



SOUTH PLATTE  
**RENEW**

# Quality Assurance/Quality Control

What It Is and Why It's Important



The Basics



Manual Analysis QC



Instrument Analysis QC



Sample Prep/Digestions QC



Can I Trust My Results?



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# The Basics

Not just standard recoveries and duplicates:

- Establishes standard operating procedures (SOPs) for each step of the laboratory testing process, ranging from specimen handling to instrument performance validation
- Defines administrative requirements, such as mandatory recordkeeping, data evaluation, and internal audits to monitor adherence to SOPs
- Specifies corrective actions, documentation, and the persons responsible for carrying out corrective actions when problems are identified
- Sustains high-quality employee performance





# SPR Quality Assurance Manual

1. Facility and Security
2. Personnel
3. Instrumentation and Preventative Maintenance
4. Sample and Glassware Handling, Storage, and Disposal
5. Analytical Methodology and Standard Operating Procedures (SOP)
6. Calibrants and Reagents
7. QC Requirements
8. Calibration
9. Data Review, Corrective Action, and Data Handling
10. Reporting
11. Audit Functions and Corrective Action
12. Archives and Record Retention
13. Safety
14. Contract Laboratories

# QC for Manual Analyses

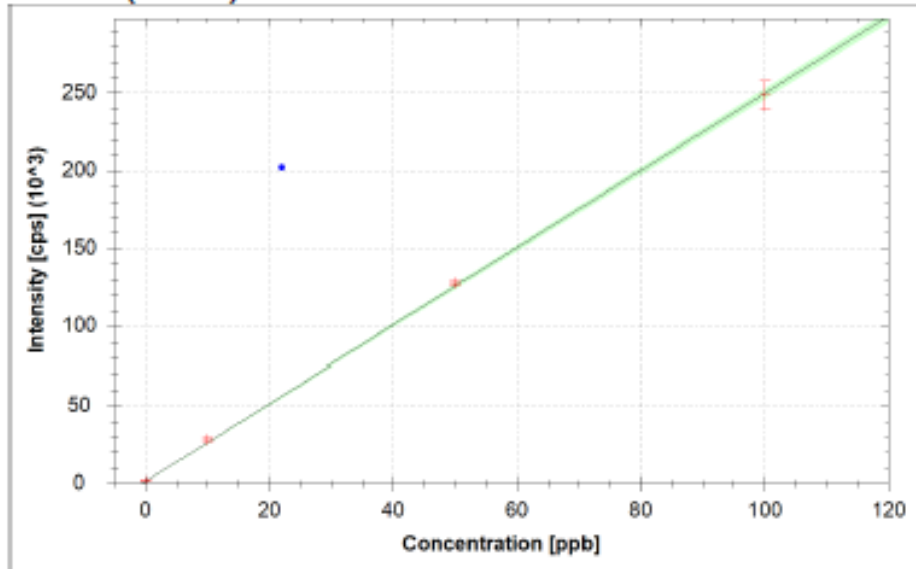
## A batch = 20 samples

- ❖ Laboratory Reagent Blank (LRB)
- ❖ Laboratory Fortified Blank (LFB)
- ❖ Laboratory Control Standard (LCS)
- ❖ Equipment Blank (if appropriate)
- ❖ Field Blank (if appropriate)
- ❖ Method Duplicate (MD) – frequency of 5 % (1 per batch)
- ❖ Field Duplicate (if appropriate)
- ❖ Matrix Spike (MS) – frequency of 10% (2 per batch)

ALK	Sample #	Sample ID	Sample Date	Aliquot (ml)	H <sub>2</sub> SO <sub>4</sub> (ml)	Result (mg/L as CaCO <sub>3</sub> )		
1	0	B_ALK	N/A	25		0	<10	
2	0	L_ALK	N/A	25		0	#DIV/0!	%R
3	0	LFB_ALK	N/A	25		0	0.0	%R
4		Sample 1		25		0		
5	0		D_ALK	25		0	#DIV/0!	RPD
6	0		S_ALK	25		0	0.0	%R
7		Sample 2		25		0		
8		Sample 3		25		0		
9		Sample 4		25		0		
10		Sample 5		25		0		
11		Sample 6		25		0		
12		Sample 7		25		0		
13		Sample 8		25		0		
14		Sample 9		25		0		
15		Sample 10		25		0		
16		Sample 11		25		0		
17	0		S_ALK	25		0	0.0	%R
18		Sample 12		25		0		
19		Sample 13		25		0		
20		Sample 14		25		0		
21		Sample 15		25		0		
22		Sample 16		25		0		
23		Sample 17		25		0		
24		Sample 18		25		0		
25		Sample 19		25		0		
26		Sample 20		25		0		

