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DATABASE APPLICATION FOR INPUT AND REVIEW OF INFORMATION ON ANALYTICAL MEASUREMENTS

**Usha I. Narayanan, M. Irene Spaletto, David T. Baran,
Alma V. Stiffin and Eric Dallmann**

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13. ABSTRACT (Maximum 200 Words) <p>An Analytical Measurements Information Database Application was developed to give an overall view of the criteria involved in the selection of an analytical measurement technique. This specific database application was developed for the measurement of elemental concentration of uranium. It includes information on many components of each measurement technique and allows easy comparison of different techniques. The integrated data information for the methods contained in this program include the specific technique, expected precision and bias, materials applicability, interferences, analysis time, reagents needed, training time, instrumentation required and its associated costs, and resulting process streams.</p> <p>Process stream information may be used to determine the method of preference based on pollution prevention opportunities. Use of this information also serves as an up-front indication of the types of waste generated when different analytical methods are implemented. Most sites, through pollution prevention programs and departmental mandates, are required to generate annual waste forecasts. The use of the process stream information greatly reduces the difficulty of predicting waste generation rates for different analytical methods, while the accuracy of such predictions is substantially increased.</p>				
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U.S. DEPARTMENT OF ENERGY

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AND REVIEW OF INFORMATION ON
ANALYTICAL MEASUREMENTS**

Usha I. Narayanan, M. Irene Spaletto, David T. Baran,
Alma V. Stiffin and Eric Dallmann

Research and Development Report

NEW BRUNSWICK LABORATORY

Argonne, Illinois

March, 1995

H. Rodney Martin, Acting Director

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ABSTRACT

An Analytical Measurements Information Database Application was developed to give an overall view of the criteria involved in the selection of an analytical measurement technique. This specific database application was developed for the measurement of elemental concentration of uranium. It includes information on many components of each measurement technique and allows easy comparison of different techniques. The integrated data information for the methods contained in this program include the specific technique, expected precision and bias, materials applicability, interferences, analysis time, reagents needed, training time, instrumentation required and its associated costs, and resulting process streams.

Process stream information may be used to determine the method of preference based on pollution prevention opportunities. Use of this information also serves as an up-front indication of the types of waste generated when different analytical methods are implemented. Most sites, through pollution prevention programs and departmental mandates, are required to generate annual waste forecasts. The use of the process stream information greatly reduces the difficulty of predicting waste generation rates for different analytical methods, while the accuracy of such predictions is substantially increased.

BACKGROUND

The value of an analytical measurement informational database was demonstrated when waste disposal problems were encountered with products from this laboratory's current analytical techniques. At the New Brunswick Laboratory (NBL), uranium is the element most commonly analyzed. NBL's present method of choice for analysis of elemental concentration of uranium involves titration with dichromate, a known human carcinogen. This results in the creation of mixed waste, for which disposal options are limited. (A waste is characterized as mixed if it contains both a radioactive component and another hazardous component.) Thus, there was a need to search for and select other, more environmentally sound analytical techniques. In this search, it was apparent that many factors needed to be considered and balanced to make the best choices. Therefore, the different criteria to be considered in the selection of any measurement technique were identified. Screen displays which represented the major decision-making criteria were selected and created. Relevant information was then input into each of the appropriate screens of the database application for selected techniques.

Each facility has its own criteria for selection of measurement techniques. With a change in mission, sample analyses that were once performed primarily in support of nuclear material accountability may now be performed primarily for environmental purposes. This may have an impact on the precision and accuracy required, or on the selection of measurement equipment. Budgetary cutbacks may require a closer look at the costs involved in the performance of a given technique. Personnel cutbacks may make training time or analysis time major points of consideration. Environmental concerns and waste disposal are becoming increasingly important.

