

FUNCTION:	SYSTEM:		UNIT:	B.S.I.:	
ANALYTICAL PROCEDURE	CHEMISTRY LABORAT	ORY	1	78	3200
		0111	-	,	200
TITLE:		PROCEDURE NO	D.:		REV.:
GADOLINIUM BY ARSENAZO III		78200)-AP-GA	.1	12
		,0200		••	

POINT LEPREAU GENERATING STATION

CHEMISTRY LABORATORY ANALYTICAL PROCEDURE

GADOLINIUM ARSENAZO III

78200-AP-GA1

Issued By: _____ Date: _____

PREPARED BY:	DATE:	REVIEWED BY:	DATE:	APPROVED BY:	BATE:	PASE:		
K. MacGibbon	02-08-14	R. Culligan		G.H. Brown		1	of	16



Point Lepreau G.S.						
FUNCTION:	SYSTEM:			UNIT:	B.S.I.:	
ANALYTICAL PROCEDURE		CHEMISTRY LABORATOR	Y	1	78	200
TITLE:			PROCEDURE NO	.:		REV.:
GADOLINIUM BY ARSENAZO III			78200	-AP-GA	.1	12

REVISION RECORD

AUTHOR	REV	DESCRIPTION	DATE
	0	Initial Issue	
W. Mawhinney	1	Not Tracked	Oct./91
W. T. Underhill	2	 Revised to incorporate 3 point calibration. Quality Control Standard verification and calibration linearity checks added. Added tables for sample dilutions. 	July/97
		 Added precision statistics for aliquots between 0.200 and 0.300 mg Gd/L H₂O. Added detection limits based on INPO criteria. Incorporated use of Quality Control Report Form. 	
W. T. Underhill	3	 Added requirement to verify calibration standards and Quality Control standards per 1-78200-QC-01. Changed volume of ARSENAZO III solution from 1.0 ml to 2.0 ml. Changed dilution factor for Moderator Poison tank samples. Added precision statistics for aliquots between 0.100 and 0.200 mg Gd/L H₂O. 	Aug./97
W. T. Underhill	4	 Changed calibration standards to 0.100, 0.200 and 0.300 mg GD/L H₂O. Added computer calculation procedure and report form. Revised result reporting criteria (Sect. 5.2#2). Changed QC Check Standard from 9000 to 10000 mg Gd/L H₂O. Revised precision statistics and detection limits. Added references. 	Oct./97
W. MacKeigan	5	 Added references to use of new Varian Cary 1C UV/VIS Spectrophotometer. Deleted manual calculation section. Added requirement to cool standards to 25°C. Removed requirement to zero the Reagent Blank Absorbance. Deleted upper limit on calibration slope. Added Revision Record 	April/98

PREPARED BY:	DATE:	REVIEWED BY:	DATE:	ABBROVED BY	BATE:	BARE:		
K. MacGibbon	02-08-14	R. Culligan		G.H. Brown	Brite.	2	of	16



 Point Lepreau G.S.
 SYSTEM:
 UNIT:
 B.S.I.:

 ANALYTICAL PROCEDURE
 CHEMISTRY LABORATORY
 1
 78200

 TITLE:
 GADOLINIUM BY ARSENAZO III
 PROCEDURE NO.:
 REV.:

 12
 12

<u>REVISION RECORD</u> (Cont'd)

AUTHOR	REV	DESCRIPTION	DATE
K. MacGibbon	6	 Changed mgGd/Kg to mgGd/Kg. Section 4.0 – Deleted reference to DMS200 and added reference to CARY50. Section 8.0 – Added reference to the w:\Chemical\Forms directory for location of report form. Section 10.0 – Added note to divide LLD and MDL by 1.105 to account for density of D₂O. Added shelf life of 1 month for arsenazo reagent. 	Oct/99
K. MacGibbon	7	 Section 2.0 made reference to control charts. Section 5.2 - added requirement to verify prepared standards if control charts are not used for validating calibration. Section 6.0 - Removed preparation steps for low concentration standards, removed requirement to verify prepared standards. Section 7.1 - Added requirement to prepare working calibration and Q.C. check standard. Changed Q.C. check standard dilution factor from 40000 to 50000. Section 7.2 - Deleted requirement to repeat analysis if sample absorbance is less than 0.200 ppm standard. Section 8.1.3 made reference to control chart, deleted reference to report form limits. Appendix 1 deleted lower and upper limits on report form 	Mar/00
K. MacGibbon	8	 Section 6 – Minor changes to Arsenazo reagent preparation. 	Feb/01
K. MacGibbon	9	 Section 7.2 - Changed step 4 to zero spectrometer on the blank, not nanopure water. Changed step 5 to use standard #1 as the first standard in the calibration set. Changed sample stabilization time to 10 minutes from 5 minutes. Section 7.2.2 - Changed step 4 to zero spectrometer on the blank, not nanopure water. Changed step 5 to use standard #1 as the first standard in the calibration set. Changed standard #1 as the first standard in the calibration set. Changed sample stabilization time to 10 minutes from 5 minutes. 	Apr/01