# What Is A Calibration Curve?

66Zn (KED)



$$f(x) = 2482.8212 \cdot x + 1549.5114$$

$$R^2 = 0.9997$$

$$\text{BEC} = 0.624 \text{ ppb}$$

$$\text{LoD} = 0.1687 \text{ ppb}$$

## Calibration Standards for zinc:

- 10 ppb
- 50 ppb
- 100 ppb

Concentration	Intensity, counts per second ( $\times 10^3$ )
0 ug/L	0
10 ug/L	30
50 ug/L	130
100 ug/L	250

# QC for Instrumentation Analyses

## A batch = 10 injections

- ❖ Initial Calibration Verification or Instrument Performance Check (ICV or IPC) – done at beginning of run; recovery must be within 90% - 110% of true value
- ❖ Initial Calibration Blank (ICB) – done at beginning of run; result must be <RL
- ❖ Laboratory Control Standard (LCS)
- ❖ Laboratory Reagent Blank (LRB) – frequency of 1 per 20 samples (not injections)
- ❖ Laboratory Fortified Blank (LFB) – frequency of 1 per 20 samples (not injections)
- ❖ Equipment Blank (if appropriate)
- ❖ Field Blank (if appropriate)
- ❖ Method Duplicate (MD) – frequency of 1 per 20 samples (not injections)
- ❖ Field Duplicate (if appropriate)
- ❖ Matrix Spike (MS) – frequency of 2 per 20 samples (not injections)
- ❖ Continuing Calibration Verification (CCV) – frequency of every 10 injections and at end of run; recovery must be within 90% - 110% of true value
- ❖ Continuing Calibration Blank (CCB) – frequency of every 10 injections and at end of run; result must be <RL

9	AC34868 \$IPC_IC	IPC
10	AC34868 \$ICB_IC	ICB
11	AC34868 \$LRB_IC	LRB
12	AC34868 \$LFB_IC	LFB
13	AC34868 \$IC	FE-COMP
14	AC34868 \$D_IC	FE-COMP DUP
15	AC34868 \$\$_IC	FE-COMP LFM
16	AC34871 \$IC	AS-MLSS-COMP
17	AC34875 \$IC	DN-EFF-COMP
18	AC34876 \$IC	DN-FILT-2-COMP
19	AC34877 \$IC	DN-FILT-4-COMP
20	AC34878 \$IC	DN-FILT-6-COMP
21	AC34868 \$CPC_IC	CPC
22	AC34868 \$CCB_IC	CCB
23	AC34879 \$IC	DN-FILT-8-COMP
24	AC34880 \$IC	DN-INF-COMP
25	AC34884 \$IC	HW-GB-EFF
26	AC34885 \$IC	NTF-EFF-1-COMP
27	AC34886 \$IC	NTF-EFF-2-COMP
28	AC34868 \$IC	NTF-EFF-3-COMP
29	AC34868 \$\$_IC	NTF-EFF-3-COMP LFM
30	AC34888 \$IC	NTF-INF-COMP
31	AC34889 \$IC	PC-EFF-AUTO
32	AC34900 \$IC	SC-EFF-COMP
33	AC34868 \$CPC_IC	CPC
34	AC34868 \$CCB_IC	CCB
35	AC34910 \$IC	TF-EFF
36	AC34911 \$IC	TF-INF-AUTO
37	AC34911 \$CPC_IC	CPC
38	AC34911 \$CCB_IC	CCB



# QC for ICP-MS Analyses – The Run

## A batch = 10 injections

- ❖ QCSA and QCSAB standards - % recovery range based on control charts
- ❖ Initial Calibration Verification (ICV) – done at beginning of run; recovery must be within 90% - 110% of true value for each metal
- ❖ Initial Calibration Blank (ICB) – done at beginning of run; result must be <RL for each metal
- ❖ Laboratory Reagent Blank (LRB) – frequency of 1 per 20 samples (not injections)
- ❖ Laboratory Fortified Blank (LFB) – frequency of 1 per 20 samples (not injections)
- ❖ Equipment Blank (if appropriate)
- ❖ Field Blank (if appropriate)
- ❖ Method Duplicate (MD) – frequency of 1 per 20 samples (not injections)
- ❖ Field Duplicate (if appropriate)
- ❖ Matrix Spike (MS) – frequency of 2 per 20 samples (not injections)
- ❖ Continuing Calibration Verification (CCV) – frequency of every 10 injections and at end of run; recovery must be within 90% - 110% of true value for the first CCV, and then the range becomes 85% - 115% for each metal
- ❖ Continuing Calibration Blank (CCB) – frequency of every 10 injections and at end of run; result must be <RL for each metal

