Acceptance Criteria for Bioassay data HSR-12-DAC-02.01

November 12, 2002

1 General

Specifications for acceptable bioassay data to be provided by the bioassay services team of HSR-4 to the dose assessment team of HSR-12 are given in this document. These data are the results of radiochemical alpha spectroscopy (RAS), thermal ionization mass spectroscopy (TIMS), and liquid scintillation (LS) measurements.

Numerical (floating point) data shall have a precision of at least 1 in 1000 (*e.g.* 1.234e+10). Data shall be available in electronic form to Dose Assessment within 3 working days of the turn-around times specified in the Analytical Service Agreement (ASA) (that is, the due date plus 3 days). Because of the inevitability of error, hand entry of electronic data must be minimized.

Documentation of the analytical and quality assurance (QA) procedures used is also to be provided in electronic form identified by unique document numbers.

In Section 2 the required data field/components for each type of bioassay data are separately listed, giving the field/component #, name, a detailed description, datatype, and units. Primary (measured) and secondary (calculated) fields are distinguished by an "M" or "C" in the field #. Fields distinguished by as asterisk (*) are currently essential for basic dose assessments. Other fields provide more in-depth information that may be used in some dose assessment situations, are used for documentation, or are used to monitor quality.

Descriptions of outcome codes are given in Section 3.

2 Required data field/components

2.1 RAS and TIMS measurements

Measurements using RAS and TIMS have the field/components groups: kit data, planchette data, analysis identification (ID) data, as well as RAS and TIMS results.

2.1.1 Kit data

Kit data for RAS and TIMS shall consist of the field/components listed in Table 2. Specific Gravity is required only for urine analyses.

2.1.2 Planchette data

For samples analyzed using RAS or TIMS a chemical separation is performed and the analyte electrodeposited onto a stainless steel disk (planchette). Planchette data shall consist of the field/components listed in Table 2. Usually there is only one planchette produced from each kit, although multiple aliquots used to produce multiple planchetes may be important in the future (for example, with analysis for both plutonium americium). The excretion time period fields are required only for urine analyses.

Table 1: Kit data

#	field name (symbol)	What is it?	datatype	units
KM1*	Kit number	HSR kit number	integer	
KM2*	Person-Z	Z# of person submitting sample	text	
KM3*	Person-PID	PID# of person submitting sample	integer	
KM4	Group	Person's group at the time of the sample	text	
KM5*	Matrix	urine, feces, water	text	
KM6*	Kit type	true 24-hr, simulated 24-hr, spot sample, timed, 500-ml sample, home drinking water	text	
KM7	Schedule type	routine, prompt action, diagnostic, followup, baseline, external request, internal study, other (explained in comments)	text	
KM8	Scheduled date	date kit scheduled to be picked up by person filling kit	date	
KM9*	Collected date	date and time of sample collection	date/time	
KM10*	Mass (M)	mass of sample	number	gm
KM11*	Specific gravity (ρ)	specific gravity of urine sample. Urine sample specific gravity must exceed 1, which is the specific gravity of pure water. If $\rho < 1 + \sigma_\rho$, ρ is replaced by $1 + \sigma_\rho$ where σ_ρ is the uncertainty SD of the specific gravity measurement	number	none
KM12*	Uncertainty of specific gravity (σ_ρ)	uncertainty SD of specific gravity	number	none
KM13	Method description	documentation number	text	
KM14*	Outcome	unique code identifying possible outcomes (also referred to as problem code)	text	
KM15	Comments	additional comments	text	