NALYTICAL PRC	CEDUI	RE	SYSTEM: CHEMISTRY LABORATORY	7	UNIT: 1	в.s.i.: 78	3200
E: ADOLINIUM BY	ARSEN	AZO II	I	PROCEDURE NO 78200	-AP-GA	1	REV.: 12
	10		<u>REVISION RECORD</u> (Cont'd)				
K. MacGibbon	10	- Se ar: - Se 3 d - Se sp ind	ection 6.0 – Changed Step 3 to 0.05% not 0.1% senazo III solution and 1.0 g to 0.5 g of arsena ection 7.1, table 1 – added Std labels to flask n & 4. ection 7.2, step 5 – clarified step to indicate that ectrometer must be zeroed on the blank. Added dicate that blank value in not recorded.	6 of 120 III. 10 umbers 2, at ed note to	Ju	ne 02	
K. MacGibbon	11	- Se - Se	ection $7.2.2$ – renumbered to Section 7.3 . ection 7.3 – Changed Steps $4 - 7$.		Ju	ne 02	
K. MacGibbon	12	- Se	ection 7.2.1 – Changed Table 2 sample volume	e for	Au	ıg. 02	

PREPARED BY: DATE: REVIEW	WED BY: DATE:	APPROVED BY: DATE:	PAGE:		
K. MacGibbon 02-08-14 R. C	Culligan	G.H. Brown	4	of	16



FUNCTION: ANALYTICAL	PROCEDURE	SYSTEM: CHEMISTRY LABORATOR	RY	UNIT: 1	B.S.I.: 78	3200
TITLE: GADOLINIUM	BY ARSENAZO II	I	PROCEDURE NO)-AP-GA	1	REV.: 12
		INDEX				
1.0	INTRODUCTIO	N				
2.0	OUTLINE OF M	ETHOD				
3.0	INTERFERENC	ES				
4 0	APPARATUS					
5.0	PRECAUTIONS					
	5.1 Personnel 5.2 Procedura	Precautions Il Precautions				
6.0	REAGENTS					
7.0	PROCEDURE					
	7.1 Preparation7.2 Preparation7.3 Main Mode	on of Standards and QC Check Sample on & Analysis of Samples derator Samples (Less than 20 mgGd/Kg	g D ₂ O)			
8.0	CALCULATION	IS				
9.0	PRECISION					
10.0	METHOD DETE	ECTION LIMIT				
11.0	REFERENCES					
FORM – A	PPENDIX 1: REP-7	8200-AP-14.1				
PREPARED BY: K MacGibbon	DATE: REVIEWE	DBY: DATE: APPROVED BY: Iligan G H Brown		BATE: P#	NGE:	f 16



Point Lepreau	G.S.					
FUNCTION:		SYSTEM:		UNIT:	B.S.I.:	
ANALYTICA	L PROCEDURE	CHEMISTRY LABORATORY	Y	1	78	3200
TITLE:			PROCEDURE NO	0.:		REV.:
GADOLINIUN	M BY ARSENAZO III		78200	-AP-GA	.1	12
1.0	INTRODUCTIO	N				
1.0						
	Gadolinium is inj	ected as a solution of gadolinium nitrate i	into the Mo	oderator	Systen	n for
	reactivity control	and for reactor shutdown			-	
	reactivity control					

This procedure shall be used to determine the concentration of gadolinium in samples from the Main Moderator System, Moderator Auxiliaries, Moderator D2O cleanup and special safety system SDS#2 (LISS). It will also be used for the verification of gadolinium standards.

2.0 <u>OUTLINE OF METHOD</u>

Arsenazo III is a dye that forms a colored complex with gadolinium in an acidic solution at pH 3. This procedure outlines the method for analysis of these solutions using either the Varian Cary 50 UV/VIS or Varian Cary 1C UV/VIS. spectrophotometer. The optimum absorbance for analysis of solutions containing this complex occurs at a wavelength of 653 nanometers.

This procedure is linear up to an aliquot gadolinium concentration of 0.300 mgGd/Kg H₂O. Standard aliquots are prepared and analyzed at concentrations of 0.100 mgGd/Kg H₂O, 0.200 mgGd/Kg H₂O, and 0.300 mgGd/Kg H₂O. A linearity check of the standard absorbances is required. All samples analyzed must be diluted such that the sample absorbances are within the range of absorbances corresponding to the standards.

A Quality Control (Q.C.) check standard will be analyzed with each sample run. The result of the Q.C. check standard must be within control chart criteria limits for the process sample result(s) to be valid, refer to 78200-QM-01.

All absorbances and calculated values shall be recorded on report form #REP-78200-AP-14.1. An example of this form is included in Appendix I of this procedure.