Often, different groups at a facility are concerned with different aspects of an analysis, with no single group being aware of all areas of concern. Integrating analytical information into a single database application displays the selection criteria to all interested groups and facilitates the selection of an optimum method. In particular, waste generation information, while useful and frequently necessary to an organization, is often difficult to obtain. Typically, the people performing or overseeing the analytical work, and consequently responsible for generating the waste, are not the same people responsible for its disposal. This database allows for the expeditious transfer of predicted waste generation information from one group to another.

In this report, data on techniques commonly used for the determination of the elemental concentration of uranium are compiled and grouped. This database application can be expanded to incorporate new methods for uranium analysis as they are developed either on or off site. The application can also be expanded to include other related materials, such as plutonium, or be adapted to apply to virtually any other material and analytical method.

DESCRIPTION

The methods selection application is a DOS based, FoxPro generated, user-friendly computerized system designed to permit easy access, input and modification of the various components associated with a chemical technique. The ultimate goal of the application is the comparison of many techniques for the analysis of the elemental concentration of uranium by a knowledgeable individual in order to select the technique best suited for the individual's specific operation.

The application makes use of multi-linked databases to present information to the user. The user views an initial, General Information, screen for each particular technique. This general information includes the chemical element the method assays, the general method type and the specific technique (e.g.: Method = Spectrophotometry, Technique = X-Ray Emission), a range of the technique's known precision and bias, and a text box for any detailed notes the user may find helpful. Sample preparation, incorporating dissolution as a technique, is included as a separate method. Not every analytical technique included in this database requires dissolution; when it is required, the information from the Dissolution screens is to be compiled with the information for the specific analytical technique. If dissolution is required, the note "Use with sample prep screen" appears on the General Information screen for the individual technique.

The General Information screen also contains the usual data manipulation option "buttons," such as <Next>, <Back>, <Browse>, <Add>, <Delete>, <Edit>, <Save> and <Cancel>. These "buttons" permit the user to page through, add, delete, or edit different methods or techniques, and save or cancel editing changes. Also included on the main screen are five additional "buttons", < Materials >, < Interferences >, < Analytical >, < Equipment > and < Process Streams >, which lead the user to specific components associated with the selected technique. Each of these "buttons" calls up another screen with additional information about the specific component topic. Each screen also includes a text box for additional notes. As with the main screen, each of these screens have the usual data manipulation buttons for easy user interface.

The < Materials > screen details the types of materials for which the technique is applicable.

The < Interferences > screen details the specific types of interferences to the technique and treatments, to eliminate the interferences, if known.

The < Analytical > screen contains detailed information about the analysis component of the technique. The screen shows the typical sample size, any additional material amounts used in the analysis, the analysis time and the training time for an analyst to learn the technique. A <Reagent> button generates a screen showing reagents required for the analysis.

The < Equipment > screen contains detailed information listing the equipment major components, the associated capital and maintenance costs and the training time for analysts to learn to operate the equipment.

Finally, the < Process Streams > screen contains information about wastes generated by the technique. The wastes are categorized into Hazardous, Non-Hazardous, Radioactive, Mixed, and Other, and sub-categorized into solid, liquid, and gaseous phase. The units displayed on this screen are in, or are easily converted to, those typically requested in reports to federal and state regulatory agencies. Data for solid waste are in cubic feet, for liquids are in milliliters, and for gases are in pounds. All waste streams generated by the analytical method, including indirectly generated waste such as paper towels, are included. Details regarding the wastes, especially those in the "Other" category, may be entered into the process stream screen text box. Process stream data is entered for sets of ten samples; sample set data is more conveniently used than single sample data because single samples are rarely, if ever, analyzed. Also, indirectly generated wastes remain relatively stable for one to ten samples and are more accurately estimated for a set.

This screen may also be used to estimate annual waste forecasts, as required by pollution prevention programs and departmental mandates. Previously, the upcoming calendar year's forecast was often based on some percentage of the current year's generation rate, taking into account anticipated changes in sample load. If changes in analytical methods were proposed, the resultant waste generation data became elusive. As this database allows for the incorporation of methods performed at other sites, comparison of predicted waste generation rates for different analytical methods becomes much easier and more accurate.