Table 2: Planchette data

#	field name (symbol)	What is it?	datatype	units
PM1	Sample id	analysis lab sample number	integer	
PM2	Batch id	analysis lab batch number	integer	
PM3*	Mass analyzed (M_A)	mass analyzed	number	gm
PM4*	Analyte	chemical analyte: PU, U, AM	text	
PM5*	Tracer activity (A_T)	activity of tracer added to sample	number	dpm
PM6*	Tracer uncertainty (σ_T)	uncertainty SD of tracer activity	number	dpm
PM7	Method description	documentation number	text	
PM8*	Outcome	unique code identifying possible outcomes (also referred to as problem code)	text	
PM9	Comments	additional comments	text	
PC1*	Urine excretion time period (Δt_{ex})	calculated urine excretion time period. If kit type is true-24hr, $\Delta t_{ex} = 1\,\text{day} \times M_A/M$; otherwise $\Delta t_{ex} = 1\,\text{day} \times \frac{M_A(\rho-1)}{C}$, where C is a constant of the excretion time calculation, normally equal to 1440 gm (the nominal mass excreted per day) $\times$ 0.02 (the nominal excess specific gravity of urine). The measured sample activity A in urine is normalized to produce urine excretion rate using the formula $\dot{A} = A/\Delta t_{ex}$	number	days
PC2*	Urine excretion time uncertainty ($\sigma_{\Delta t_{ex}}$)	uncertainty SD of calculated excretion time period. If kit type is true-24hr, $\sigma_{\Delta t_{ex}} = 0$; otherwise $\sigma_{\Delta t_{ex}} = \Delta t_{ex}\sqrt{(\frac{\sigma_\rho}{\rho})^2 + (\frac{\sigma_C}{C})^2}$. The uncertainty SD of urine excretion rate is given by $\sigma_{\dot{A}} = \frac{\sigma_A}{\Delta t_{ex}}\sqrt{1 + (\frac{\sigma_{\Delta t_{ex}} A}{\Delta t_{ex}\sigma_A})^2}$, where σ_A is the uncertainty SD of sample activity	number	days

2.1.3 Analyses

Analysis data shall consist of the field/components listed in Table 3. Multiple analyses may be performed on the same planchette, for example, RAS and TIMS, or RAS followed by a second RAS with a longer count time.

Table 3: Analysis data

#	field name (symbol)	What is it?	datatype	units
AM1	Priority	analysis priority: normal (3) or expedited (1)	number	
AM2	Requested date	date of request for analysis	date	
AM3	Due date	the date sample results are due according to the ASA	date	
AM4	Completion date	Date that results become available in the dose assessment database	date	
AM5	Method description	documentation number	text	
AM6*	Outcome	unique code identifying possible outcomes (also referred to as problem code)	text	
AM7	Comments	additional comments	text	
AM8	Technique	analysis technique: RAS or TIMS	text	
AM9	Analyst	Z# of person approving results	text	
AM10	QA'd by	Z# of person who QA'd the data	text	
AM11	QA documentation	document number of QA procedure	text	

2.1.4 RAS results

Radiochemical alpha spectroscopy (RAS) measurements using a tracer detect counts in the tracer window of the alpha energy spectrum and at least one isotope energy window. Tracers and isotopes for various analytes are shown in Table 4. Field/components associated with the tracer and the various isotopes are described in Tables 5 and 6 below.

Table 4: RAS tracers and isotopes

analyte	tracer	isotopes
PU	PU-242	PU-239, PU-238
AM	AM-243	AM-241
U	U-232	U-234, U-235, U-238

