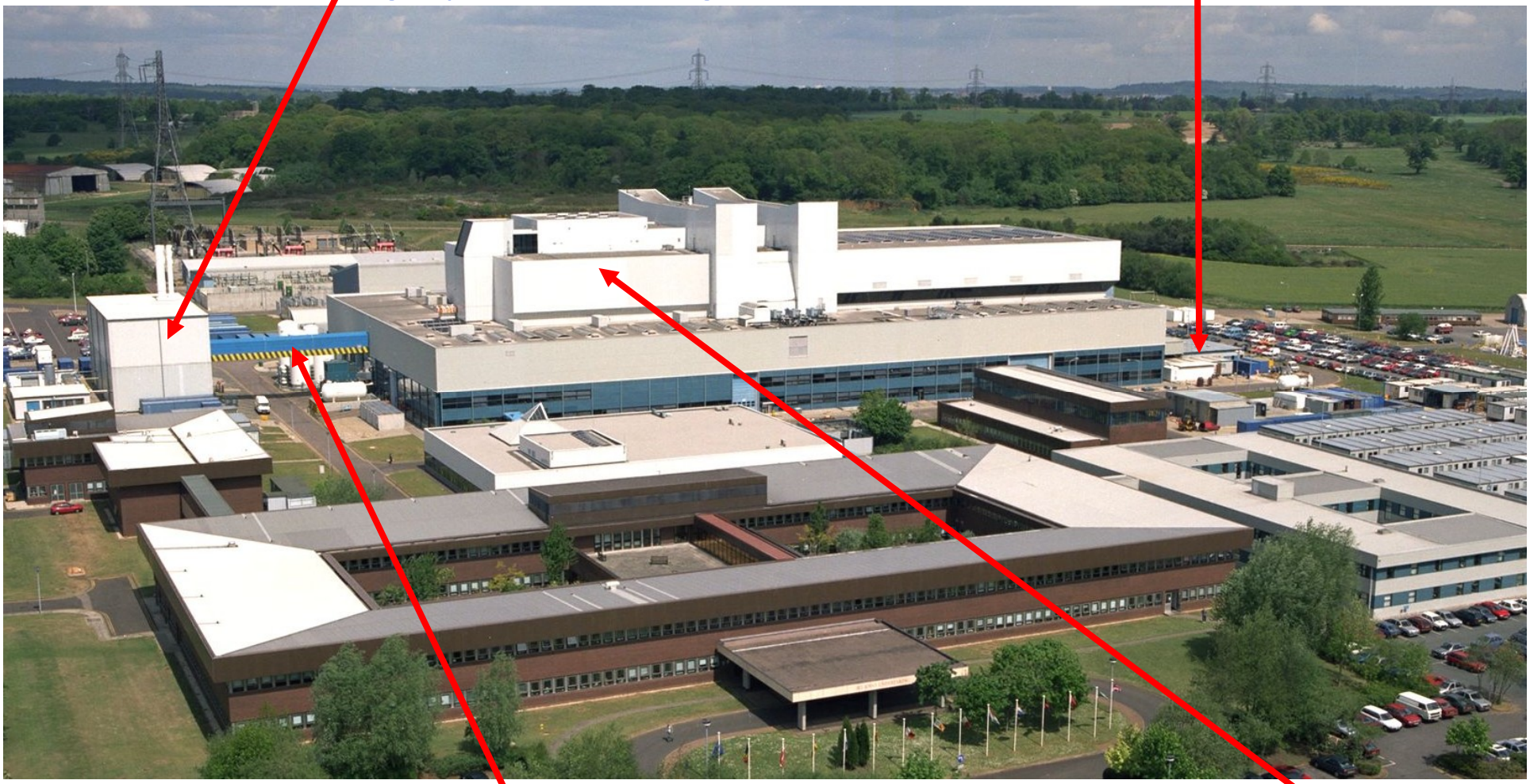
CCFE's experience in gaining and maintaining UKAS ISO17025 accreditation for beryllium personal air sampling and analysis.

