

A DEFENSIBLE DATA PACKAGE FOR RADIOCHEMISTRY

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ABSTRACT

We have developed a unique computer software package in response to increasing demands by government and industry to present radiochemistry data in a manner that is both technically and legally defensible. The demand for defensible data has arisen within the U.S. EPA, DOE and DOD because of the potential for litigation related to site clean up activities. The Contract Laboratory Program has addressed this issue for certain organic and inorganic analyses; however, the presentation of radiochemistry data in a defensible format has not yet been standardized. The software presented is adaptable to various methods and protocol requirements, which are incorporated as an integral part of the program. The data package produced is designed to aid the reviewer in making data quality decisions.

INTRODUCTION

The U.S. Environmental Protection Agency (EPA), the U.S. Department of Energy (DOE), and the U.S. Department of Defense (DOD) have been increasingly concerned with the quality and credibility of analysis of radionuclides by commercial laboratories because of the potential for litigation related to site clean up under their jurisdiction. Creating a software package for the presentation of radiochemistry data in a technically and legally defensible format required us to take into consideration both the varied and proprietary nature of radiochemistry methods currently being used by commercial laboratories and the variety of requirements imposed by each major client.

Currently available radiochemistry procedures such as those developed by Los Alamos Scientific Laboratory (1), the U.S. DOE Environmental Measurements Laboratory (2), and the U.S. DOE Radiological and Environmental Sciences Laboratory (3) are often recommended as guides for analysis to commercial laboratories but are not compulsory. Methods have also been specified by the U.S. Environmental Protection Agency (EPA) for the analysis of drinking water and waste water (4,5,6). However, variations on published methods are often required to achieve success in the analysis of soil, groundwater, sludge, waste materials, and biota encountered in remedial investigation activities. In many cases, these adapted procedures are developed by industry and are proprietary.

The EPA has developed SOW for organic and inorganic chemical analysis that specifies analytical methods, quality control procedures, and an analysis structure that generates data of known and documented quality and that must be followed under contract with qualified laboratories (the Contract Laboratory Program, CLP). Data packages under this program are strictly formatted, have flags on reported parameters to indicate variation from strict compliance with specified values, and contain sufficient raw data to allow independent quality control review (7). Most clients defer to these CLP protocols when specifying chemical analysis, although some minor variations may be requested. For radiochemistry, several of the DOE facilities have provided analytical protocols that specify data quality objectives and the documentation that is required for reporting. Also, the EPA is currently involved in designing a statement of work for

radiochemical analysis (8). Recently Wegner and Thurman (9) have discussed the requirements of a complete CLP type program model for radiochemistry. However, to date there are no standard radiochemistry analytical protocols for remedial investigation and remedial action activities.

The software package describe here is adaptable to various radiochemical procedures and protocol requirements. The data package produced is designed to aid the reviewer in making data quality decisions and determining defensibility.

DISCUSSION

The software program, that we refer to as the Data Validation and Deliverable (DVD) system, produces a CLP-like set of deliverables for radiochemistry. The software is written System-J and is of a modular, database design. Data input can be either manual or by way of electronic transfer from other computer systems; the latter is much preferred to minimized transcription errors. Data gathered during sample receipt and login can be retrieved electronically from the Radian Sample Analysis Management (SAM) or similar laboratory management systems. Once specific quality control and reporting requirements are incorporated into the DVD system, these parameters are used to cross-check laboratory input to determine if all protocol work has been done and to flag deficiencies. Flagging of deficient data allows the laboratory to either correct deficiencies or address them in the case narrative. Various client requirements for data quality parameters such as minimum detectable activities (MDA), comparability of replicates, spike recovery, and counting times can be entered into individual program modules and can be used to produce client specific data packages.