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3.0 INTERFERI The reaction is when the pH is begins to hyd The following Aluminum, ir does not inter concentration 4.0 APPARATU 7829-EQ47: \frac{1}{7829-EQ50: \frac	D III ENCES is not very selective. All elements rea at which they are hydrolyzed is lower rolyze. g substances do not act as inhibitors in ron, sodium, potassium, magnesium, s fere at concentrations up to 1000 mg/ s. ² S Varian Cary 1C UV-Visible Spectroph Varian Cary 50 UV-Visible Spectroph w-thru (visible) Flasks, 100 mL, 500 mL, and 1000 ml pettes, 0.5 mL and 1 mL tetor (or equivalent) timere Filter paper Whetever 41 D	PROCEDURI 782 acting with arser than the pH at v n quantities up to sulfate, chloride 'kg. Nitrate does hotometer totometer L; Beaker, 250 r	nazo III act which gado o 200 mg/k and phospl s not interf	t as inhib olinium ag: hate. Bo fere in lo
 3.0 INTERFERI The reaction is when the pH begins to hyd The following Aluminum, in does not inter concentration 4.0 <u>APPARATU</u> 7829-EQ47: Y 7829-EQ47: Y 7829-EQ47: Y 7829-EQ50: Y Cell, 1 cm flo Volumetrics I Calibrated Pip Oxford Pipett Hot plate & s PC with Micr 5.0 <u>PRECAUTIO</u> 	ENCES is not very selective. All elements rea at which they are hydrolyzed is lower rolyze. g substances do not act as inhibitors in ron, sodium, potassium, magnesium, s fere at concentrations up to 1000 mg/ s. ² S Varian Cary 1C UV-Visible Spectroph Varian Cary 50 UV-Visible Spectroph w-thru (visible) Flasks, 100 mL, 500 mL, and 1000 ml pettes, 0.5 mL and 1 mL tetor (or equivalent)	acting with arser than the pH at v n quantities up to sulfate, chloride /kg. Nitrate does hotometer notometer L; Beaker, 250 r	nazo III act which gado o 200 mg/k and phospl s not interf	t as inhib olinium cg: hate. Bc ère in lo
 The reaction is when the pH is begins to hyd. The following Aluminum, in does not interconcentration 4.0 APPARATU 7829-EQ47: V 7829-EQ50: V Cell, 1 cm flove Volumetrics I Calibrated Pip Oxford Pipett Hot plate & s PC with Microsci Alexandre A	 is not very selective. All elements rea at which they are hydrolyzed is lower rolyze. g substances do not act as inhibitors in ron, sodium, potassium, magnesium, stere at concentrations up to 1000 mg/s.² S Varian Cary 1C UV-Visible Spectrophysical Cary 50 UV-Visible Spectrophysical Cary 50 UV-Visible Spectrophysical Cary 50 mL, and 1000 ml pettes, 0.5 mL and 1 mL tetor (or equivalent) 	acting with arsen t than the pH at v n quantities up to sulfate, chloride 'kg. Nitrate does hotometer totometer L; Beaker, 250 r	nazo III act which gado o 200 mg/k and phospl s not interf	t as inhit olinium (g: hate. Bo fere in lo
 The following Aluminum, in does not interconcentration 4.0 APPARATU 7829-EQ47: V 7829-EQ50: V Cell, 1 cm flo Volumetrics I Calibrated Pip Oxford Pipett Hot plate & s PC with Microsometrics 	g substances do not act as inhibitors in ron, sodium, potassium, magnesium, s fere at concentrations up to 1000 mg/ s. ² Varian Cary 1C UV-Visible Spectroph Varian Cary 50 UV-Visible Spectroph ow-thru (visible) Flasks, 100 mL, 500 mL, and 1000 ml pettes, 0.5 mL and 1 mL tetor (or equivalent)	n quantities up to sulfate, chloride /kg. Nitrate doe: hotometer notometer L; Beaker, 250 r	o 200 mg/k and phospl s not interf	kg: hate. Bo Pere in lo
 4.0 <u>APPARATU</u> 7829-EQ47: Y 7829-EQ50: Y Cell, 1 cm flo Volumetrics I Calibrated Pip Oxford Pipett Hot plate & s PC with Micr 5.0 <u>PRECAUTION</u>	Varian Cary 1C UV-Visible Spectrop Varian Cary 50 UV-Visible Spectroph ow-thru (visible) Flasks, 100 mL, 500 mL, and 1000 ml pettes, 0.5 mL and 1 mL tetor (or equivalent)	hotometer 10tometer L; Beaker, 250 r	nL	
 7829-EQ47: Y 7829-EQ50: Y Cell, 1 cm flo Volumetrics I Calibrated Pip Oxford Pipett Hot plate & s PC with Micr 5.0 <u>PRECAUTIO</u> 	Varian Cary 1C UV-Visible Spectroph Varian Cary 50 UV-Visible Spectroph ow-thru (visible) Flasks, 100 mL, 500 mL, and 1000 ml pettes, 0.5 mL and 1 mL tetor (or equivalent)	hotometer 10tometer L; Beaker, 250 r	nL	
5.0 <u>PRECAUTIO</u>	rosoft Excel as well as Report File (R	AP1401.xls) lo	aded	
	ONS			
5.1 <u>Personnel Pre</u>	ecautions			
1. Proces proces	ss samples will be highly tritiated. All dures must be followed.	l applicable radi	ation prote	ection
2. Conce	entrated nitric acid is used, appropriate	e chemical prote	ection is ree	quired.



Point Lepreau G.S.	5.								
FUNCTION: ANALYTICAL	PROCE	DURE	SYSTEM:	HEMISTRY LABOR	RATORY		UNIT: 1	^{в.ѕ.і.:} 78	200
TITLE: GADOLINIUM	BY ARS	SENAZO III	[PRO	78200	-AP-GA	A1	rev.: 12
5.2	Proce	edural Preca	utions		·				
	1.	The proce using sam H2O.	dure is linea ple aliquots	r to 0.300 mgGd/Kg in the range of 0.200	H2O. The mgGd/Kg	best pre H2O to	ecision i 0.300 n	s obtair ngGd/K	ned Lg
		<u>Do not re</u> absorbanc	port results e correspond	from aliquots with a ding to the 0.300 mg	absorbance v Gd/Kg H2C	values h) standa	ngher th rd.	an the	
	2.	Do not rep For results results as Detected"	port numeric s between th "Detected".	al results lower than e Lower Limit of De Results lower than t	the Method etection (LL the LLD sha	l Detect D) and all be qu	ion Lim the MD toted as	iit (MD) L, quoto "Not	L). e the
	3.	Care must transferred	re must be taken during standard preparation to ensure all gadolinium oxide is nsferred when using multiple flasks.						
	4.	Arsenazo may occur	III does not r between ba	dissolve totally. Ens tches as observed by	sure reagent / blank abso	is filter orbance.	ed. Sor	ne varia	ation
	5.	Always tra Pipetteing	ansfer standa . Do not pip	ard solutions from th bette any standard so	eir containe lution direct	ers into a tly from	a beaker	prior to rage bo	o ttle.
	6.	Calibratio control ch	n and Q.C. c arts are not u	check standards must used to validate calib	t be verified prations.	using 7	78200-Q	C-01 if	