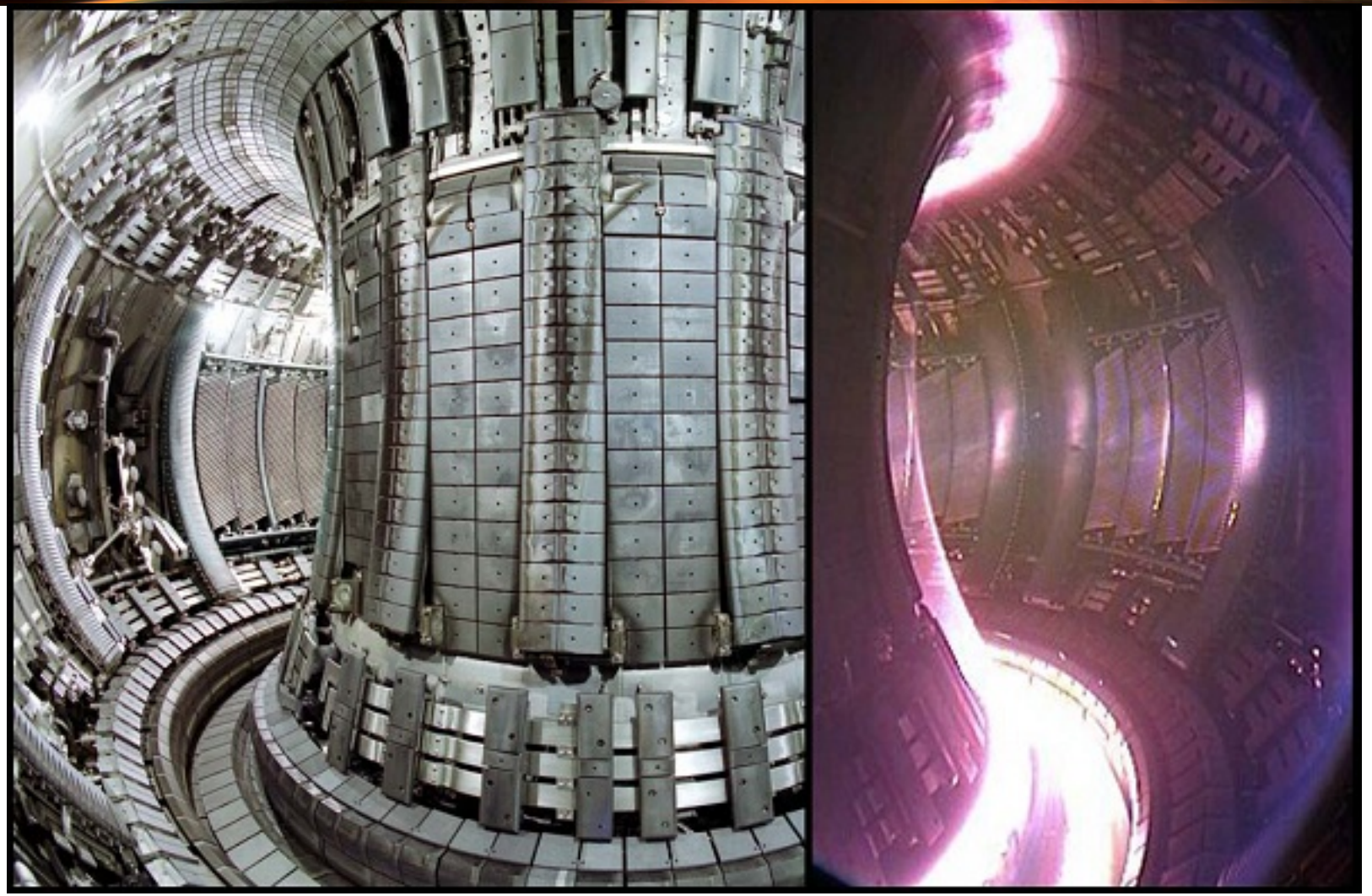
D.Campling (darryl.campling@ccfe.ac.uk),
M.Chandler, C.Roberts and the staff of the Health Physics Laboratory.

JET-EFDA, Culham Centre for Fusion Energy,
Culham Science Centre,
Abingdon,
OX14 3DB, UK.

CCFE's experience in gaining UKAS ISO17025 accreditation



CCFE's experience in gaining UKAS ISO17025 accreditation



New plasma heating systems can deliver up to 34MW of power.

http://www.efda.org/ http://www.iter.org/

Policy

- 100% Personal Air Sampling (PAS) policy. Work to the UK 2µg/m^3 WEL & an internal <10µg/m^2 surface contamination limit.

Motivation to gain Accreditation

- Beryllium (Be) exposures should be assessed as robustly as our radiological exposures.
- Radiological exposures assessed by an ADS. Licence issued by the HSE (the UK regulator).
- UKAS Accreditation - nearest equivalent standard we could aim for.
- Assurance for workers and management.
- Parallel to processing 50-70k samples/year.

Quality Management System; challenges, opportunities, qualifications.