APPENDIX A

METHODS AND TECHNIQUES INCLUDED IN DATABASE APPLICATION

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METHODS AND TECHNIQUES INCLUDED IN DATABASE APPLICATION

Sample Preparation

Dissolution

Electrochemical

U coulometry

Gamma Spectroscopy

High Resolution

Gravimetry

U Gravimetry

Isotopic

U-IDMS

Spectrophotometry

Fluorometry

X-Ray Emission

Phosphorimetry

Titration

Davies and Gray

Ceric Titration

Printouts of all the different screens for Dissolution and the Davies and Gray Titration techniques appear in the following Appendices B and C.

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APPENDIX B

SCREENS FOR SAMPLE DISSOLUTION

Preceding Page Blank

General Information	
Element: U Method: Sample Preparation Technique: Dissolution	Notes: To prepare a solution of 10 g/L. Precision and bias not applicable.
Precision <input type="text"/>	Bias <input type="text"/>
<div><Materials> <Interferences></div> <div><Analytical> <Equipment> <Process Streams></div> <div>< Next >< Back ><Browse>< Add ><Delete>< Edit >< Save ><Cancel><Locate></div>	

Preceding Page Blank

File Edit Application Window Help

General Information	
Element: U	
Method: Sample Preparation	Notes:
Methods: Materials	
Technique: Dissolution	
Material: pure uranium compounds	
Notes: use HNO3	
<div style="text-align: right;"> </div>	
< Next > < Back > <Browse> < Add > <Delete> < Edit > < Save > <Cancel>	
< OK >	

File Edit Application Window Help Browse

Material Type	MATERIAL
pure uranium compounds	
scrap	
waste	
U-Al	
U-Si	
U-Zr	
carbides	
< OK >	

File Edit Application Window Help

Methods: Interferences

Technique: Dissolution

Interference: insoluble impurities

Notes: measure residue by gamma spectroscopy

Comments: Insure that all U has been leached out

< Next > < Back > <Browse> < Add > <Delete> < Edit > < Save > <Cancel>

< OK >

Methods: Sample Analysis

Technique: Dissolution

U sample size:

0.5

g

Analysis time:

8

hr

Additional
material:

50

mL

Training time:

5

day

Notes: "Analysis time" = dissolution time

<Reagents>

< Next > < Back > <Browse> < Add > <Delete> < Edit > < Save > <Cancel>

< OK >

File Edit Application Window Help

General Information			
Methods: Equipment			
Technique: Dissolution			
Instrument: 1000°C muffle furnace			
Purchase cost:	\$2000	Training time:	2 hr
Maintenance cost:	\$0		
Notes:			
<div></div>			
< Next > < Back > <Browse> < Add > <Delete> < Edit > < Save > <Cancel>			
< OK >			

File Edit Application Window Help Browse

INSTRUMENT			
Name	Purcost	Maincost	Traintim
1000°C muffle furnace	2000	0	2
4-pl balance	3000	40	8
2-pl large cap. balance	2000	40	2
Pt ware	0	0	0
hot plate	150	0	0

Mai

< Ne

Cancel>

General Information					
Methods: Process Streams					
Technique: Dissolution			The values listed are for a set of 10 samples dissolved in 1L of solution per sample.		
	Solid		Liquid		Gas
Hazardous		ft^3		ml	lbs
Non-Hazardous		ft^3		ml	lbs
Radioactive	3.0	ft^3	10,000	ml	lbs
Mixed		ft^3		ml	lbs
Other		ft^3		ml	lbs
< Next > < Back > <Browse> < Add > <Delete> < Edit > < Save > <Cancel>					
< OK >					

APPENDIX C

SCREENS FOR DAVIES AND GRAY TITRATION TECHNIQUE

General Information

Element: U
Method: Titrimetry
Technique: Davies and Gray

Notes:
Use with sample prep
screen.