PREPARED BY:	DATE:	REVIEWED BY:	DATE:	ABBROVED BY	BATE:	BARE:		
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Point Lepreau G.S.							r		
FUNCTION: ANALYTICAL P	ROCEDU	JRE	SYSTEM:	HEMISTRY	LABORATORY	ſ	UNIT: 1	в.s.i.: 78	3200
TITLE: GADOLINIUM E	BY ARSE	NAZO III	[PROCEDURE NO	-AP-GA	A1	REV.: 12
6.0	<u>REAG</u>	ENTS							
	1.	Gadoliniu Use a N.I.	im standar S.T. grade s	d solution (P) tandard (Mat	rimary Calibra t'l No. 0000170	tion), 100 8)	0 mgGd	l/KgH ₂	0
		Weigh out Add 150m cool to 25 is transfer mL using prepared.	t 1.1526 g of hL of 2 N HN °C. Careful red by rinsin nanopure wa	$f Gd_2O_3$ (drie NO ₃ . Heat an ly transfer to ng the beaker ater. Transfe	ed at 105°C for 2 ad stir until disso a 1000 mL volu thoroughly with r to a Nalgene b	2 hours) in blved (do n metric flas nanopure ottle and la	to a 250 not boil) k. Ensu water. abel with	mL be Allow are all s Dilute h the da	aker. w to olution to 1000 ate
		<u>Note</u> : Th	is standard	must have a	different lot #	than prim	ary Q.(C. stan	dard.
	2.	Gadoliniu Use a N.I. Weigh out Add 150m cool to 25 is transfer mL using prepared.	Im standard S.T. grade s t 11.526 g of the of 2 N HP °C. Careful red by rinsin nanopure wa	d solution (P) tandard (Mat f Gd_2O_3 (drie NO_3 . Heat an ly transfer to ng the beaker ater. Transfer	rimary Q.C.), 1 terial No. 00001 OR ed at 105°C for 2 id stir until disso a 1000 mL volu thoroughly with r to a Nalgene b	2 hours) in 2 hours) in olved (do n umetric flass nanopure ottle and la	Gd/KgH to a 250 not boil) k. Ensu water. abel with	$f_2 O$ mL be Main ML be Main ML be much all s Dilute the h the data	aker. w to solution to 1000 ate
		<u>Note</u> : Th sta	is standard Indard.	must have a	different lot #	than prim	ary cal	ibratio	n
	3.	Arsenazo nanopure as 1 montl	III solution water. Filten h.	a, 0.05 % - D r solution thro	issolve 0.5 g of ough Whatman	arsenazo I 41 filter pa	II in 100 per. Re	0 mL c cord sh	of Ielf life
	4.	Buffer sol	lution - Diss water. Add	solve 10.21 g 223 mL of 0.	of potassium hy 1 N HCl.	drogen ph	thalate i	n 500 r	nL of
	5.	Nitric aci	d, 2 N - Dilı	ate 126 mL of	f ACS Grade Co	onc. HNO ₃	to 1 L.		
	6.	Hydrochl	oric acid, 0.	1 N - Dilute	8.2 mL of ACS	Grade Cor	ic. HCl 1	to 1 L.	
DEDADE: TW		. 	\ 	F. & T				ACE.	
K. MacGibbon	02-08-14	R. Cul	lligan	DATE:	APPROVED BY: G.H. Brown		BATE: P	age: 9 01	f 16



Point Lepre	eau G.S.		SYSTEM:			UNIT:	B.S.I.:	
ANALYTIC	AL PROCI	EDURE	C	HEMISTRY LABORATORY		1	73	8200
TITLE: GADOLINIU	UM BY AR	RSENAZO	III	PRC	DCEDURE NO 78200	-AP-GA	A1	REV.: 12
7.0	PRO	DCEDURI	<u>E</u>					
7.1	Prep	paration of	Standards and	Q.C. Check Sample				
	1.	Open th 78200-	ne appropriate IP-47 or 78200	method file for the UV/Vis spec -IP-52.	trometer	r used.	Refer t	0
	2.	Label e	each of the requ	ired 100 mL volumetric flasks a	as per Ta	able #1:		
				TABLE #1				
			Flask #	Contents				
			1	BLANK				
			2	0.100 mgGd/Kg H ₂ O STAND	ARD (S	td 1)		
		_	3	0.200 mgGd/Kg H ₂ O STAND	ARD (S	td 2)		
			4	0.300 mgGd/Kg H ₂ O STAND	ARD (S	td 3)		

5

6

7

8 etc.

3. Pipette 2.0 mL of arsenazo III solution and 10 mL of buffer solution into each 100 mL volumetric flask.

PROCESS SAMPLE # etc.

PROCESS SAMPLE #1

PROCESS SAMPLE #2

Q.C. CHECK SAMPLE (0.20 mg/Kg)

- 4. Prepare a 10.0 mgGd/Kg H₂O working calibration standard by pipetting 1.0 mL of the 1000 mgGd/Kg H₂O primary calibration standard into a 100 mL volumetric flask. Dilute to the mark with nanopure water.
- Pipette 1.0 mL of 10 mgGd/Kg H₂O working calibration standard into Flask #2 to 5. obtain a 0.100 mgGd/Kg H₂O standard.
- 6. Pipette 2.0 mL of 10 mgGd/Kg H₂O working calibration standard into Flask #3 to obtain a 0.200 mgGd/Kg H₂O standard.
- 7. Pipette 3.0 mL of 10 mgGd/Kg H₂O working calibration standard into Flask #4 to obtain a 0.300 mgGd/Kg H₂O standard.