The DVD system is designed to be user friendly, with menus used to prompt the user. The user screen in Fig. 1 shows an example of the expanding menu feature. New users of the DVD system can become proficient quickly with minimal training. Additional support is provided in help screens and imbedded documentation. The user screens (as displayed in Fig. 2) are highlighted to draw attention to important parameters. Pop up screens give additional information and provide menu options for further investigation. The parameters that are out of compliance with the operational protocol are highlighted or underlined as in the case of the low yield and high MDA on the uranium result shown. By using the function keys

01/27/92 16:16:29

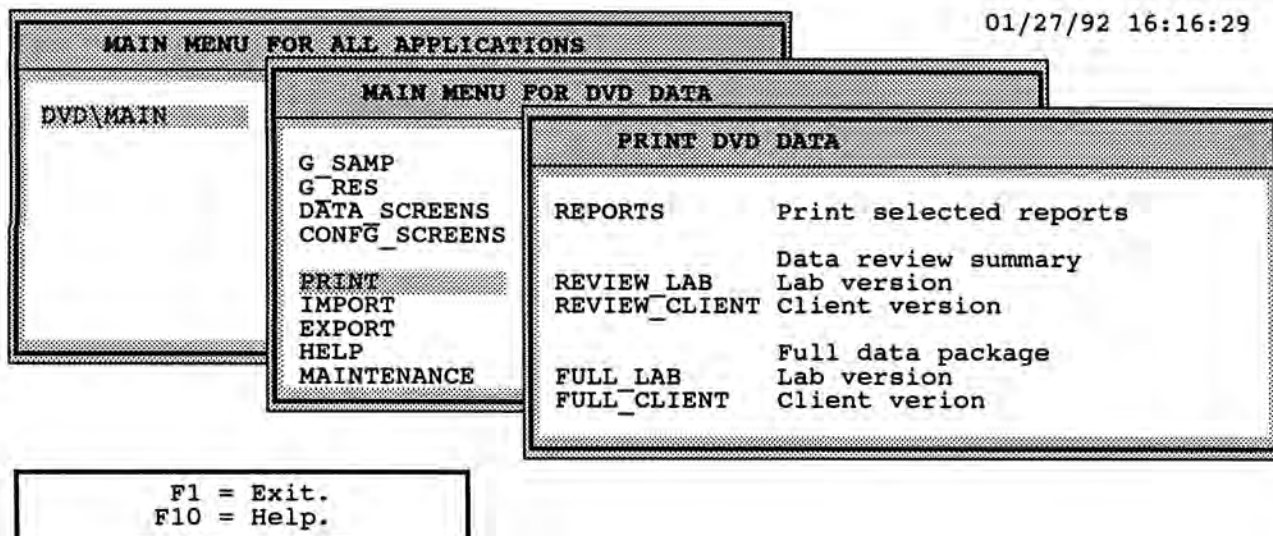


Fig. 1. Reproduction of the expanding menu feature of the DVD system showing the various reporting options available for printing. All reports are available on screen displays as well allowing the user to review data by browsing prior to printing. Also note the availability of the help screen.

as defined in the bottom panels, one can readily move to other screens for browsing and checking results.

A few of several possible pages from a typical report are provided to give the reader a sense of the DVD system. The reports follow a CLP-like style and are named to be consistent with CLP terms when feasible.

One set of reports provides information on the samples that are included in the data package and how they are batched for QC purposes. Lab Summary reports (not shown) are used to verify that all desired samples are actually reported. The QC Summary (Fig. 3) is used to check that the client sample ID's are properly transcribed from the chain-of-custody documents and provides information on the QC samples that were run with the submitted samples. The Lab Work Summary (not shown) is used to check that all of the desired analyses are being reported. The Preparation Batch Summary (not shown) allows the user to check that all desired data is reportable, that all QC was performed, that compatible matrices were run together, and that all occurring result qualifiers have been addressed.

A second set of reports provide per sample information similar to a CLP data package. Sample Data Sheets and reports on blanks, laboratory control samples (LCS), and replicates are available. In Fig. 4, a Laboratory Control Sample report demonstrates how data that are associated with problems are underlined. Such problems need to be corrected or addressed in the data package narrative. In this example, MDA's were not met for three analytes.

A third set of reports presents the results of analysis. The Results Summary (Fig. 5) provides the result of an analysis for each sample and QC sample in the batch with flags on non-compliant data. The Method Summary (Fig. 6) provides information about the parameters related to the chemistry preparation and counting aspects of the analysis. Here the measured MDA and yield do not meet the protocol requirements and are, therefore, flagged. Note that the procedures used for the analysis are listed on this summary; each procedure is written in the DVD system and is retrievable for review.

