS Sample Preparation**

- Milestone EthosUP digestion system
  - > No hot plate; high T/P; reduces digestion to mins.
  - > ensures full recoveries
  - no cross-contamination
- Pyro-260 microwave ashing system (PTFE, acrylics, ...)
- Acid distillation and reflux cleaning systems
- Reproducible, high throughput closed system screening
- Installation in UCL cleanroom April 2015
- Digestions routines under development with Analytix
  - IAEA385 standard (soil)
  - Ti
  - PTFE
  - R11410 PMT components







#### **ICP-MS Sample Preparation**

Microwave digestion (with D. Rowe, Milestons UK Product Manager at Analytix Ltd's facility)

- High pressure reactors constructed from materials transparent to microwaves
- Microwave energy couples directly to ions, rotates around the dipole to cause friction and release heat (hence TFM, etc; low or no dipole moment)
- Acids have higher dipole moments, absorb microwaves readily, for fast and even heating of reactant solutions
- Reaction sped up with HP closed vessel; acids to be heated beyond boiling points
- For PMT components used 220C with mixture of Nitric, Hydrochloric and Hydrofluoric acids to fully dissolve materials
- Optimised ratio and quantity of acid required for complete dissolution of samples
- Each acid has specific purpose during digestion, optimisation required:
  - Nitric acid commonly used to digest any organic material present (CH<sub>2</sub>)<sub>X</sub> + HNO<sub>3</sub> → CO<sub>2</sub>(g) + NO<sub>X</sub>(g) + H<sub>2</sub>O
  - > HCl for Fe-based alloys due to ability to hold high chloro-complex in solution
  - HF acid used for decomposing silicates

#### **ICP-MS Sample Preparation**



Sample	Weight	Acids
1 Quartz Plate	0.200g	4ml HF
3 Kovar Sheet	0.492g	3ml HCl 3ml HF 3ml HNO3
3 Kovar Sheet	0.469g	3ml HCl 3ml HF 3ml HNO3
4 Cobalt free metal sheet	0.491g	3ml HCl 3ml HF 3ml HNO3
4 Cobalt free metal sheet	0.490g	3ml HCl 3ml HF 3ml HNO3
6 Stainless steel sheet (mat surface)	0.475g	3ml HCl 3ml HF 3ml HNO3
6 Stainless steel sheet (mat surface)	0.488g	3ml HCl 3ml HF 3ml HNO3
7 Stainless steel sheet (mat surface)	0.477g	3ml HCl 3ml HF 3ml HNO3
7 Stainless steel sheet (mat surface)	0.461g	3ml HCl 3ml HF 3ml HNO3
8 Quartz insulator	0.1966g	4ml HF
10 Kovar sheet	0.482g	3ml HCl 3ml HF 3ml HNO3
10 Kovar sheet	0.498g	3ml HCl 3ml HF 3ml HNO3



Int T (thermocouple) Int P (transducer) Ext T (infra-red) Microwave power



### Radon Emanation Detector at UCL (MSSL)

...see Xin Ran Liu's talk

- Radon cannot be "fiducialised" away
- Sensitivity at ~0.1 mBq required
- System developed for SuperNEMO
- Silicon PIN diode in 70 litre electro-polished vessel
- Connected to radon concentration line
- Emanation chamber for screening components





#### **Radon Emanation Sensitivity**

- Regular calibration with <sup>226</sup>Ra 'flow-through' source
- High efficiency,  ${}^{214}Po = 31.6 \pm 1.6\%$
- Samples prepared in clean room, UV inspection
- Emanation sensitivity (90% C.L.)

><sup>214</sup>Po < 90 μBq ><sup>218</sup>Po < 120 μBq





#### Summary

#### **Direct Gamma Counting**

- Required for U/Th mid-late chain measurements
  - Boulby Underground Laboratory facility with 4 ULB counters
  - Varied detector types provide range for sample types and sub-chain sensitivity
  - Detectors integrated into LZ screening program

#### **Mass Spectrometry**

- Required for progenitor U/Th measurements and high throughput
  - Dedicated ultra-low background ICP-MS facility
  - Agilent 7900 ICP-MS with HF and H<sub>2</sub> line reaction capability
  - Microwave digestion and ashing closed, clean systems for sample prep
  - LZ construction material screening initiated

#### Radon

- Required for <sup>222</sup>Rn and <sup>220</sup>Rn; backgrounds impervious to self-shielding
  - Radon emanation measurement capability to <90 µBq</li>
  - SuperNEMO demonstrator on-line screening