K. MacGibbon	02-08-14	REVIEWED BY: R. Culligan	DATE:	APPROVED BY: G.H. Brown	BATE	PAGE: 10	of	16



ALYTICA	AL PROCE	CDURE CHEMIST	RY LABORATORY		7820
DOLINIU	M BY AR	SENAZO III	PRO	CEDURE NO.: 78200-AP-GA	A1
7.1	Prepa	aration of Standards and Q.C. Che	eck Sample (Cont'd)		
	8.	Prepare a 10.0 mgGd/Kg work primary Q.C. check standard in mL using nanopure water.	ing Q.C. check standard tto a 1000 mL volumetr	l by pipetting 1 ic flask. Make	.0 mL of th up to 1000
	9.	Pipette 2.0 mL of the working (prepared in Step 7.1.7) into Fl	Q.C. check standard fro ask #5. (<u>Note</u> : Dilution	m the 1000 mL n Factor D.F. =	flask 50000).
7.2	Prepa	aration and Analysis of Samples			
7.2.1	<u>SDS</u> #	#2 (LISS), and Moderator Liquid	Poison Samples		
	1.	For SDS#2 (LISS) Samples & sample into a 1000 mL volume water.	10000 mg/kg Q.C. Stan tric flask. Make up to 1	<u>dards</u> , Pipette 1 1000 mL using	.0 mL of nanopure
		For Moderator Liquid Poison S	Samples and 1000 mg/kg	a Standarde Pi	n atta 1 0 m
		of Process Sample into a 100 n nanopure water.	1L volumetric flask. Ma	ake up to 100 n	nL using
	2.	of Process Sample into a 100 m nanopure water. Use the guidelines in Table #2 the 1000 mL or 100 mL flask p required.	to Pipette a suitable vol prepared in Step 7.2.1.1	ume of process into flask #6, #	s sample fr 7, etc. as
	2.	of Process Sample into a 100 m nanopure water. Use the guidelines in Table #2 the 1000 mL or 100 mL flask p required.	to Pipette a suitable vol prepared in Step 7.2.1.1 TABLE #2	ume of process into flask #6, #	s sample fr 7, etc. as
	2.	of Process Sample into a 100 m nanopure water. Use the guidelines in Table #2 the 1000 mL or 100 mL flask p required. PROCESS SYSTEM	to Pipette a suitable vol orepared in Step 7.2.1.1 TABLE #2 VOLUME (mL)	ume of process into flask #6, # DILUTI FACTO	s sample fr 7, etc. as
	2.	of Process Sample into a 100 m nanopure water. Use the guidelines in Table #2 the 1000 mL or 100 mL flask p required. PROCESS SYSTEM SDS#2 (LISS)	to Pipette a suitable vol orepared in Step 7.2.1.1 TABLE #2 VOLUME (mL) 2.5	ume of process into flask #6, # DILUTI FACTO (D.F. 4000	S sample fr 7, etc. as ION OR) 0
	2.	of Process Sample into a 100 m nanopure water. Use the guidelines in Table #2 the 1000 mL or 100 mL flask p required. PROCESS SYSTEM SDS#2 (LISS) Moderator Poison TK3	to Pipette a suitable vol brepared in Step 7.2.1.1 TABLE #2 VOLUME (mL) 2.5 1.0	ume of process into flask #6, # DILUTI FACT((D.F. 4000	s sample fr 7, etc. as ION OR 0
	2.	of Process Sample into a 100 m nanopure water. Use the guidelines in Table #2 the 1000 mL or 100 mL flask p required. PROCESS SYSTEM SDS#2 (LISS) Moderator Poison TK3 *1000 mg/kg Standard Verification	to Pipette a suitable vol orepared in Step 7.2.1.1 TABLE #2 VOLUME (mL) 2.5 1.0 2.5	ume of process into flask #6, # DILUTI FACT((D.F. 4000 4000	s sample fr 7, etc. as ION OR 0 0
	2.	of Process Sample into a 100 m nanopure water. Use the guidelines in Table #2 the 1000 mL or 100 mL flask p required. PROCESS SYSTEM SDS#2 (LISS) Moderator Poison TK3 *1000 mg/kg Standard Verification	to Pipette a suitable vol orepared in Step 7.2.1.1 TABLE #2 VOLUME (mL) 2.5 1.0 2.5	ume of process into flask #6, # DILUTI FACT((D.F. 4000 1000	in L using s sample fr 7, etc. as iON OR) 0 0 0
	2. 3.	of Process Sample into a 100 m nanopure water. Use the guidelines in Table #2 the 1000 mL or 100 mL flask p required. PROCESS SYSTEM SDS#2 (LISS) Moderator Poison TK3 *1000 mg/kg Standard Verification	to Pipette a suitable vol orepared in Step 7.2.1.1 TABLE #2 VOLUME (mL) 2.5 1.0 2.5	ume of process into flask #6, # DILUTI FACT((D.F. 4000 1000 4000	in L using sample fr 7, etc. as ion on on on on on on on on on on on
	2. 3. 4.	of Process Sample into a 100 m nanopure water. Use the guidelines in Table #2 the 1000 mL or 100 mL flask p required. PROCESS SYSTEM SDS#2 (LISS) Moderator Poison TK3 *1000 mg/kg Standard Verification Add nanopure water to all alique Stopper and mix well. Allow 1	to Pipette a suitable vol orepared in Step 7.2.1.1 <u>TABLE #2</u> VOLUME (mL) 2.5 1.0 2.5 1.0 2.5 0 minutes for complete	ume of process into flask #6, # DILUTI FACTO (D.F. 4000 1000 4000	S sample fr 7, etc. as ION OR) 0 0
	2. 3. 4.	of Process Sample into a 100 m nanopure water. Use the guidelines in Table #2 the 1000 mL or 100 mL flask p required. PROCESS SYSTEM SDS#2 (LISS) Moderator Poison TK3 *1000 mg/kg Standard Verification	to Pipette a suitable vol orepared in Step 7.2.1.1 TABLE #2 VOLUME (mL) 2.5 1.0 2.5 1.0 2.5 1.0 2.5	ume of process into flask #6, # DILUTI FACT((D.F. 4000 1000 4000	is sample fr sample fr f, etc. as ion o o o