Table 5: RAS data

#	field name (symbol)	What is it?	datatype	units
RTM1*	Count time (Δt)	sample count time	number	minutes
RTM2*	Background count time ratio (R_B)	background count time divided by sample count time	number	none
RTM3	Detector number (i)	detector number	text	
RTM4*	Tracer Gross counts (G_T)	tracer gross counts for detector i	number	counts
RTM5*	Tracer background counts (B_T)	average tracer background counts for detector i in time Δt (background counts are measured in time period $R_B \Delta t$)	number	counts
RTM6*	Detector efficiency (ϵ)	efficiency of detector i	number	fraction
RTC1*	Tracer recovery (R)	per cent recovery $R = 100\,\frac{G_T - B_T}{A_T \epsilon \Delta t}$	number	%
RTC2*	Tracer background uncertainty (σ_{B_T})	uncertainty SD of tracer background, $\sigma_{B_T} = \sqrt{B_T / R_B}$	number	counts
RTC3*	f factor (f)	f-factor relating activity units to counts, $f = \frac{1}{2.22}\frac{A_T}{G_T - B_T} f_A$. The quantity f_A, the acceptance factor, corrects for loss of counts from the acceptance region of the α-energy spectrum. For PU-239, PU-238, AM-241, U-234, and U-238 the acceptance factor is 1, and for U-235 it is 1.11.	number	pCi/count
RTC4*	f factor uncertainty (σ_f)	uncertainty SD of f, $\sigma_f = \lvert f \rvert \sqrt{\left(\frac{\sigma_T}{A_T}\right)^2 + \frac{G_T + \sigma_{B_T}^2}{(G_T - B_T)^2}}$	number	pCi/count

Table 6: RAS results–isotope specific

#	field name (symbol)	What is it?	datatype	units
RIM1*	Isotope	Isotope corresponding to α-energy spectral window. Isotopes are shown in Table 4.	text	
RIM2*	Isotope gross counts (G)	gross counts	integer	counts
RIM3*	Isotope background counts (B)	average background counts in time Δt (background counts are measured in time period $R_B \Delta t$)	number	counts
RIM4*	Tracer contamination (f_{TC})	fractional tracer contamination	number	none
RIM5*	Tracer contamination uncertainty ($\sigma_{f_{TC}}$)	uncertainty SD of f_{TC}	number	none
RIC1*	Isotope background uncertainty (σ_B)	uncertainty SD of average background, normally $\sigma_B = \sqrt{B/R_B}$ with B replaced by 1 count if $B = 0$	number	counts
RIC2*	Isotope activity (A)	activity in sample, $A = f(G - B) - f_{TC}A_T$	number	pCi
RIC3*	Isotope activity uncertainty (σ)	uncertainty SD of sample activity $\sigma = \sqrt{f^2(G + \sigma_B^2) + \sigma_f^2(G - B)^2 + A_T^2\sigma_{f_{TC}}^2 + f_{TC}^2\sigma_{A_T}^2}$	number	pCi
RIC4	Isotope MDA (MDA)	$MDA = 3.29\sigma_0 + 3f$ where $\sigma_0 = \sqrt{f^2(B + \sigma_B^2) + A_T^2\sigma_{f_{TC}}^2 + f_{TC}^2\sigma_{A_T}^2}$ is the uncertainty SD of blank	number	pCi

2.1.5 TIMS results

Samples analyzed using TIMS shall have the field/components listed in Tables 7 and 8. Table 7 contains measured fields and Table 8 contains calculated fields.