Finally, the DVD system will produce an electronic deliverable from the same data that is presented in hard copy. This approach eliminates inconsistencies between the two formats that could be caused by input or transcription errors. The electronic deliverable can be customized to meet various protocol requirements.

As in a CLP data package, the radiochemistry data package produced by the DVD system is supplemented with copies of raw data, laboratory notebooks, and shipping documents. The completed package provides a complete set of information that allows a data reviewer to judge the validity of the data submitted. This combine with periodic background information either submitted or obtained during laboratory audits would give the client confidence that the data provided is defensible.

SUMMARY

The DVD system is designed to produce a CLP-like set of deliverables for radiochemistry using input from varying laboratory methods and client protocols. The modular, database design allows the methods and protocols to become an integral part of the system. The data are presented in formats that allow the laboratory to browse the data which can aid in initial and final quality reviews. Data that does not meet the protocol requirements are flagged so that the laboratory can either correct deficiencies or address them in the narrative. The final hard copy and electronic deliverables are produced from the same data base.

This system is currently *in use* on two major contracts in support of RI/FS activities at DOE sites that involve organic, inorganic and radiochemical analysis. The radiochemistry results provided from the DVD system compliment the CLP deliverables for organic and inorganic results.

REFERENCES

1. A. GAUTIER, Editor, "Health and Environmental Chemistry Analytical Techniques, Data Management and Quality Assurance," LA-10300-M Manual, Los Alamos National Laboratory (September 1987).

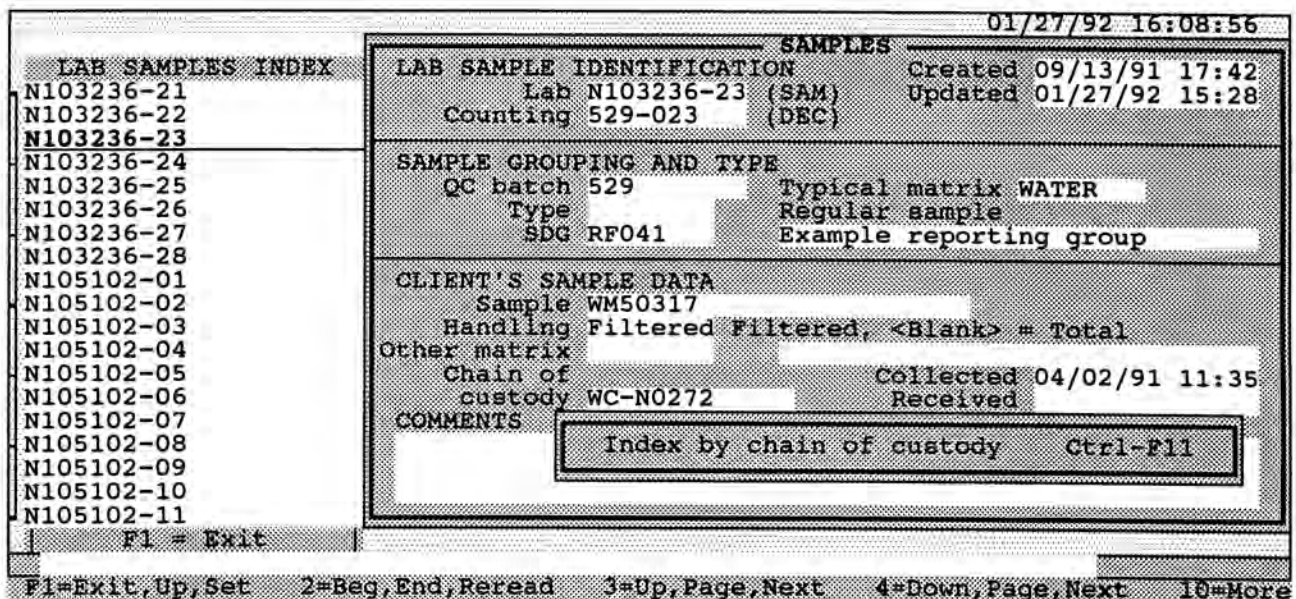
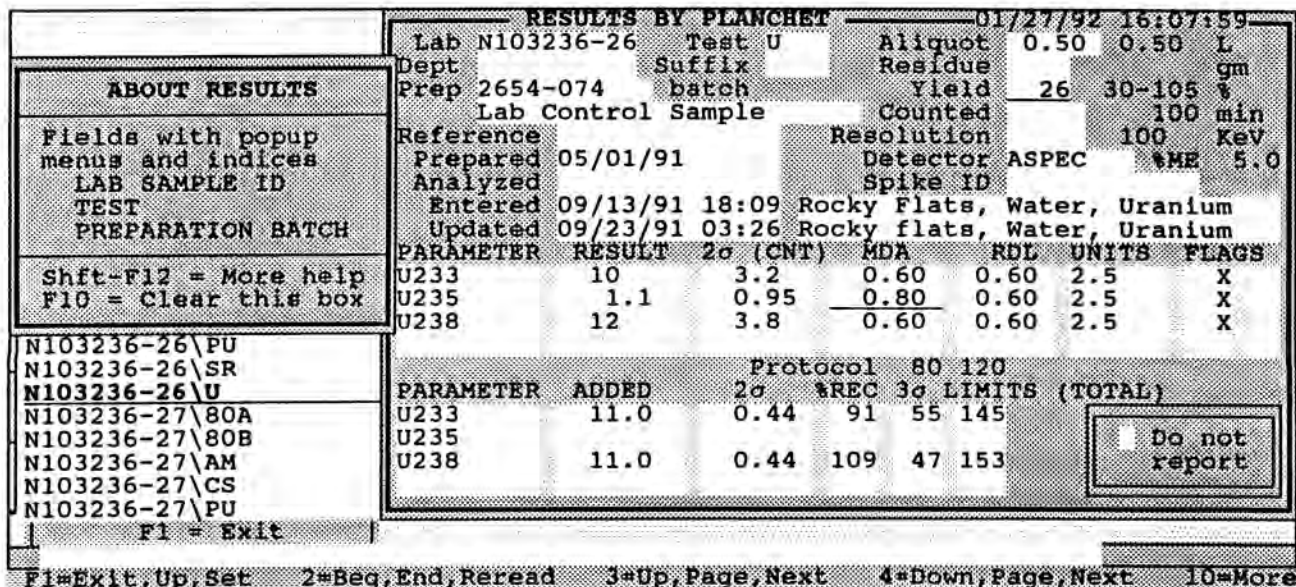