ANALYTICAL	PROCE	DURE	CHEMIS	TRY LABORATORY	Ţ	1	7	8200
								REV
GADOLINIUM	BY AR	SENAZO III	[78200)-AP-GA	A 1	12
								·
7.2	Prepa	aration and A	Analysis of Samples	(Cont'd)				
	5.	Zero the s	pectrophotometer u	sing the blank solution	n.			
	<u>Note</u>	: If using the ensure the value for t	ne Cary 50, prior to e dip probe is in th the blank, it is recor	o starting the method e blank solution. Do ded on the report form	l (clicking o not record n as 0.000.	g the 'St d the ab	art' bu sorbane	utton) ce
	6.	Analyze F form.	Flask #2 (Std #1). R	ECORD the absorba	nce of Star	ndard #1	on the	e report
	7.	Analyze th the report	he contents of the re form.	emaining flasks. REC	C ORD all a	absorbar	nce val	ues on
	8.	Calculate	results as per Section	on 8 of this procedure.	Attach the	e spectro	ophoto	meter
		printer out	tput to the report for	rm.				
7.3	<u>Main</u>	Moderator S	tput to the report for Samples (Less than	rm. <u>20 mgGd/Kg D₂O)</u>				
7.3	<u>Main</u> 1.	<u>Moderator S</u> Use the gu	tput to the report for Samples (Less than aidelines in Table #:	rm. <u>20 mgGd/Kg D₂O)</u> 3 to Pipette a suitable	volume of	process	s sampl	le into
7.3	<u>Main</u> 1.	Moderator S Use the gu Flask #6, #	tput to the report for Samples (Less than uidelines in Table #3 #7, etc. as required.	rm. <u>20 mgGd/Kg D₂O)</u> 3 to Pipette a suitable	volume of	process	sampl	le into
7.3	<u>Main</u> 1.	Use the gu Flask #6, #	tput to the report for Samples (Less than uidelines in Table #. #7, etc. as required.	rm. <u>20 mgGd/Kg D₂O)</u> 3 to Pipette a suitable TABLE #3	volume of	process	sampl	le into
7.3	<u>Main</u> 1.	Moderator S Use the gu Flask #6, #	tput to the report for Samples (Less than uidelines in Table #: #7, etc. as required.	rm. <u>20 mgGd/Kg D₂O)</u> 3 to Pipette a suitable <u>TABLE #3</u> VOLUME	volume of	process	s sampl	le into
7.3	<u>Main</u> 1.	Moderator S Use the gu Flask #6, # EXPECT	tput to the report for <u>Samples (Less than</u> uidelines in Table #. #7, etc. as required. FED SAMPLE ENTRATION	rm. 20 mgGd/Kg D ₂ O) 3 to Pipette a suitable TABLE #3 VOLUME	volume of	process iLUTIC	s sampl	le into
7.3	<u>Main</u> 1.	Moderator S Use the gu Flask #6, # EXPECT CONCE (mgG	tput to the report for <u>Samples (Less than</u> uidelines in Table # #7, etc. as required. FED SAMPLE ENTRATION Ed/Kg D ₂ O)	rm. <u>20 mgGd/Kg D₂O)</u> 3 to Pipette a suitable <u>TABLE #3</u> <u>VOLUME</u> (mL)	volume of DI F	process ILUTIC ACTO (D.F.)	s sampl DN R	le into
7.3	<u>Main</u> 1.	Moderator S Use the gu Flask #6, # EXPECT CONCE (mgG	tput to the report for <u>Samples (Less than</u> uidelines in Table #. #7, etc. as required. FED SAMPLE ENTRATION Ed/Kg D_2O) < 0.3	rm. 20 mgGd/Kg D ₂ O) 3 to Pipette a suitable TABLE #3 VOLUME (mL) 88	volume of DI F	process [LUTIC [ACTO] [D.F.] 1.136	s sampl	le into
7.3	<u>Main</u> 1.	Moderator S Use the gu Flask #6, # EXPECT CONCE (mgG	tput to the report for Samples (Less than uidelines in Table # #7, etc. as required. FED SAMPLE ENTRATION Ed/Kg D_2O) < 0.3 1	rm. 20 mgGd/Kg D ₂ O) 3 to Pipette a suitable TABLE #3 VOLUME (mL) 88 20	volume of DI F	process iLUTIC ACTO (D.F.) 1.136 5	s sampl DN R	le into
7.3	<u>Main</u> 1.	Moderator S Use the gu Flask #6, # EXPECT CONCE (mgG	tput to the report for <u>Samples (Less than</u> uidelines in Table #. #7, etc. as required. FED SAMPLE ENTRATION Ed/Kg D_2O) < 0.3 1 2	rm. 20 mgGd/Kg D ₂ O) 3 to Pipette a suitable TABLE #3 VOLUME (mL) 88 20 10	volume of DI F	process [LUTIC [ACTO] (D.F.) 1.136 5 10	s sampl	le into
7.3	<u>Main</u> 1.	Moderator S Use the gu Flask #6, # EXPECT CONCH (mgG	tput to the report for Samples (Less than uidelines in Table # #7, etc. as required. FED SAMPLE ENTRATION Ed/Kg D_2O) < 0.3 1 2 3 - 5	rm. 20 mgGd/Kg D ₂ O) 3 to Pipette a suitable TABLE #3 VOLUME (mL) 88 20 10 5.0	volume of DI F	⁵ process ILUTIC ACTO (D.F.) 1.136 5 10 20	s sampl	le into
7.3	<u>Main</u> 1.	Moderator S Use the gu Flask #6, # EXPECT CONCE (mgG	tput to the report for Samples (Less than uidelines in Table #. #7, etc. as required. FED SAMPLE ENTRATION Ed/Kg D_2O) < 0.3 1 2 3 - 5 5 - 9	rm. 20 mgGd/Kg D ₂ O) 3 to Pipette a suitable TABLE #3 VOLUME (mL) 88 20 10 5.0 3.0	volume of DI F	process ILUTIC ACTO (D.F.) 1.136 5 10 20 33.3 5	s sampl	le into
7.3	<u>Main</u> 1.	Moderator S Use the gu Flask #6, # EXPECT CONCE (mgG	tput to the report for Samples (Less than uidelines in Table # #7, etc. as required. FED SAMPLE ENTRATION Ed/Kg D_2O) < 0.3 1 2 3 - 5 5 - 9 9 - 13	rm. 20 mgGd/Kg D ₂ O) 3 to Pipette a suitable TABLE #3 VOLUME (mL) 88 20 10 5.0 3.0 2.0	volume of DI F	The second se	s sampl	le into
7.3	<u>Main</u> 1.	Image: printer out Moderator S Use the gu Flask #6, # EXPECT CONCE (mgG	tput to the report for Samples (Less than uidelines in Table #: #7, etc. as required. FED SAMPLE ENTRATION Ed/Kg D_2O) < 0.3 1 2 3 - 5 5 - 9 9 - 13 13 - 18 10	rm. 20 mgGd/Kg D ₂ O) 3 to Pipette a suitable TABLE #3 VOLUME (mL) 88 20 10 5.0 3.0 2.0 1.5 1.0	volume of DI F	The second state	s sampl	le into