Table 7: TIMS data–measured fields

#	field name (symbol)	What is it?	datatype	units
TM1	PU-239 (A_{239})	activity of PU-239 in sample, given by $2.4622\ 10^{-11} \times N_{239}$, where N_{239} is the measured number of atoms of PU-239 in the sample	number	pCi
TM2	PU-239 uncertainty (σ_{239})	uncertainty SD of PU-239 activity	number	pCi
TM3	PU-240 (A_{240})	activity of PU-240 in sample, given if PU-240 is measurable by $9.0440\ 10^{-11} \times N_{240}$, where N_{240} is the measured "number of atoms of PU-240 in the sample, blank otherwise.	number	pCi
TM4	PU-240 uncertainty (σ_{240})	uncertainty SD of PU-240 activity, if measurable, blank otherwise.	number	pCi
TM5	Number of blank measurements for PU-239 (M_{239})	Number of PU-239 blank measurements used in making blank correction	integer	none
TM6	Average value of blank for PU-239 (B_{239})	Average of PU-239 blank measurements in pCi, $B_{239} = \sum B_i / M_{239}$, where B_i is the i^{th} blank measurement, for $i = 1, M_{239}$	number	pCi
TM7	Uncertainty of blank for PU-239 ($\sigma_{B_{239}}$)	Standard deviation of PU-239 blank measurements, $\sigma_{B_{239}} = \sqrt{\frac{\sum (B_i - B_{239})^2}{M_{239}-1}}$. The MDA for PU-239 is given by $MDA_{239} = 3.29\,\sigma_{B_{239}}$	number	pCi
TM8	Matrix code for PU-239	Unique code identifying the matrix for the PU-239 blank measurements, for example, U30 stands for 30 urine blanks (the rest of the M_{239} blanks, if any, being water)	text	
TM9	Number of blank measurements for PU-240 (M_{240})	Number of PU-240 blank measurements used in making blank correction	integer	none
TM10	Average value of blank for PU-240 (B_{240})	Average of PU-240 blank measurements, $B_{240} = \sum B_i / M_{240}$, where B_i is the i^{th} blank measurement, for $i = 1, M_{240}$	number	pCi
TM11	Uncertainty of blank for PU-240 ($\sigma_{B_{240}}$)	Standard deviation of PU-240 blank measurements, $\sigma_{B_{240}} = \sqrt{\frac{\sum (B_i - B_{240})^2}{M_{240}-1}}$. The MDA for PU-240 is given by $MDA_{240} = 3.29\,\sigma_{B_{240}}$. The MDA for PU-239 plus PU-240 is $MDA_{239+240} = \sqrt{MDA_{239}^2 + MDA_{240}^2}$ if PU-240 is measurable. If PU-240 is not measurable, $MDA_{239+240} = 1.221\,MDA_{239}$	number	pCi
TM12	Matrix code for PU-240	Unique code identifying the matrix for the PU-240 blank measurements, for example, U30 stands for 30 urine blanks (the rest of the M_{240} blanks, if any, being water)	text	