Fig. 2. Two examples of the DVD system screens. Here the Samples screen (above) provides information about the sample. Information on the samples displayed on the left can be browsed by using the function keys. The Results by Planchet (below) are presented along with the parameters required by the operational protocol. Out-of-spec values are underlined or highlighted on the screen. Note the pop-up "About Results" on the left.

2. A. CHIECO, C. BOGEN, and O. KNUTSON, Editors, "EML Procedures Manual," HASL 300, 27th Edition, Environmental Measurements Laboratory, U.S. DOE, New York (November 1990).
3. Z. BODNAR, and R. PERCIVAL, Editors, "RESL Analytical Chemistry Branch Procedures Manual," Radiological and Environmental Sciences Laboratory Branch, U.S. DOE, Idaho Falls (1982).
4. L. KRIEGER and L. WHITTAKER, "Prescribed Procedures for Measurement of Radioactivity in Drinking Water," EPA 600/4-80-032, Environmental Monitoring and Support Laboratory, Office of Research and Development, U.S. EPA, Cincinnati (1980).
5. S. CLESCERI, E. GREENBERG, and T. R. RHODES, "Standard Methods for the Examination of Water and Wastewater," Part 7000, 17th Edition, American Public Health Association, Wash., DC (1989).
6. ASTM, "Annual Book of ASTM Standards, Nuclear, Solar, and Geothermal Energy," Vol. 1201, Nuclear Energy ASTM, Philadelphia (1987).