Point Lepreau G.S	•		OVOTEM				D.C.	
ANALYTICAL I	PROCEI	DURE	CHEM	STRY LABORATO	RY	1	B.S.I.: 78	3200
GADOLINIUM	BY ARS	SENAZO III	[PROCEDURE NO)-AP-GA	A 1	REV.: 12
								<u> </u>
7.3	Main	Moderator S	Samples (Less tha	n 20 mgGd/Kg D ₂ O)	(Cont'd)			
	2.	Add nano	pure water to all a	liquot flasks to the 10	00 mL mark.			
	3.	Stopper an	nd mix well. Allo	w at least 10 minutes	for complete	e reactio	on.	
	4.	Zero the s	pectrophotometer	using the blank solut	ion.			
	<u>Note</u> :	If using th ensure th value for t	ne Cary 50, prior e dip probe is in the blank, it is rec	to starting the meth the blank solution. I orded on the report fo	od (clicking Do not recor rm as 0.000.	the 'St d the ab	art' bu sorbanc	i tton) xe
	5.	Analyze F form.	Flask #2 (Std #1).	RECORD the absorb	oance of Star	ndard #1	on the	report
	6.	Analyze the report	he contents of the form.	remaining flasks. RE	ECORD all a	absorbar	nce valu	ies on
	7.	Calculate printer ou	results as per Sect tput to the report f	tion 8 of this procedur form.	re. Attach the	e spectro	ophotor	neter
8.0	CAL	CULATION	NS					
8.1	<u>Calcu</u>	lation Using	g Computer					
8.1.1	Load W:\Cl C:\RE	Excel and o hemicalForr EPFORMS of	pen the spreadshe ns directory. If th lirectory on the lo	et file named R_AP14 e network is unavaila cal computer.	401.xls from ble, load the	the form fr	om the	
8.1.2	Enter	the absorba	nce values, sampl	e I.D.'s, and dilution	factors into t	he shad	ed cells	5.
			D BY:			BATE: I R	AGE:	

PREPARED BY:	DATE:	REVIEWED BY:	DATE:	APPROVED BY:	BATE	PAGE:		
K. MacGibbon	02-08-14	R. Culligan		G.H. Brown		13	of	16