Index	Label
1	BLANK
2	STD 1
3	STD 2
4	STD 3
5	STD 4
6	STD 5
7	STD 6
8	AD23181 \$QA_ICPMS
9	AD23181 \$QAB_ICPMS
10	AD23181 \$I_ICPMS
11	AD23181 ICV CATIONS
12	AD23181 \$ICB_ICPMS
13	AD23181 \$B_ICPMSD
14	AD23181 \$LFB_ICPMSD
15	AD23181 \$F_ICPMSD
16	AD23178 \$ICPMSD RIV-BEAR 10X
17	AD23178 \$ICPMSD RIV-BEAR
18	AD23179 \$ICPMSD RIV-BIG 20X
19	AD23179 \$ICPMSD RIV-BIG
20	AD23180 \$ICPMSD RIV-DOWN 10X
21	AD23180 \$ICPMSD RIV-DOWN
22	AD23181 \$C_ICPMS
23	AD23181 CCV CATIONS
24	AD23181 \$CCB_ICPMS
25	AD23181 \$ICPMSD RIV-EFF 10X
26	AD23181 \$ICPMSD RIV-EFF
27	AD23181 \$FD_ICPMSD RIV-EFF-FD 10X
28	AD23181 \$FD_ICPMSD RIV-EFF-FD
29	AD23181 \$\$_ICPMSD RIV-EFF 10X
30	AD23181 \$\$_ICPMSD RIV-EFF
31	AD23182 \$ICPMSD RIV-LIT 20X
32	AD23182 \$ICPMSD RIV-LIT
33	AD23182 \$C_ICPMS
34	AD23182 CCV CATIONS
35	AD23182 \$CCB_ICPMS
36	AD23183 \$ICPMSD RIV-UP 10X
37	AD23183 \$ICPMSD RIV-UP
38	AD23183 \$C_ICPMS
39	AD23183 CCV CATIONS
40	AD23183 \$CCB_ICPMS



## Optimizing the instrument:

### Performance Report



#### System

Start time: 3/8/2022 12:19:38 PM  
 Instrument: iCAP Q  
 Operator: ENGLEWOOD\laboratory  
 Template: STD\_3Minutes  
 Instrument Serial Number: 03296R  
 Last Autotune: Autotune-SourceTune High Matrix-20220308-095757072.imatdat  
 Solution: 1 ppb Tune B in 2% HNO3 and 0.5% HCl.

#### Sensitivity & Stability Test

Result	Runs	Sweeps
Passed	3	60

#### Sensitivity

Analyte	Result	Value	Condition	Limit
Bkg4.5	Passed	0.83 CPS	Less than	5.0 CPS
Bkg220.7	Passed	0.94 CPS	Less than	5.0 CPS
7Li	Passed	94,584.0 CPS	Greater than	40,000.0 CPS
59Co	Passed	176,374.0 CPS	Greater than	80,000.0 CPS
238U	Passed	481,248.0 CPS	Greater than	200,000.0 CPS
140Ce.16O/140Ce	Passed	0.0273	Less than	0.03
137Ba+/137Ba	Passed	0.0278	Less than	0.035
115In	Passed	394,174.0 CPS	Greater than	190,000.0 CPS

#### Stability

Analyte	Value	Limit
7Li	0.2%	2
59Co	0.3%	2
238U	0.6%	2
115In	0.2%	2

#### Mass Calibration Test

Result	Channels	Dwell	MeasureWidth	PointSpacing	Sweeps
Passed	75	0.04	1.5	0.02	5

Analyte	Result	Centroid Mass [u]	Offset	Peak width [u]	Peak width min [u]	Peak width max [u]
7Li	Passed	6.9951	0.0209	0.713	0.650	0.850
59Co	Passed	58.9194	0.0138	0.727	0.650	0.850
115In	Passed	114.8976	0.0063	0.737	0.650	0.850
238U	Passed	238.0606	0.0098	0.736	0.650	0.850