- Benchmarked with nearby laboratories. (2009)
- Defining the QMS (QM, WI's, QA data, inter-comparisons & admin documentation etc) **was not** the main challenge. About 80 documents; procedures, WI's, admin control doc's, QA documents.
- Demonstrating compliance – using audits & proving technical competence **was**.
- Site ISO 9001 accreditation – only used to support procurement activities.
- Overcoming resistance to change.
- Not providing a commercial service – much simpler.

Technical challenges

- Decided to validate our current methodology
- Based upon HSE's MDHS 29/2 (hot H_2SO_4 + HNO_3 sample digestion of filter papers and FAA) but uses a lower temperature (180 C) and smaller volumes & diluted acids.
- System validated using high calcined BeO on Mixed Cellulose Ester filters (HPS) in the range 0.1 to 1µg/ml. System further cross referenced with Be acetate standards (HPS) 1µg/ml and liquid beryllium CRM's (PE & Sigma–Aldrich) 1000 µg/ml diluted to give a working range of 0.25 to 1 µg/ml

Technical challenges – sampling

- Pumps comply with BS EN1232. Acceptance criteria 0.05litre/min at 2litre/min. Calibrated with a Bios Defender 520 + Optimizer Collect Light software.
- Rapid re-charge, quick turn around.

- Stability on the flow rate over long sampling times. Run times up to 14hrs (with 1hr rest); <3% reduction in flow.
- Assessment of the affect of a blocked filter. Satisfactory up to 75% blockage.

Technical challenges – sampling

HSE – MDHS 14/3 (General methods for sampling and gravimetric analysis of desirable and inhalable dust) specifies this type of head.

- Validation for using a 2nd 'o' ring in PAS head. Implemented due to end- cap losses; no effect observed.
- Validation of the accuracy of pumps sampling timer.
- Number of sample 'reads' required to calibrate the sampler.
- Affect on flow rate of not recharging the pump. Within 5 days the variability is ≈2%.

Technical challenges – analysis

- Key validation experiments or work instructions
- Thermal mapping of the digestion blocks,
- Affect of temperature on the digestion of BeO samples. Results showed no affect on Be recovery >300ºC.

CCFE's experience in gaining UKAS ISO17025 accreditation

BLOCK: ERN 028 — **300°**

CONTROLLER: 021

	1	2	3	4	5
A	289	289	290	291	291
B	289	291	291	289	291
C	288	291	291	292	292
D	291	291	292	292	293
E	291	292	293	294	294
F	292	292	293	293	293
G	290	292	292	292	292
H	288	291	292	292	291

BLOCK: ERN 028 — **400°**

CONTROLLER: 021

	1	2	3	4	5
A	384	386	383	389	388
B	386	387	390	388	391
C	389	389	390	391	391
D	389	390	390	391	391
E	390	390	389	391	389
F	390	390	389	389	390
G	387	388	390	390	390
H	386	388	389	390	391

This indicates that the digester blocks operate to within 5% of the selected temperature

Technical challenges – analysis (cont'd)

- Pipetting errors; small changes (-20%) did not affect Be recovery.
- Al; used a 100ppm aluminium standard solution to test 1ppm Be recovery - no effect.

Thermo Fisher iCE3000 – Compact, Dual Beam, Computer controlled, Deuterium Background corrector.

Perkin Elmer A300 – Dual Beam, Deuterium Background corrector.

Technical - routine

- Calibration and determination of LoD's & LoQ's.
- PE A300 LoD=<0.02µg & LoQ=<0.06µg
- TF iCE3300 LoD=<0.06µg & LoQ=<0.2µg

in a 10ml sample.

- Balance calibration & checking, pipettes, bottle top dispensers & thermometers.
- Lab environmental monitoring; temperature & humidity. Later removed from accreditation system.
- 6 months after accreditation we introduced the use of an electronic LIMS system. The results had to be validated for the PAS process. Significantly improved the audit trail of samples.