FUNCTION: ANALYTICA	AL PROCEDURE CHEM	IISTRY LABORATORY	r	UNIT: 1	B.S.I.: 782	200
ITLE: GADOLINIU	M BY ARSENAZO III		procedure no 78200	-AP-GA	.1	rev.: 12
8.1	Calculation Using Computer (Cont'c	1)				
8.1.3	The Q.C. standard result and the p Analytical results cannot be used	rocess results will be aut d if control chart criteri	omatically a is not m	calculat et.	ted.	
	Record process sample results in t the completed REP-78200-AP-14.	he lab database, and othe 1 form and forward to lab	r applicab b Q.C. sen	le works ior.	heets. F	Print
9.0	PRECISION					
	The precision of this procedure ha	s been determined to be a	as follows:			
	For Aliquot Concentrations betwe	en 0.2 mgGd/Kg H ₂ O and	d 0.3 mgG	d/Kg H ₂	O:	
	95.0 % confidence level: (1-tail) 95.0 % confidence level: (2-tail) 99.9 % confidence level: (1-tail) 99.9 % confidence level: (2-tail)	2.6 % 3.1 % 5.0 % 5.3 %				
	For Aliquot Concentrations betwe	en 0.1 mgGd/Kg H ₂ O and	d 0.2 mgG	d/Kg H ₂	0:	
	95.0 % confidence level: (1-tail) 95.0 % confidence level: (2-tail) 99.9 % confidence level: (1-tail) 99.9 % confidence level: (2-tail)	5.1 % 6.1 % 9.8 % 10.5 %				
	For Aliquot Concentrations betwe	en the MDL and 0.1 mgC	Gd/Kg H ₂ C):		
	95.0 % confidence level: (1-tail) 95.0 % confidence level: (2-tail) 99.9 % confidence level: (1-tail) 99.9 % confidence level: (2-tail)	22.4 % 26.8 % 43.1 % 46.1 %				
REPARED BY: K. MacGibboi	n 02-08-14 R. Culligan	DATE: APPROVED BY: G.H. Brown		BATE: BA	^{®E∷} 14 of	1



FUNCTION:	SYSTEM:		UNIT:	B.S.I.:	
ANALYTICAL PROCEDURE	CHEMISTRY LABORATORY	V	1	78	200
A WEI HEILIKOULDOKL		1	1	70	200
TITLE:		PROCEDURE NO	.:		REV.:
GADOLINILIM BY ARSENAZO III	-	78200	$\Delta P_{G} \Delta$	1	12
		78200	-/11-0/1	. 1	12

10.0 METHOD DETECTION LIMIT

The LLD for this procedure is 0.011 mgGd/Kg H₂O, and the MDL is 0.022 mgGd/Kg H₂O. (INPO criteria)

<u>Note</u>: Divide the above LLD and MDL by 1.105 for detection limits in D_2O .

11.0 <u>REFERENCES</u>

- 1. Method Validation File No. MV-78200-AP-14.
- 2. Memo "Verification Of Gadolinium By Arsenazo III Procedure" W.R. Mahwinney to C.K. MacNeil; February 22, 1982; File No. 87-78200.

REPARED BY: K. MacGibbon	DATE: 02-08-14	REVIEWED BY: R. Culligan	DATE:	APPROVED BY: G.H. Brown	BATE	PAGE: 15	of	16



FUNCTION	NI ALYTICAL PROCE	DURE	SYSTEM: CHEMISTRY LABORATORY				UNIT: 1	B.S.I.: 78200)0	
GADOLINIUM BY ARSENAZO III						PRO	ROCEDURE NO.: 78200-AP-GA1		GA1	R	^{≣v.:} 12
APPENDIX I: Example of Form # REP-78200-AP-14.1 Analytical Procedure Quality Control Report Form REP-78200-AP-14.1									P-14.1		
	PROCESS SYSTEM:	ev. 00)	LISS TANK #	6 11 12	LIQUID POISON	D MODE			ERATOR		
	PROCEDURE	FLASK	ABSORBANCE		(CONTENT	TS 123	4.5	(Juner	ſ
	ABSORBANCE VALUES 7.2.1 / 7.2.2	$ \begin{array}{r} 1 \\ 2 \\ 3 \\ 4 \\ 5 \\ 6 \\ 7 \\ 8 \\ 9 \\ 10 \\ 11 \\ 12 \\ 13 \\ 14 \\ 15 \\ \end{array} $		0.000 0.100 0.200 0.300 Q.C. CHECK SAMPLE I.D SAMPLE I.D SAMPLE I.D SAMPLE I.D SAMPLE I.D SAMPLE I.D SAMPLE I.D SAMPLE I.D SAMPLE I.D	mGd/Kg <u>2</u> O B mGd/Kg <u>2</u> O S mGd/Kg <u>2</u> O S mGd/Kg <u>2</u> O S SAMPI F	LANK TANDARD TANDARD TANDARD	Dilution F Dilution F Dilution F Dilution F Dilution F Dilution F Dilution F Dilution F Dilution F	Factor= Factor= Factor= Factor= Factor= Factor= Factor= Factor= Factor= Factor= Factor=			
	SLOPE CHECK 8.1.3	CALCULA	TED SLOPE OF SLOPE SLOPE RESU	ELEAST SQU	ARES FIT:				-R ²		
	Q.C. CHECK STANDARD 8.1.3	ANALYZED Q.C. CHECK STANDARD mgGd/Kg H2O Q.C. STANDARD RESULT:									
	RESULT(S) REPORTED 8.1.3		(Standard Veri 	fications) mgGd/Kg HO mgGd/Kg HO mgGd/Kg HO mgGd/Kg HO mgGd/Kg HO mgGd/Kg HO mgGd/Kg HO mgGd/Kg HO mgGd/Kg HO mgGd/Kg HO	ignificant fig	ures are rep	(Proces	s Sam	ples) mg Gd/kg mg Gd/kg mg Gd/kg mg Gd/kg mg Gd/kg mg Gd/kg mg Gd/kg mg Gd/kg	D ₂ O D ₂ O	
Completed By:											
PREPARED BY:DATE:REVIEWED BY:DATE:ABPROVED BY:DATE:PAGEK. MacGibbon02-08-14R. CulliganG.H. Brown1							page: 16	of	16		