### Performance Report

#### Tune Settings

Parameter	Value
Additional Gas Flow 1	0.00
Additional Gas Flow 2	0.00
Additional Gas Flow 3	0.00
Anqular Deflection	-374.00
Auxilliary Flow	0.80
CCT Bias	-2.00
CCT Entry Lens	-78.00
CCT Exit Lens	-160.00
CCT Focus Lens	-2.76
CCT1 Flow	0.00
CCT1 Shut-Off Valve	0.00
CCT2 Flow	0.00
CCT2 Shut-Off Valve	0.00
Cool Flow	14.00
D1 Lens	-200.00
D2 Lens	-80.00
Deflection Entry Lens	-35.00
Dry Pump Speed	0.00
Extraction Lens 1 Negative	0.00
Extraction Lens 1 Polarity	0.00
Extraction Lens 1 Positive	0.00
Extraction Lens 2	-120.00
Focus Lens	21.40
Nebulizer Flow	1.07
Peristaltic Pump Speed	20.00
Plasma Power	1550.00
Pole Bias	-1.00
Quad Entry Lens	-26.60
Sampling Depth	5.00
Spray Chamber Temperature	2.70
Torch Horizontal Position	0.46
Torch Vertical Position	-0.50
Virtual CCT Mass Maximum Dac Limit Set	4095.00
Virtual CCT Mass parameter b	0.65
Virtual CCT Mass to Dac Factor	130.00
Virtual CCT Mass to Dac Offset	0.00

#### Vacuum Check

Parameter	Result	Value
Analyzer Pressure	Vacuum ok	3.368e-7
Interface Pressure		1.546e+0

#### Detector Voltages

Analog	Counting
-2050.00	1425.00



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# QC for Sample Prep/Digestions

## ICP-MS Total Recoverable Digestion EPA 200.8

Equipment	Calibration Due
Digestion Block: LAB- 0275	03/15/2022
Display Temp: 111 °C	
Pipettes: LAB- 0004	03/01/2022
LAB- 0005	03/01/2022
LAB- 0341	07/26/2022

Standards and Reagents	Log # and Expiration Date
Spike Solution #1: PS02020	03/30/2022
Spike Solution #2: PS02021	03/30/2022
Spike Solution #3: PS02022	03/30/2022
Nitric Acid: C02334	07/09/2022
Hydrochloric Acid: C02390	10/01/2025

QA Batch #: 128440
Preparation Batch ID: 021822_TR
Preparation Date: 02/18/2022
Start Time: 8:22
Stop Time: 13:55
Analyst: LALLEN
Reviewed Date: 02/18/2022
Reviewed By: AWO0

Volume
0.05 mL per 50 mL sample
0.05 mL per 50 mL sample
0.50 mL per 50 mL sample

Tube #	Analysis Code	Sample ID#	Location	Initial Volume (ml)	Final Volume (ml)
1	B PREP-TR	AD22584	RIV-EFF	50	50
2	LFB PREP-TR	AD22584	RIV-EFF	50	50
3	FB PREP-TR	AD22584	RIV-EFF	50	50
4	PREP-TR	AD22581	RIV-BEAR	50	50
5	PREP-TR	AD22582	RIV-BIG-DRY	50	50
6	PREP-TR	AD22583	RIV-DOWN	50	50
7	PREP-TR	AD22584	RIV-EFF	50	50
8	FD PREP-TR	AD22584	RIV-EFF	50	50
9	S PREP-TR	AD22584	RIV-EFF	50	50
10	PREP-TR	AD22585	RIV-LIT-DRY	50	50
11	PREP-TR	AD22586	RIV-UP	50	50

## QC for the digestion:

- ✓ Laboratory Reagent Blank (LRB)
- ✓ Laboratory Fortified Blank (LFB)
- ✓ Field Blank (FB)
- ✓ Field Duplicate (FD)
- ✓ Laboratory Fortified Matrix (LFM)



# Can I Trust My Results?

The instrument is working properly if:

- ❖ Successful performance check
- ❖ CCV/CCB every 10 injections and within 90% - 110% of true value (85% - 115% after the first CCV)

The analytical run is working properly if:

- ❖ Calibration curve:  $r^2 \geq 0.995$
- ❖ Internal standards: 60% - 125%

The sample digestion procedure was successful if:

- ❖ All QC samples from the digestion are within acceptable limits

**QC results won't confirm:**

- That samples were poured and placed into the correct autosampler positions
- That all sample dilutions were prepared correctly
- That no contaminants made their way into any of the samples, either while sitting in the autosampler or during the digestion procedure
- That the analysis was performed within holding time



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# Do You Have Confidence In The Data?

