Horia Hulubei National Institute for R&D in Physics and Nuclear Engineering (IFIN-HH)

Quality assurance of analytical measurements – a vital element in safety performance in nuclear field

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Virtual Scientific Workshop
Eurachem 2020
Quality Assurance for Analytical Laboratories in the University Curriculum
14 – 15 July 2020

INTRODUCTION

The approach of reporting results of chemical measurements together with measurement uncertainty is relatively new – 35 years

No Quality Assurance System → **no accurate results**

→ **not fit for purpose**

INTRODUCTION

Example

No	Parameter	Method	Maximum allowed value
1.	Chlorides	Turbidimetric	0.02 ppm
2.	Conductivity	Potentiometric	1 µS/cm
3.	pH	Electrometric	5.5 – 7.5

Turbidimetric method – LOD – 1.5 ppm visual
- 1.0 ppm UV-VIS

Conductivity cell sensitivity - 1 mS/cm

pH electrod - not suitable for pure water

} not fit for purpose

INTRODUCTION

- ***IUPAC***

"Protocol for the Design, Conduct and Interpretation of Method Performance Studies"

"Harmonized Guidelines for Internal Quality Control in Analytical Chemistry Laboratories"

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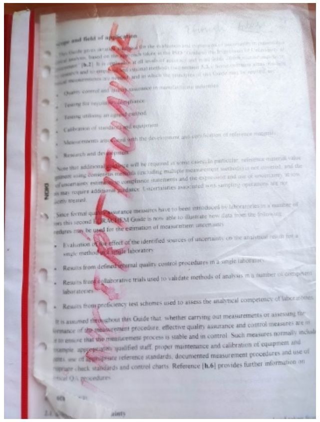
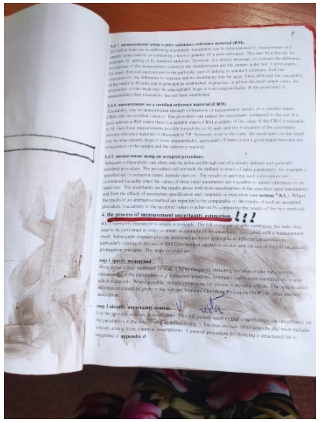
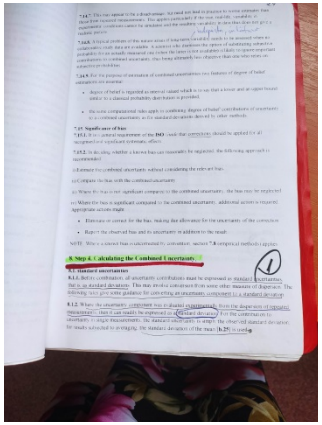
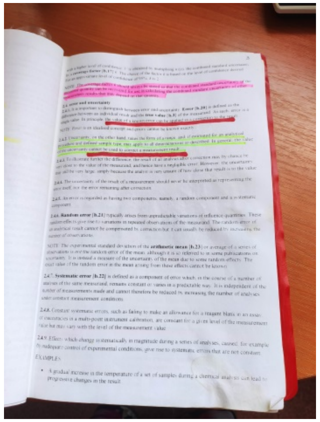
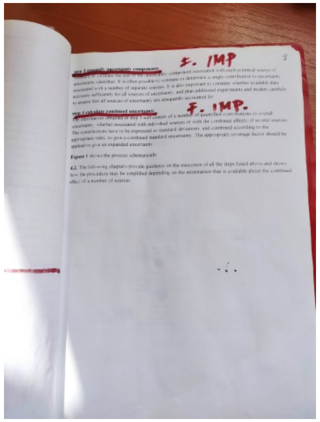
INTRODUCTION

- **EURACHEM**

"Quantifying Uncertainty in Analytical Measurement"

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INTRODUCTION

IAEA

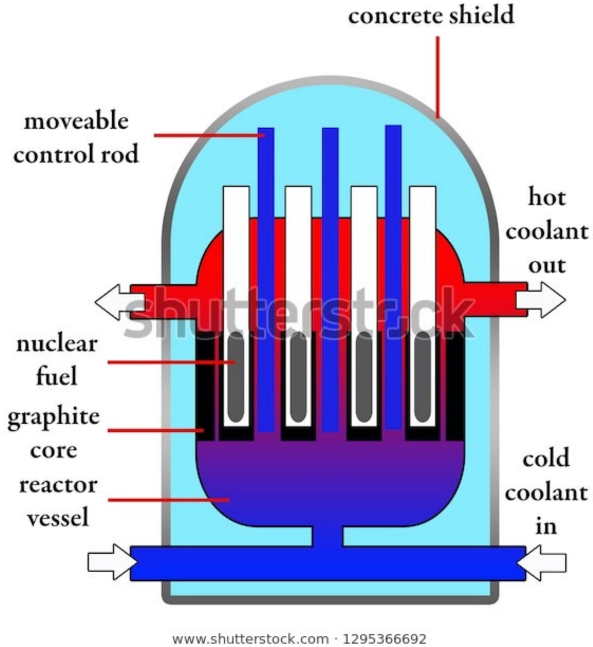
Safety Standards, Fundamental Safety Principles

