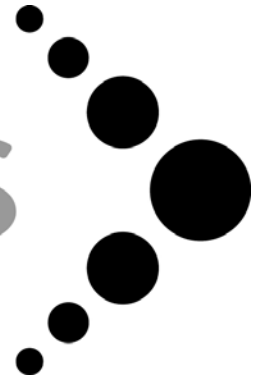


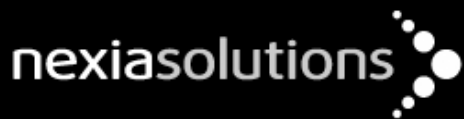
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*Nuclear expertise intelligently applied*



# Determination of Trace Uranium in Concentrated Plutonium Nitrate Solutions

Presented by : Marie Busquet



*Nuclear expertise intelligently applied*

# Introduction

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## ∞ Determination of trace U in Pu nitrate solutions : Current Method

- Reduction of Pu(IV) to Pu(III) by ferrous sulphamate
- Plutonium Elution & Retention of U(VI) on UTEVA Column
- Elution of Uranium with dilute nitric acid
- Analysis by ICP-MS or TRLF

# Introduction

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## ☞ Customer Requirement

- Turn-around of the analysis (<24h)
- Reduction of costs (hands on 3h)
- Waste generation (24cm<sup>3</sup> aqueous PCM waste)
- No Pressurisation of waste containers (sulphamic acid decomposition)

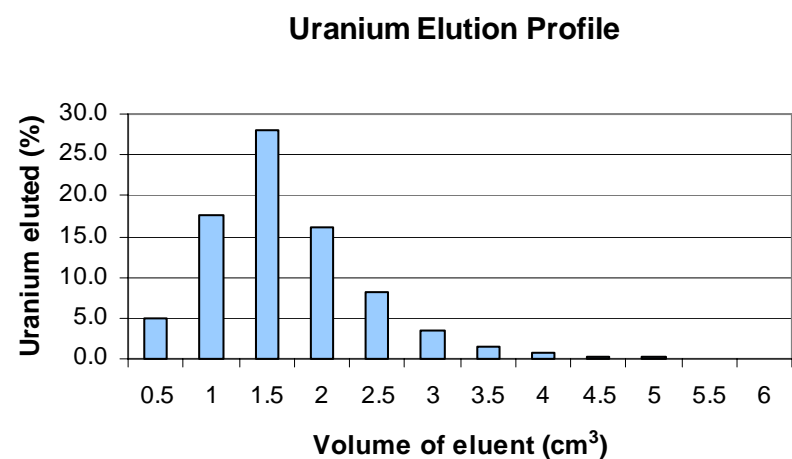
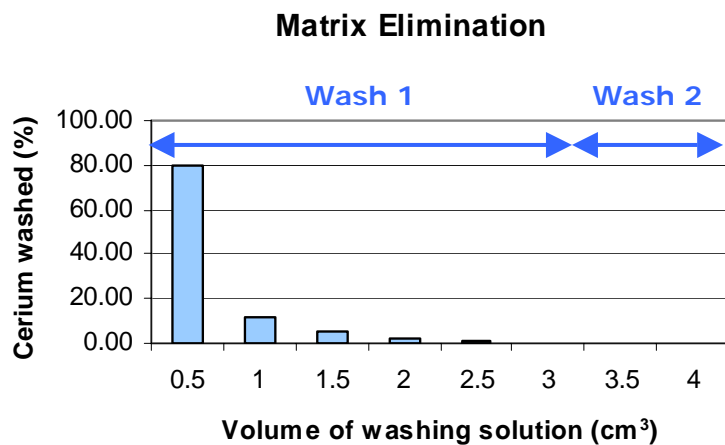
## ☞ Options

- Scaling down separation process
- Augmenting the flow through the SPE system
- Introducing galacturonic acid as plutonium reductant

# 1<sup>st</sup> Approach: UTEVA Packed Pipette Tips

## ☞ Efficient and Reproducible Extraction of Uranium

- Efficient Matrix Elimination: < alpha Fumehood limits
- >80% of Uranium Recovery



# 1<sup>st</sup> Approach: UTEVA Packed Pipette Tips

## ☞ Reduction of waste generation



<5cm<sup>3</sup> per sample

## ☞ Better turn-around



1h on the open bench

**BUT**

## ☞ Risk of repetitive strain injuries



large number of pipette operations (~66 per sample)

# 2<sup>nd</sup> Approach: Combination of options

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## ⌘ Improving the turn-around

- Implementation of a Vacuum Manifold
- Use of phosphoric acid as eluent for direct analyse by TRLF

## ⌘ Scaling down to reduce the PCM waste

- 1cm<sup>3</sup> UTEVA cartridge
- Smaller aliquot of sample

## ⌘ Pressurisation of waste

- Use of galacturonic acid

# Separation Process: Experimental

## ☞ Sample preparation

- Reduction of Pu(IV) to Pu(III) by addition of galacturonic acid

## ☞ Sample Loading

## ☞ Matrix Elimination

- 1<sup>st</sup> Wash with a mixture of nitric acid and galacturonic acid (1 x 1cm<sup>3</sup> then 2 x 0.5cm<sup>3</sup>).
- 2<sup>nd</sup> Wash with nitric acid only (2 x 0.5cm<sup>3</sup>).

## ☞ Elution of Uranium

- Elution of U with phosphoric acid (3 x 2cm<sup>3</sup>).

## ☞ Analysis by TRLF



# Method Characteristics

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☞ Matrix elimination

< alpha fumehood limits

☞ Analytical Recovery

95 – 104%

☞ Repeatability

< 9% RSD

# Method Validation

## Spike Recovery

	Recovery (%)
<b>High Standard (100mg.dm<sup>-3</sup>)</b>	86
<b>Sample point 333</b>	<b>Spike recovery (%)</b>
333 1	82
333 2	81
333 3	83
Mean	82
Standard deviation	1
<b>Sample point 2423</b>	<b>Spike recovery (%)</b>
2423 1	81
2423 2	68
2423 3	70
2423 4	82
Mean	75
Standard deviation	7

# Method Validation

## ☞ Accuracy of the Overall Method

- Preparation of Reference Materials by ID-TIMS
- Reference materials submitted to the optimised separation process and analysed by TRLF

	Uranium Concentration (mg.dm <sup>-3</sup> )
<b>Reference Material 1</b>	
Mean	12.6
Standard deviation	1.1
RSD (%)	9.0
Recovery of the "true" uranium concentration (%)	95
<b>Reference Material 2</b>	
Mean	591
Standard deviation	30
RSD (%)	5.1
Recovery of the "true" uranium concentration (%)	104
<b>Reference Material 3</b>	
Mean	4.5
Standard deviation	0.3
RSD (%)	5.7
Recovery of the "true" uranium concentration (%)	96

# Conclusion

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☞ Efficient matrix elimination

☞ Accurate Method                      95 – 104% Recovery / <9% RSD

☞ Reduction of waste                      6cm<sup>3</sup> per sample vs. 24cm<sup>3</sup>

☞ Better Total Turn-around                      5h vs. >24h

☞ Reduction of waste pressurisation



*Any Questions?*

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