## Analysis results:

- ICV/ICB passed
- CCVs/CCBs passed
- QCSA and QCSAB passed
- Duplicates were within 20% RPD
- Spike recoveries between 75% - 125%
- Method Blank from the digestion failed for 3 metals – results above the Reporting Limit

## Analysis results:

- ICV/ICB passed
- CCVs/CCBs passed
- QCSA passed but QCSAB was outside acceptable limits
- One duplicate out of 3 was outside 20% RPD
- Spike recoveries between 75% - 125%
- Method Blank from the digestion passed

## Analysis results:

- ICV/ICB passed
- CCVs/CCBs passed except for the final CCV
- QCSA and QCSAB passed
- Duplicates were within 20% RPD
- Spike recoveries between 75% - 125%
- Method Blank from the digestion failed for 16 metals – results above the Reporting Limit





# Do You Have Confidence In The Laboratory?

Method Blank results from  
14 separate digestions

xxx indicates result is greater than the RL

xxx indicates result is between the MDL and the RL

Date	Li	Be	B	Na	Mg	Al	K	Ti	V	Cr	Mn	Fe	Co	Ni	Cu	Zn	As	Se	Sr	Zr	Nb	Mo	Pd	Ag	Cd	Sn	Sb	Ba	W	Pt	Tl	Pb	Th	U
11/26/2021	34.847	<0.5	17.455	<25	<25	<15	<50	<0.5	<1	0.242	4.13	<25	<0.5	0.035	<1	-0.611	0.032	0.024	0.025	33.52	0.237	0.023	0.007	0.005	0.039	-0.331	0.031	-0.105	0.033	-0.013	0.111	0.117	0.001	0.005
11/26/2021	18.459	<0.5	10.448	<25	<25	<15	<50	<0.5	<1	0.214	3.705	<25	<0.5	<1	<1	-0.398	0.004	0.04	-0.009	-5.255	0.128	-0.01	-0.002	0.005	0.01	307.49	0.121	-0.062	0.142	0.004	0.063	0.203	0.026	0.002
11/26/2021	41.32	<0.5	11.141	<25	<25	<15	<50	<0.5	<1	0.306	<0.5	<25	<0.5	<1	<1	-0.347	-0.012	0.005	0.042	10.528	0.175	0.046	0.008	0.009	0.001	-0.344	-0.028	-0.025	0.271	0.005	0.08	0.183	0.011	0
11/26/2021	<10	<0.5	15.914	<25	<25	<15	<50	<0.5	<1	1	<0.5	<25	<0.5	0.767	<1	4.200	0.013	-0.011	0.028	42.91	13.505	0.303	0.083	0.007	0.034	10.751	0.064	0.214	0.326	-0.007	0.076	0.137	0.138	0.029
11/26/2021	<10	<0.5	6.2838	<25	<25	<15	<50	<0.5	<1	0.178	0.79	<25	<0.5	<1	<1	0.125	-0.023	0.009	-0.132	63.78	6.217	-0.008	-0.006	0.003	-0.01	1.018	0.059	0.028	0.074	-0.014	0.068	0.158	0.013	0.003
11/27/2021	13.118	<0.5	99.884	535.816	27.307	<15	<50	<0.5	<1	0.229	0.766	<25	<0.5	0.663	<1	2.774	0.073	0.031	0.747	-20.752	5.052	-0.017	0.024	0.004	0.001	-0.148	0.011	-0.040	0.027	-0.007	0.001	0.004	0.071	0.025
11/27/2021	21.452	<0.5	24.752	73.066	<25	<15	<50	<0.5	<1	0.313	<0.5	<25	<0.5	0.186	<1	-0.084	-0.014	-0.016	0.44	42.035	5.903	-0.04	0.024	-0.001	-0.011	-0.322	0.003	-0.048	-0.012	-0.004	-0.011	-0.032	0.047	0.009
11/27/2021	18.874	<0.5	15.109	<25	<25	<15	<50	<0.5	<1	0.123	0.66	<25	<0.5	0.038	<1	0.026	-0.004	0.011	0.381	187.933	5.829	-0.016	0.045	0.002	0.011	-0.237	0.027	-0.023	-0.015	-0.01	-0.009	-0.051	0.068	0.004
11/27/2021	<10	<0.5	17.61	<25	<25	<15	<50	<0.5	<1	0.311	<0.5	<25	<0.5	0.051	<1	-0.076	0.003	0.013	-0.16	-18.825	0.562	0.037	0.027	0	-0.004	0.095	0.015	-0.103	0.239	-0.006	0.065	0.13	0.035	0.003
11/27/2021	<10	<0.5	11.946	<25	<25	<15	<50	<0.5	<1	0.213	<0.5	<25	<0.5	0.044	<1	1.177	-0.003	-0.012	-0.176	-48.802	4.468	-0.008	0.037	0.006	0.009	-0.07	0.042	-0.058	0.023	0	0.045	0.18	0.12	0
11/27/2021	<10	<0.5	1.225	<25	<25	<15	<50	<0.5	<1	0.058	<0.5	<25	<0.5	0.1663	<1	0.679	0.048	0.035	-0.117	-24.522	-1.454	-0.025	0.044	-0.005	0.009	0.026	0.013	-0.054	0.01	-0.007	0.205	0.048	0.014	0.001
11/27/2021	<10	<0.5	<0.5	35.392	<25	<15	<50	<0.5	<1	0.284	8.482	<25	<0.5	0.117	<1	-0.090	0.013	0.015	-0.17	-42.243	-1.67	-0.015	0.013	0.004	0.017	-0.227	-0.047	-0.055	0.039	-0.005	0.027	0.073	-0.003	-0.003
11/27/2021	<10	<0.5	<0.5	<25	<25	<15	<50	<0.5	<1	0.4	7.434	<25	<0.5	0.032	<1	-0.401	0.018	0.009	-0.163	-30.068	-1.409	0.024	0.026	0	0.021	-0.158	-0.01	-0.077	0.265	-0.006	0.052	0.127	0.021	0.009
11/27/2021	<10	<0.5	<0.5	<25	<25	<15	<50	<0.5	<1	0.269	17.884	<25	<0.5	0.075	<1	-0.749	0.023	-0.014	-0.189	6.854	-1.779	0.032	0.017	-0.006	-0.003	-0.225	-0.011	-0.030	0.079	0	0.059	0.133	-0.004	0.003