THERMO ANALYTICAL
EXAMPLE REPORTING GROUP
LAB CONTROL SAMPLE

N103236-26

LAB CONTROL SAMPLE

SDG RF041
Contact Julie Wose

Client Company
Contract DS-JK-LL

Lab sample id N103236-26
Dept sample id 529-026
Last analysis 05/05/91

Matrix WATER
Material _____

PARAMETER	RESULT pCi/L	2 σ ERR (COUNT)	MDA pCi/L	RDL pCi/L	QUALI- FIERS	SUF- TEST FIX	ADDED pCi/L	2 σ ERR pCi/L	REC %	3 σ RECOV (TOTAL)	LIMITS PROTOCOL
Gross Alpha	14	3.2	1.2	2.0	X	80A	19.9	0.80	70	67-133	70-130
Gross Beta	26	2.2	1.0	4.0	X	80B	27.8	1.1	94	75-125	78-122
Tritium				400							
Plutonium 239/240	0.16	0.033	<u>0.016</u>	0.010	X	PU	0.180	0.007	89	71-129	80-120
Uranium 233/234	10	3.2	0.60	0.60	X	U	11.0	0.44	91	55-145	80-120
Uranium 235	1.1	0.95	<u>0.80</u>	0.60	X	U					80-120
Uranium 238	12	3.8	0.60	0.60	X	U	11.0	0.44	109	47-153	80-120
Americium 241	0.18	0.036	<u>0.012</u>	0.010	X	AM	0.180	0.007	100	68-132	80-120
Strontium 89/90	8.7	0.68	1.0	1.0	X	SR	10.4	0.42	84	83-117	75-125
Cesium 137	12	0.41	0.30	1.0	X	CS	10.7	0.43	112	80-120	75-125
Radium 226				0.50							
Radium 228				1.0							
Curium 244				1.0							
Neptunium 237				1.0							
Thorium 230/232				1.0							

Underlined MDA's are greater than the corresponding RDL's. Other out-of-spec data would be underlined as well.

2 σ percent non-counting method error by test.

80A	20.0
80B	15.0
AM	5.0
CS	10.0
PU	5.0
SR	10.0
U	5.0

Lab id TMAN
Protocol GRRASP
Version Ver 1.0
Form DVD-LCS
Version 01/22/92
Report date 01/27/92

Fig. 3. The QC Summary report that is used to check that the client sample ID's are properly transcribed from the chain-of-custody documents and that adequate QC samples were run.

- U.S. EPA, "Users Guide to the Contract Laboratory Program," 64-71, U.S. EPA Office of Emergency and Remedial Response, Wash., DC (1988).
- R. McCALISTER, "Status of Low Level Radioactive/Mixed Waste Statement of Work," CLP Analytical Caucus, San Diego, (March 1991).
- K. WEGNER and R. THURMAN, "Tuning in on Radiochemistry," Environmental Lab, (Oct-Nov. 1991).

THERMO ANALYTICAL
EXAMPLE REPORTING GROUP

SDG RF041
Contact Julie Wose

QC SUMMARY

Client Company
Contract DS-JK-LL

QC BATCH	CHAIN OF CUSTODY	CLIENT SAMPLE ID	MATRIX	COLLECTED	RECEIVED	LAB SAMPLE ID	DEPARTMENT SAMPLE ID
529	WC-N0264	WM60062	WATER	02/22/91		N103236-01	529-001
529	WC-N0264	WM60063	WATER	02/22/91		N103236-02	529-002
529	WC-N0264	WM60064	WATER	02/22/91		N103236-03	529-003
529	WC-N0265	WM00957	WATER	03/19/91		N103236-06	529-006
529	WC-N0265	WM00957	Filtered WATER	03/19/91		N103236-07	529-007
529	WC-N0265	WM50308	WATER	03/19/91		N103236-04	529-004
529	WC-N0265	WM50308	Filtered WATER	03/19/91		N103236-05	529-005
529	WC-N0266	WM50306	WATER	03/18/91		N103236-08	529-008
529	WC-N0266	WM50306	Filtered WATER	03/18/91		N103236-09	529-009
529	WC-N0266	WM50307	WATER	03/18/91		N103236-10	529-010
529	WC-N0266	WM50307	Filtered WATER	03/18/91		N103236-11	529-011
529	WC-N0267	WM50309	WATER	03/19/91		N103236-12	529-012
529	WC-N0268	WM60065	WATER	03/19/91		N103236-13	529-013
529	WC-N0268	WM60066	WATER	03/19/91		N103236-14	529-014
529	WC-N0268	WM60067	WATER	03/19/91		N103236-15	529-015
529	WC-N0269	WM50311	WATER	03/26/91		N103236-18	529-018
529	WC-N0269	WM50312	Filtered WATER	03/26/91		N103236-16	529-016
529	WC-N0269	WM50313	Filtered WATER	03/26/91		N103236-17	529-017
529	WC-N0270	WM50310	Filtered WATER	03/25/91		N103236-19	529-019
529	WC-N0271	WM50314	Filtered WATER	04/01/91		N103236-20	529-020
529	WC-N0271	WM50315	Filtered WATER	04/01/91		N103236-21	529-021
529	WC-N0272	WM50316	WATER	04/02/91		N103236-22	529-022
529	WC-N0272	WM50317	Filtered WATER	04/02/91		N103236-23	529-023
529		Reagent Blank	WATER			N103236-28	529-028
529		Low LCS	WATER			N103236-26	529-026
529		High LCS	WATER			N103236-27	529-027
529		Replicate (N103236-08)	WATER	03/18/91		N103236-24	529-024
529		Replicate (N103236-20)	Filtered WATER	04/01/91		N103236-25	529-025