Quantifying uncertainty in nuclear analytical measurements

NUCLEAR/RADIOACTIVE MATERIAL CHARACTERIZATION

Analytical chemistry of nuclear materials

- Nuclear fuels: uranium, thorium, plutonium
- Moderators
- Coolants
- Structural materials
- Reprocessed spent nuclear fuel

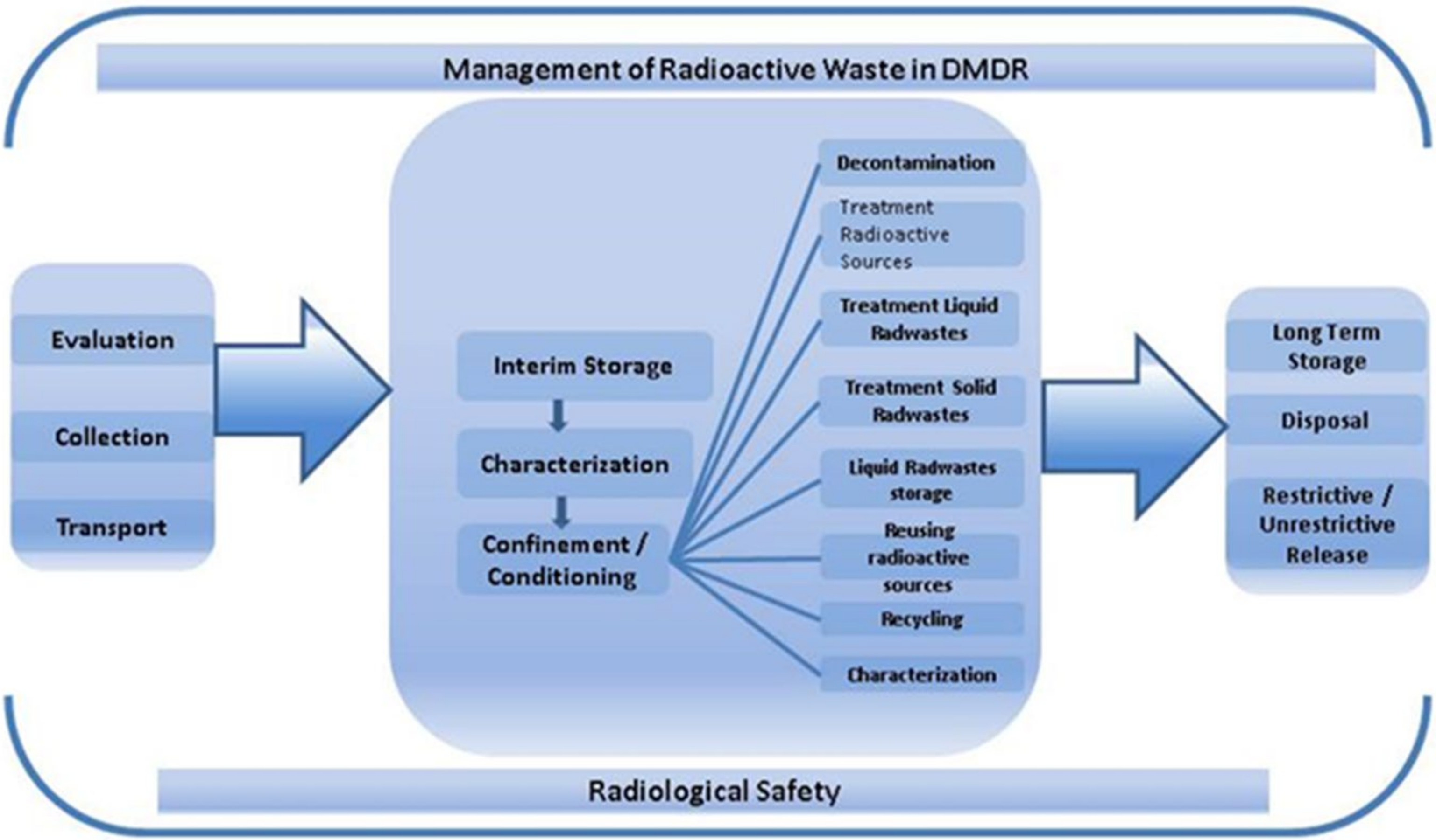


Characterization of radioactive waste

Steps:

- Collection
- Segregation ⟹ change waste streams' characteristics
- Treatment -
- Conditioning ⟹ immobilization ⟹ package
- Storage
- Disposal

Characterization of radioactive waste



Characterization of radioactive waste

Characteristics:

- physical - density, volume, shape, position of the waste and embedding matrixes, quality control, mechanical toughness, cracking, diffusion coefficient, gas release, thermal power, etc.

- chemical - elemental composition, content of toxic or reactive substances, etc.
-
- radiological - dose rate, alpha, beta and gamma activity, isotopic composition and mass of nuclear materials, etc.

Nuclear forensic

- prevention and detection of:
  - theft
  - sabotage
  - unauthorized access
  - illegal transfer
  - malicious acts

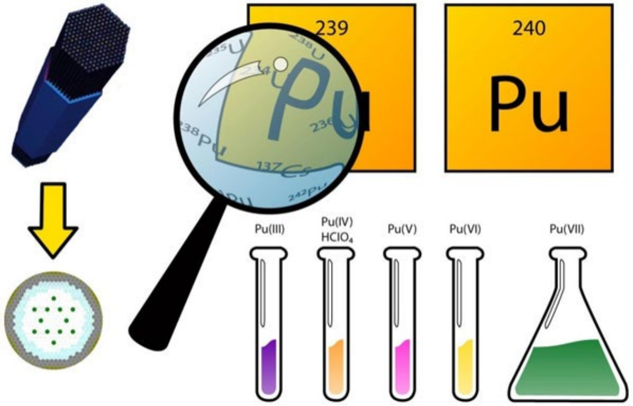
  involving nuclear material

- support law enforcement or nuclear security

Nuclear forensic

"'Nuclear forensic signatures"

- chemical or isotopic composition,
- elemental concentrations
- chemical impurities
- physical form
- chemical form
- physical dimensions
- visual appearance
- geometry



NUCLEAR ANALYTICAL MEASUREMENTS

- mass spectrometry
- ion beam analysis
- nuclear magnetic resonance spectrometry
- Mössbauer spectrometry
- neutron scattering and diffraction
- neutron activation analysis
- isotopic dilution analysis
- stable isotope and radiotracer studies
- direct radioactivity determinations

NUCLEAR ANALYTICAL MEASUREMENTS

Specific uncertainty features:

- error sources are traceable
- accuracy ⟶ $\dfrac{C^{f}_{Tr}}{C^{i}_{Tr}}$
- relative ⟶ calibration ⟶ uncertainty contributions - CRMs
  - calibration line fitting
- data acquisition ⟶ uncertainty – standard deviation of the Poisson distribution

NUCLEAR ANALYTICAL MEASUREMENTS

Specific uncertainty features:

- radioactive decay → uncertainty - non-linear components
  ↓
  minimization of the uncertainty by scaling of time intervals

- radiation → detector → uncertainity components – efficiency
  - saturation
  - dead time

- irradiation → uncertainty components – radiation field intensity
  - spectral field distribution
  - spatial field distribution

NUCLEAR ANALYTICAL MEASUREMENTS

Specific uncertainty features:

- background → uncertainty quantification for the results close to detection limit requires special attention

CHALLENGES IN THE QA OF ANALYTICAL TECHNIQUES IN THE NUCLEAR FIELD

Challenges
- Lack of matrix-matched certified reference materials
- Specialized equipment and processing
- Maintaining relevant expertise and capabilities
- Need for further improvements

Overcome:
- Comparing the analytical results obtained with different analytical techniques
- Combining the analytical results obtained with different analytical techniques
- Proficiency tests

CONCLUSION

- Quality Assurance (QA) for an analytical laboratory is an essential tool to ensure good comparability of data

- Educational initiatives are taken for re-enforcing the analytical chemistry curriculum, and this both on the conventional chemistry and measurement science

- Practices from nuclear field require a wide range of modern instrument-based analytical techniques, specialized equipment and processing and relevant expertise to lower radiation hazard

- Extensive research for the development of advanced methods for physical and chemical analysis with increased sensitivity, reliability and thereby enhanced accuracy are conducted to overcome present limitations

Thank you for your attention！