# What Constitutes A Good Laboratory

- 1) Does the lab have a quality control manual? And will it be possible to share an outline or table of contents with you?
- 2) What testing does the lab perform in-house and what testing does it outsource to other labs? Have they audited the other labs? How do they handle questionable results from the outside lab?
- 3) Does the lab have an independent quality assurance (QA) department?
- 4) Is the lab accredited with any organization? If so, which one(s)? Note: ISO 17025 certification is on a per-method basis. If the lab has ISO 17025, ask for which methods.
- 5) Does the lab follow any published standard of good laboratory practices? If so, which ones.



## What Constitutes A Good Laboratory – cont'd

- 6) What does the lab do when test results are unexpected or don't meet specifications?  
What does the lab do when there are conflicting test results?
- 7) How does the lab handle samples from the time of receipt, through lab analysis, report issuance and data archiving?
- 8) Does the lab provide support and transparency with the test results?
- 9) Does the lab have written procedures and schedules for instrument and equipment maintenance and calibration? Is this documented and can they share that documentation?
- 10) Does the lab have written valid test methods for the sample analysis? Can the lab disclose the test procedures and methods used? How does the lab assure test results are accurate and precise? This will enable you to independently verify results by providing the lab's method of analysis to another lab that can duplicate the test.





# What Constitutes A Good Laboratory – cont'd

- 11) Who performs the testing and what are their qualifications? How much experience does the lab and the analyst have in dealing with the specific analyte and matrix?
- 12) What is the average cost and time it takes to complete testing and deliver results?
- 13) How does the lab record raw data? Can a final report be tracked back to the original raw data? How long does the lab retain raw data and does the lab provide that data upon request?
- 14) Does the lab provide documentation when there is no scientifically valid method for testing or examining any exempted product specification at the finished batch stage?

**Taken from:**

**<https://www.thefreelibrary.com/Choosing+the+right+analytical+lab%3a+here+are+some+important+questions...-a0459074774>**



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# Questions?

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