Table 8: TIMS data–calculated fields

#	field name (symbol)	What is it?	datatype	units
TC1	PU-239+PU-240 ($A_{239+240}$)	activity of PU-239 plus PU-240, $A_{239+240} = A_{239} + A_{240}$. If PU-240 is not measurable, a PU-240/239 atom ratio of 0.06 ± 0.01 is assumed, and $A_{239+240} = 1.221A_{239}$	number	pCi
TC2	PU-239+PU-240 uncertainty ($\sigma_{239+240}$)	uncertainty SD of PU-239 plus PU-240 activity, $\sigma_{239+240} = \sqrt{\sigma_{239}^2 + \sigma_{240}^2}$. If PU-240 is not measurable, $\sigma_{239+240}$ is calculated assuming a PU-240/PU-239 atom ratio of 0.06 ± 0.01, $\sigma_{239+240} = \sqrt{(1.221\sigma_{239})^2 + (0.0368A_{239})^2}$	number	pCi
TC3	PU-240/239 AR (r)	atom ratio PU-240/PU-239 $r = N_{240}/N_{239}$, blank otherwise	number	atom ratio
TC4	PU-240/239 AR uncertainty (σ_r)	if PU-240 is measurable, uncertainty SD of atom ratio PU-240/PU-239, $\sigma_r = \lvert r\rvert\sqrt{(\sigma_{239}/A_{239})^2 + (\sigma_{240}/A_{240})^2}$, blank otherwise	number	atom ratio
TC5	Blank-corrected PU-239 ($\hat{A}_{239}$)	blank-corrected activity of PU-239 in sample in pCi, $\hat{A}_{239} = A_{239} - B_{239}$	number	pCi
TC6	Blank-corrected PU-239 uncertainty ($\hat{\sigma}_{239}$)	blank-corrected uncertainty SD of PU-239 activity, $\hat{\sigma}_{239} = \sqrt{\sigma_{239}^2 + \sigma_{B_{239}}^2}$	number	pCi
TC7	Blank-corrected PU-240 ($\hat{A}_{240}$)	blank-corrected activity of PU-240 in sample in pCi, $\hat{A}_{240} = A_{240} - B_{240}$	number	pCi
TC8	Blank-corrected PU-240 uncertainty ($\hat{\sigma}_{240}$)	blank-corrected uncertainty SD of PU-240 activity in pCi, $\hat{\sigma}_{240} = \sqrt{\sigma_{240}^2 + \sigma_{B_{240}}^2}$	number	pCi
TC9*	Blank-corrected PU-239+PU-240 ($\hat{A}_{239+240}$)	blank-corrected activity of PU-239 plus activity of PU-240 in sample in pCi, $\hat{A}_{239+240} = A_{239+240} - B_{239} - B_{240}$. If PU-240 is not measurable, $\hat{A}_{239+240}$ is calculated assuming a PU-240/PU-239 atom ratio of 0.06 ± 0.01, $\hat{A}_{239+240} = 1.221\hat{A}_{239}$	number	pCi
TC10*	Blank-corrected PU-239+PU-240 uncertainty ($\hat{\sigma}_{239+240}$)	blank-corrected uncertainty SD of PU-239 plus PU-240 activity, $\hat{\sigma}_{239+240} = \sqrt{\sigma_{239+240}^2 + \sigma_{B_{239}}^2 + \sigma_{B_{240}}^2}$. If PU-240 is not measurable, $\hat{\sigma}_{239+240}$ is calculated assuming a PU-240/PU-239 atom ratio of 0.06 ± 0.01, $\hat{\sigma}_{239+240} = \sqrt{(1.221\hat{\sigma}_{239})^2 + (0.0368\hat{A}_{239})^2}$	number	pCi
TC11	Blank-corrected PU-240/239 AR ($\hat{r}$)	blank-corrected atom ratio PU-240/PU-239, $\hat{r} = \frac{\hat{A}_{240}}{\hat{A}_{239}} \times \frac{2.0714\times10^{11}\text{sec}}{7.6084\times10^{11}\text{sec}}$	number	atom ratio
TC12	Blank-corrected PU-240/239 AR uncertainty ($\hat{\sigma}_r$)	blank-corrected uncertainty SD of atom ratio PU-240/PU-239, $\hat{\sigma}_r = \lvert\hat{r}\rvert\sqrt{(\hat{\sigma}_{239}/\hat{A}_{239})^2 + (\hat{\sigma}_{240}/\hat{A}_{240})^2}$	number	atom ratio

2.2 LS data

Samples analyzed using LS shall contain the field/components listed in Table 9.

Table 9: LS data

#	field name (symbol)	What is it?	datatype	units
LM1	Kit number	HSR kit number	integer	
LM2*	Person-Z	Z# of person submitting sample	text	
LM3*	Person-PID	PID# of person submitting sample	integer	
LM4	Group	Person's group at the time of the sample	text	
LM5*	Matrix	urine, water	text	
LM6*	Kit type	true 24-hr, simulated 24-hr, spot sample, timed, 500-ml sample, home drinking water	text	
LM7	Schedule type	routine, prompt action, diagnostic, followup, baseline, external request, internal study, other (explained in comments)	text	
LM8	Scheduled date	date kit scheduled to be picked up by person filling kit	date	
LM9*	Collected date	date and time of sample collection	date/time	
LM10	Completion date	Date that results become available in the dose assessment database	date	
LM11	Method description	documentation number	text	
LM12*	Outcome	unique code identifying possible outcomes	text	
LM13	Comments	additional comments	text	
LM14	Analyst	Z# of person approving results	text	
LM15	QA'd by	Z# of person who QA'd the data	text	
LM16	QA documentation	document number of QA procedure	text	
LM17*	Aliquot volume (V)	volume of measured aliquot	number	mL
LM18*	Count time (Δt)	count time	number	minutes
LM19*	Efficiency (ϵ)	detector efficiency	number	none
LM20*	Efficiency uncertainty (σ_ϵ)	uncertainty SD of ϵ	number	none
LM21*	Gross counts (G)	gross counts	number	counts
LM22*	Background counts (B)	average background counts	number	counts
LC1*	f factor (f)	$f = \frac{1\times10^{-3}}{2.22}\frac{1}{V\epsilon\Delta t}$	number	uCi/(L-count)
LC2*	f factor uncertainty (σ_f)	$\sigma_f = \lvert f\rvert\frac{\sigma_\epsilon}{\epsilon}$	number	uCi/(L-count)
LC3*	Background uncertainty (σ_B)	uncertainty SD of average background (depends on procedure)	number	counts
LC4*	Sample activity (A)	sample activity in μCi/L, $A = f(G-B)$	number	uCi/L
LC5*	Sample uncertainty (σ_A)	uncertainty SD of sample activity $\sigma_A = \sqrt{f^2(G+\sigma_B^2)+\sigma_f^2(G-B)^2}$	number	uCi/L
LC6	MDA (MDA)	$MDA = 3.29\sigma_0 + 3f$ where $\sigma_0 = f\sqrt{B+\sigma_B^2}$ is the uncertainty SD of blank	number	uCi/L