CCFE's experience in gaining UKAS ISO17025 accreditation

PAS ANALYSIS SURVEY SHEET

DATE: 4-Sep-2012
RO: Tom Kinsella
HP: Gabbidon, Tony
TIME IN: 04/09/2012 11:58:56
TIME OUT 06/09/2012 09:10:30

Sample Receipt
Condition: UKAS
Date & Time: 04/09/2012 11:58:56
Receipient: Tony Gabbidon

LOCATION CA(T) No (If applic):-
J30 WHF
Location Description

Block Used:ERN: 026
Controller:ERN: 022
Be Standard Used: SS0372-SS0410
AA Used:ERN: 003

Survey Sheet No. 003944
Category

QA Controls

	Serial No	(µg) in Sample
Negetive Control	1128515	0
Positive Control	1128531	1.01

Notes

Comments(0)

Bench & Heads	Sample No	Sample Description	Date On	Date Off	Total MINS	Volume (m^3)	Results (µg) in Sample	Conc in µg/m^3 (PAS) in µg/m^2 (Smear)	PAS Head No.	PAS Pump ERN No.	RPE	No. Of Entries	E or M
	1		30/8/12	31/8/12	450	0.9	0	<.067	087	161	Disposable filt	5	E
	2		30/8/12	31/8/12	531	1.062	0	<.056	094	176	Disposable filt	6	E
	3		1/9/12	1/9/12	306	0.612	0	<.098	085	175	Disposable filt	3	E
	4		31/8/12	1/9/12	274	0.548	0	<.109	017	301	Disposable filt	3	E
	5		30/8/12	31/8/12	78	0.156	0	<.385	075	100	Disposable filt	2	E
	6		29/8/12	30/8/12	556	1.112	0	<.054	002	188	Disposable filt	7	E
	7		1/9/12	1/9/12	297	0.594	0	<.101	HP10	192	Disposable filt	3	E
	8		30/8/12	31/8/12	500	1	0	<.060	031	186	Disposable filt	6	E
☑	9	B&H	Smear area in cm^2	1000 cm^2		0	0	<0.3					

Processed By: Ilona Karnowska | Analysis Completed By: Brian Mennie | Checked By: Chris Roberts | Result Reviewed By: Angie Manning

Submit | Email Analysis sheet

Training & competency

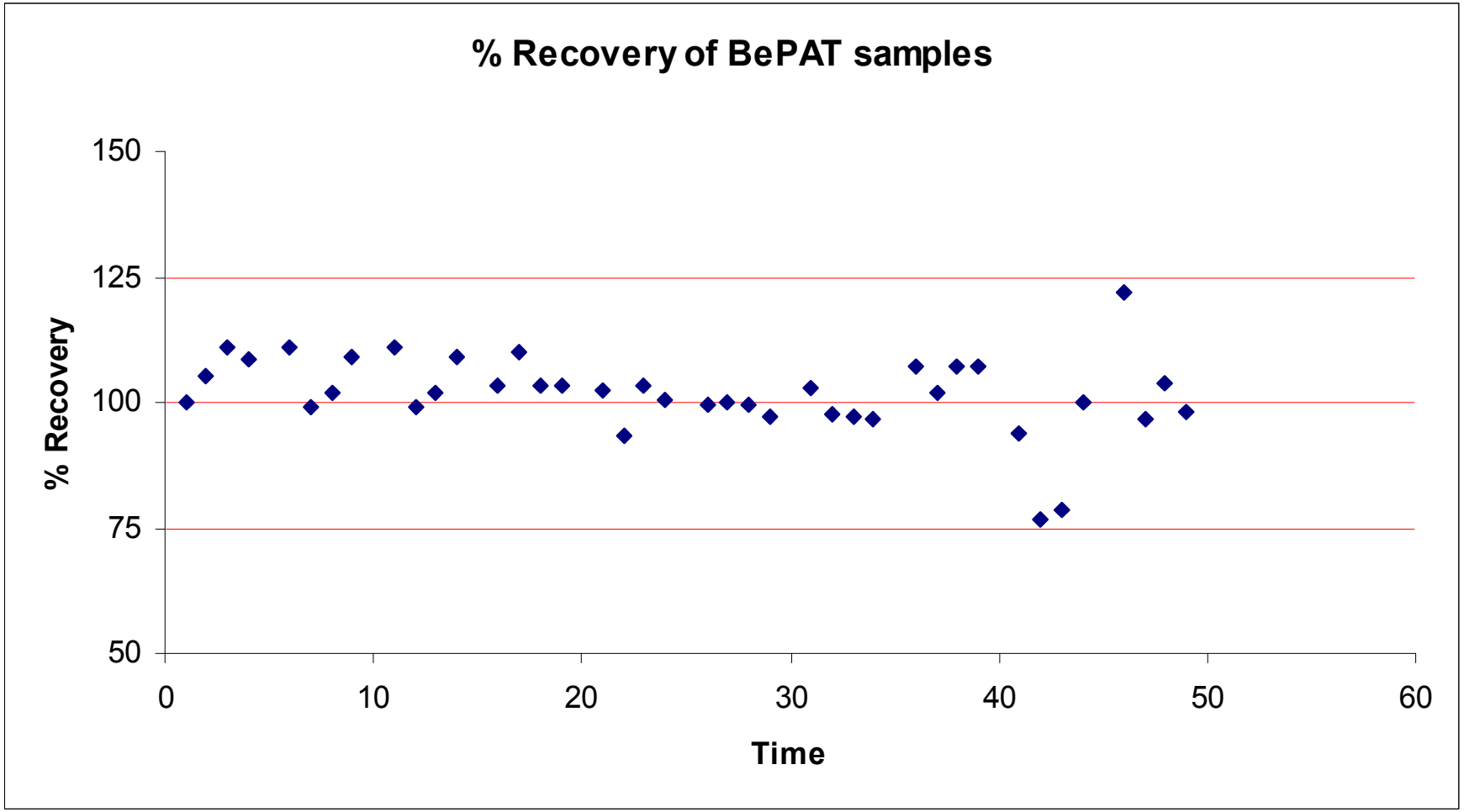
- Once WI's developed all approved operatives (5) trained 'in house' by the TM. Competence audited by the QM.
- Staff must demonstrate proficiency to become an approved operator. Re-assessed every 2 years or sooner if not used >6 months.
- LGC technique validation training for QM.
- UKAS ISO17025; awareness, auditor and measurement uncertainty training for key post-holders.
- Be workers trained to operate system as documented. Frequent staff changes! 150 approved PAS users. Video demonstration of how to enter & exit an area and wear a PAS and complete documentation. Sign to accept responsibilities.
- "Pipette Academy" training introduced to correct pipetting errors.