Lab id TMAN
Protocol GRRASP
Version Ver 1.0
Form DVD-QS
Version 01/22/92
Report date 01/27/92

Fig. 4. The Laboratory Control Sample report displays all data that is necessary to evaluate the acceptability of a laboratory control sample. Fields on this report that can have underlined values include Results, Err, MDA and Added.

THERMO ANALYTICAL
 EXAMPLE REPORTING GROUP
RESULT SUMMARY
 ROCKY FLATS, WATER, URANIUM
 ALPHA SPECTROSCOPY

Test U _____
 SDG RF041 _____
 Contact Julie Wose _____

Client Company _____
 Contract DS-JK-L4 _____

CLIENT SAMPLE ID	LAB SAMPLE ID	SUF- DEPARTMENT FIX SAMPLE ID	1: Uranium		2: Uranium		3: Uranium		RESULT RATIOS (%)			
			233/234		235		238		1+3	2σ	2+3	2σ
Preparation batch 2654-074												
WM00957	N103236-06	529-006	0.90		U		0.97		93	76	0	17
WM00957 Filtered	N103236-07	529-007	1.3		U		0.73		178	121	0	16
WM50306	N103236-08	529-008	U		U		1.7		<u>26</u>	32	16	19
WM50306 Filtered	N103236-09	529-009	1.4		U		1.5		93	53	0	9
WM50307	N103236-10	529-010	0.92		U		0.68		135	100	0	18
WM50307 Filtered	N103236-11	529-011	0.84		U		1.1		76	79	0	18
WM50308	N103236-04	529-004	0.93		U		0.72		129	88	0	14
WM50308 Filtered	N103236-05	529-005	0.80		U		0.93		86	72	0	16
WM50309	N103236-12	529-012	1.1		U		1.6		69	43	5	10
WM50310 Filtered	N103236-19	529-019	0.91		U		0.61		149	124	12	26
WM50311	N103236-18	529-018	2.2		U		0.96		229	146	8	16
WM50312 Filtered	N103236-16	529-016	2.1		U		2.3		91	45	11	12
WM50313 Filtered	N103236-17	529-017	1.6		U		2.1		76	40	6	7
WM50314 Filtered	N103236-20	529-020	1.3		U		0.79		165	108	0	15
WM50315 Filtered	N103236-21	529-021	2.0		U		1.7		118	64	0	8
WM50316	N103236-22	529-022	1.3		U		1.7		76	46	8	8
WM50317 Filtered	N103236-23	529-023	2.5		U		2.1		119	58	10	13
Reagent Blank	N103236-28		U	X	U	X	U	X				
Low LCS	N103236-26		ok	X	No data	X	ok	X	83	38	9	8
High LCS	N103236-27		ok	X	ok	X	ok	X	96	31	5	3
Replicate (N103236-08)	N103236-24		ok	X	ok	UX	ok	X	100	67	0	11
Replicate (N103236-20)	N103236-25		OUT	X2	ok	UX2	OUT	X2	103	56	6	6
Nominal values and limits from method			RDLs (pCi/L)		0.60	0.60	0.60		100		4	
2σ non-counting method error 5.0 %									Averages 111		4	

Lab id TMAN _____
 Protocol GRRASP _____
 Version Ver 1.0 _____
 Form DVD-CRS _____
 Version 01/22/92 _____
 Report date 01/27/92 _____

Fig. 5. The Results Summary provides the results of one test for all samples and QC samples in the batch, flagging non-compliant data. This allows the reviewer to look for trends in the data and to identify samples associated with QC problems. In this case the ratio of the uranium isotopes values are compared with user defined ratios (eg. natural ratios); abnormal values are underlined. The system uses a variety of CLP compliant flags, such as the "U" for not detected and "X" for manually entered data.