Precision

Bias

0.1 - 0.5 %

0.1 - 0.5 %

<Materials> <Interferences>

<Analytical> < Equipment > <Process Streams>

< Next >< Back ><Browse>< Add ><Delete>< Edit >< Save ><Cancel><Locate>

Preceding Page Blank

File Edit Application Window Help

General Information		
Element: U		
Method: Titrimetry		Notes:
Methods: Materials		
Technique: Davies and Gray		
Material: pure uranium compounds		
Notes:		
<div></div>		
< Next > < Back > <Browse> < Add > <Delete> < Edit > < Save > <Cancel>		
< OK >		

File Edit Application Window Help Browse

Material Type	MATERIAL
pure uranium compounds	
scrap	
waste	
U-Al	
U-Si	
U-Zr	
carbides	
U-Th	

< OK >

File Edit Application Window Help

Methods: Interferences

Technique: Davies and Gray

Interference: halides, Mo, Tc

Notes: fume with H2SO4

Comments:

< Next > < Back > <Browse> < Add > <Delete> < Edit > < Save > <Cancel>

< OK >

File Edit Application Window Help Browse

INTERFER	
Interference Type	Notes
I halides, Mo, Tc	fume with H2SO4
Zr, Si, Th	add HF
thorium	dry; do not fume
aluminum	do not add sulfates
reagent temperature	keep room temp. >23 and <31°C
H3PO4 conc. in sample	don't exceed specified reagent volumes
As, Sb, Sn in H3PO4	add dichromate to acid or sample
free acid volumes	fume or separate
Hg, Pt, Pd	Separate by Cu column
Ru, Os	fume with HClO4 & HNO3
< V, Mn, Pd, Ag, Ir, Pt, Hg	TBP/cyclohexane extraction

File Edit Application Window Help

Methods: Sample Analysis			
Technique: Davies and Gray			
U sample size:	25-300	<input type="text" value="mg"/>	Analysis time: 15 <input type="text" value="min"/>
Additional material:	200	<input type="text" value="mL"/>	Training time: 2 <input type="text" value="wk"/>
Notes: <div></div>			
<Reagents>			
< Next > < Back > <Browse> < Add > <Delete> < Edit > < Save > <Cancel>			
< OK >			

File Edit Application Window Help Browse

AREAGENT	
Technique: Davies	Reagent
Technique:	dichromate titrant
Sample reagent:	H3PO4
< Next > < Back >	HNO3
	sulfamic acid
	FeSO4
	ammonium molybdate
	vanadyl sulfate
	H2SO4
< Next > < Back > <	gents
	Edit > < Save > <Cancel>
	dit > < Save > <Cancel>

File Edit Application Window Help

General Information

Methods: Equipment

Technique: Davies and Gray

Instrument: 4-pl balance

Purchase cost: \$3000 Training time: 2 hr

Maintenance cost: \$40

Notes: maintenance cost for 1 yearly calibration

< Next > < Back > <Browse> < Add > <Delete> < Edit > < Save > <Cancel>

< OK >

File Edit Application Window Help Browse

Name	INSTRUMT		
	Purcost	Maincost	Traintim
4-pl balance	3000	40	2
pH/mV meter	1800	0	2
calomel electrode	50	0	0
Pt electrode, 16 gauge	0	0	0
magnetic stirring plate	150	0	0
hot plate	150	0	0

Mai

< Ne

Cancel>

General Information
Methods: Process Streams

Technique: Davies and Gray

The values listed are for a set of ten analyses.

	Solid		Liquid		Gas
Hazardous		ft^3		ml	lbs
Non-Hazardous		ft^3	500	ml	lbs
Radioactive	1.0	ft^3		ml	lbs
Mixed		ft^3	2000	ml	lbs
Other		ft^3		ml	lbs

< Next > < Back > <Browse> < Add > <Delete> < Edit > < Save > <Cancel>

< OK >

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