3 Outcome Codes

Outcome codes are associated with each bioassay task: KIT, PLANCHETTE, RAS, TIMS, and LS. The outcome codes shown in Table 25 and 26 shall be used. The outcome code shall not be left blank; if no problems occur, the outcome code "OK" is used.

Table 25: Outcome codes for KIT and PLANCHETTE

task	code	What does it mean?
KIT	OK	no problems. Results reported.
	LIA	lost in analysis–the sample was lost and not available for complete analysis. No results reported.
	ABORT	sample analysis aborted by customer request. No results reported.
	ISV	insufficient volume for analysis–the sample volume was less than required, and water was added to the required volume level. Specific gravity and mass reported after addition of water.
	NSS	No sample in container. No results reported.
	OTHER	an outcome that doesn't fit into the above categories. Outcome description in comments field
PLANCHETTE	OK	no problems. Results reported.
	LIA	lost in analysis–the sample was lost and not available for complete analysis. No results reported.
	ABORT	sample analysis aborted by customer request. No results reported.
	OTHER	an outcome that doesn't fit into the above categories. Outcome description in comments field

Table 26: Outcome codes for RAS, TIMS, and LS

task	code	What does it mean?
RAS	OK	no problems. Results reported.
	PR	poor recovery-less than 40 % tracer recovery but greater than 15 % tracer recovery. Will continue for TIMS analysis (for plutonium only). Results reported.
	LR	low recovery-less than 15 % tracer recovery. Results reported.
	LIA	lost in analysis–the sample was lost and not available for complete analysis. No results reported.
	ABORT	sample analysis aborted by customer request. No results reported.
	PS	poor spectra–the spectral output was of poor quality, and the results are suspect. Results are reported.
	OTHER	an outcome that doesn't fit into the above categories. Outcome description in comments field
TIMS	OK	no problems. Results reported.
	LIA	lost in analysis–the sample was lost and not available for complete analysis. No results reported.
	ABORT	sample analysis aborted by customer request. No results reported
	TNN	"TIMS Not Needed", TIMS measurement not carried out because RAS result/uncertainty > 5. RAS measurement by itself is sufficient
	PR	poor recovery-less than 40 % tracer recovery but greater than 15 % tracer recovery. Results reported
	PC	poor mass-spec count rate. Results reported
	OTHER	an outcome that doesn't fit into the above categories. Outcome description in comments field
LS	OK	no problems. Results reported.
	LIA	lost in analysis–the sample was lost and not available for complete analysis. No results reported
	ABORT	sample analysis aborted by customer request. No results reported
	ISV	insufficient volume for analysis–the sample volume was less than required, and water was added to the required volume level. Results reported and actual sample volume recorded
	OTHER	an outcome that doesn't fit into the above categories. Outcome description in comments field

4 Signatures

Dawn Lewis (bioassay services)	date
Ray Guilmette (dose assessment)	date