Streamlining

- Peripheral procedures discontinued. e.g. data gathered on environmental status of the lab.

- Validated samplers for use over 2 days in a 10 day period.
- Validation of samplers with a new calibration rig; parallel pump preparation.
- Changed the way data was entered on the PC (this saves about 3 min's per sampler).
- Changed processing of samplers once returned from site to allow rapid turn round. Do not fully dismantle the sampler assembly.

Intercomparisons - AIHA BePAT program



Given that we're the only lab in the scheme using FAA we consider this to be an excellent comparison.

Conclusions & audit findings

- Lesson learnt – validate existing technique; wasted time trying to work to a similar technique.
- As this was our first attempt at a technical (ISO17025) accreditation we overdid the generation of supporting procedures.
- The actions raised in the (3) surveillance visits have been minor; the assessors have been complimentary of the standard reached in 2 years.
- Last audit (Sept 2012) – 2 mandatory actions & 2 recommendations with recertification recommended.
- Achieving the accreditation has had a beneficial effect on the other processes carried out in the lab – standards raised.
- Interest from other groups and managers in getting accredited for other techniques; e.g. tritium in water. Saves the cost of using contracted accredited lab.

The future

- Currently validating moving to the ammonium bifluoride analysis technique. Reduces the need for a Safety Case for the facility, no acetylene or hot acids!!
- Full reassessment in 2013

United Kingdom Accreditation Service

ACCREDITATION CERTIFICATE

UKAS
UNITED KINGDOM ACCREDITATION SERVICE

TESTING LABORATORY
No. 4510

United Kingdom Atomic Energy Authority

is accredited in accordance with the recognised International Standard ISO/IEC 17025:2005
General Requirements for the competence of testing and calibration laboratories.

This accreditation demonstrates technical competence for a defined scope as detailed in and at the locations specified in the schedule to this certificate, and the operation of a laboratory quality management system (refer joint ISO-ILAC-IAF Communiqué dated 18 June 2005).

The schedule to this certificate is an essential accreditation document and from time to time may be revised and reissued by the United Kingdom Accreditation Service. The most recent issue of the schedule of accreditation, which bears the same accreditation number as this certificate, is available from the UKAS website www.ukas.org

This accreditation is subject to continuing conformity with United Kingdom Accreditation Service requirements. The absence of a schedule on the UKAS website indicates that the accreditation is no longer in force.

R. Bellingham
Accreditation Manager, United Kingdom Accreditation Service

Initial Accreditation date
30 March 2010

This certificate issued on
30 March 2010

The Department for Innovation, Universities and Skills (DIUS) has entered into a memorandum of understanding with the United Kingdom Accreditation Service (UKAS) through which UKAS is recognised as the national body responsible for assessing and accrediting the competence of organisations in the fields of calibration, testing, inspection and certification of systems, products and persons.

ammonium bifluoride analysis technique