THERMO ANALYTICAL
 EXAMPLE REPORTING GROUP
METHOD SUMMARY
 ROCKY FLATS, WATER, URANIUM
 ALPHA SPECTROSCOPY

Test U _____
 SDG RFO41 _____
 Contact Julie Wose _____

Client Company _____
 Contract DS-JK-LL _____

CLIENT SAMPLE ID	LAB SAMPLE ID	SUF- DEPARTMENT FIX SAMPLE ID	MAX MDA pCi/L	ALIQOT L	RESID mg	YIELD %	COUNT min	FWHM KeV	DAYS HELD	PREPARED	ANAL- YZED	DETECTOR
Preparation batch 2654-074												
WM00957	N103236-06	529-006	0.36	0.50		41			43	05/01/91		ASPEC
WM00957 Filtered	N103236-07	529-007	0.27	0.50		58			43	05/01/91		ASPEC
WM50306	N103236-08	529-008	0.64	0.50		24			44	05/01/91		ASPEC
WM50306 Filtered	N103236-09	529-009	0.30	0.50		50			44	05/01/91		ASPEC
WM50307	N103236-10	529-010	0.27	0.50		55			44	05/01/91		ASPEC
WM50307 Filtered	N103236-11	529-011	0.56	0.50		35			44	05/01/91		ASPEC
WM50308	N103236-04	529-004	0.28	0.50		63			43	05/01/91		ASPEC
WM50308 Filtered	N103236-05	529-005	0.35	0.50		41			43	05/01/91		ASPEC
WM50309	N103236-12	529-012	0.34	0.50		47			43	05/01/91		ASPEC
WM50310 Filtered	N103236-19	529-019	0.34	0.50		50			37	05/01/91		ASPEC
WM50311	N103236-18	529-018	0.34	0.50		51			36	05/01/91		ASPEC
WM50312 Filtered	N103236-16	529-016	0.30	0.50		55			36	05/01/91		ASPEC
WM50313 Filtered	N103236-17	529-017	0.29	0.50		61			36	05/01/91		ASPEC
WM50314 Filtered	N103236-20	529-020	0.28	0.50		66			30	05/01/91		ASPEC
WM50315 Filtered	N103236-21	529-021	0.35	0.50		60			30	05/01/91		ASPEC
WM50316	N103236-22	529-022	0.31	0.50		57			29	05/01/91		ASPEC
WM50317 Filtered	N103236-23	529-023	0.31	0.50		59			29	05/01/91		ASPEC
Reagent Blank	N103236-28		0.20	0.50		36				05/01/91		ASPEC
Low LCS	N103236-26		0.80	0.50		26				05/01/91		ASPEC
High LCS	N103236-27		0.80	0.50		28				05/01/91		ASPEC
Replicate (N103236-08)	N103236-24		0.40	0.50		56			44	05/01/91		ASPEC
Replicate (N103236-20)	N103236-25		0.60	0.50		29			30	05/01/91		ASPEC
Nominal values and limits from method			0.60	0.50	-	30-105	100	100	180			

PROCEDURES EP-1 Environmental water preparation
 EP-70 Uranium separation -- Chloride Column
 EP-71 Uranium purification -- Ether Extraction
 EP-5 Actinide electrodeposition

AVERAGES ± 2 SD MDA 0.40 ± 0.36
 YIELD 48 ± 26
 FWHM _____ ± _____

Lab id TMAN
 Protocol GRRASP
 Version Ver 1.0
 Form DVD-CMS
 Version 01/22/92
 Report date 01/27/92

Fig. 6. The Method Summary shows parameters related to the preparation, chemistry, counting for one test for all samples. Most parameters are compared to protocol limits that are user entered and, as throughout the system, out-of-spec values are flagged. Procedures used for the analysis are listed on this summary. Full text for the procedures is available for review within the system.