Alcohol and Marijuana Control Office
550 W 7th Avenue, Suite 1600
Anchorage, AK 99501
marijuana.licensing@alaska.gov
https://www.commerce.alaska.gov/web/amco
Phone: 907.269.0350

Alaska Marijuana Control Board

Operating Plan Supplemental
Form MJ-06: Marijuana Testing Facility

What is this form?

This operating plan supplemental form is required for all applicants seeking a marijuana testing facility license and must accompany the **Marijuana Establishment Operating Plan (Form MJ-01)**, per 3 AAC 306.020(b)(11). Applicants should review **Chapter 306: Article 6** of the **Alaska Administrative Code.** This form will be used to document how an applicant intends to meet the requirements of those regulations. If your business has a formal operating plan, you may include a copy of that operating plan with your application, but all fields of this form must still be completed per 3 AAC 306.020 and 3 AAC 306.615(2).

What additional information is required for testing facilities?

Applicants must identify how the proposed establishment will comply with applicable regulations regarding the following:

- Prohibitions
- Testing practices and procedures
- Employee qualification and training
- Security
- Reporting and records retention

This form must be submitted to AMCO's main office before any marijuana testing facility license application will be considered complete.

Section 1 – Establishment Information

Enter information for the business seeking to be licensed, as identified on the license application.

Licensee:	Peak Analytical LLC	**License Number:**	**15590**		
License Type:	**Marijuana Testing Facility**				
Doing Business As:	**Peak Analytical LLC**				
Premises Address:	**2208 Tongass Avenue**				
City:	**Ketchikan**	**State:**	ALASKA	**ZIP:**	99901

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Section 2 – Prohibitions

Applicants should review 3 AAC 306.610 and be able to answer "Agree" to all items below.

The marijuana testing facility will not:	Agree	Disagree
Sell, deliver, distribute or transfer any marijuana or marijuana product to a consumer, with or without compensation	✓	☐
Allow any person to consume marijuana or marijuana product on its licenses premises	✓	☐

Section 3 – Testing Practices and Procedures

Review the requirements under 3 AAC 306.615, 3 AAC 306.635 – 3AAC 306.645, and 3 AAC 306.660, and identify how the proposed establishment will meet the listed requirements.

Describe each test the marijuana testing facility will offer:

Potency testing will be offered for marijuana, marijuana products and marijuana concentrates. Analytes include THC, THCa, CBD, CBDa and CBN. The test specimen will undergo sample preparation prior to analysis using High Performance Liquid Chromatography. Sample preparation generally consists of homogenization, extraction and concentration or dilution. The test will result in a percentage by weight. Total THC will be calculated as:
Total THC=(%THC) + (%THCa)x0.877

Microbial testing will be offered for marijuana, marijuana products and marijuana concentrates. Analytes include shiga-toxin escherichia coli (STEC), salmonella and aspergillus fumigatus, aspergillus flavus, aspergillus niger. Microbial contaminants will be analyzed using standard plate counts. Sample preparation generally consists of homogenization and dilution prior to testing.

Residual solvents testing will be offered for solvent based marijuana concentrates. This analysis will be performed utilizing head space gas chromatography with flame ionization detection. Analytes include butanes, heptanes, benzene, toluene, hexane and total xylenes.

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Standard Operating Procedure Manual (3 AAC 306.640):

Applicants for marijuana testing facilities must have a written procedures manual with detailed instructions explaining how to perform each testing method the applicant or marijuana testing facility uses, and minimum standards for each test. Applicants should be able to answer "Agree" to all items below.

The marijuana testing facility will ensure that the standard operating procedure manual:	Agree	Disagree
Is available to each employee at all times	☑	☐
Will cover at least the required procedures listed under 3 AAC 306.640	☑	☐

Describe the marijuana testing facility's standard operating procedure for each test the facility will offer:

Potency Testing:
Plant Matrices and Concentrates:
-Into extraction vial weigh 0.500 g of homogenized sample (Concentrates: weigh 0.100 g of homogenized sample) -Pipette 10 mL of MeOH on top of the sample -Vortex for 4 minutes -Centrifuge 1 minute at 5000 rpm -Filter supernatant with syringe filter into clean transfer vial -Add 990 µl of MeOH to the autosampler vial -Transfer 10 µl of the filtered extract into the autosampler vial, then cap the vial -Mix thoroughly by shaking & analyze via HPLC
Edible Matrices:
-Into extraction vial weigh 0.500 g homogenized sample (Carbonated beverages: add 10 ml degassed sample) -Add 5 ml of reagent water and hydrate for 30-60 mins using orbital rocker -Add 5 ml of a high polar index solvent: Acetonitrile (ACN containing 1% Acetic Acid) or MeOH -Add one QuEChERS Q-sepTM Original Unbuffered Method packet -Vortex for 4 minutes -Centrifuge for 5 minutes at 5000 rpm -Transfer approximately 1 ml of the supernatant to a dSPE tube -Vortex for 1 minute -Centrifuge for 4 minute at 5000 rpm -Add 950 µl of MeOH to the autosampler vial -Transfer 50 µl of the supernatant into the autosampler vial, then cap -Mix thoroughly by shaking & analyze via HPLC

Microbial Testing:
Plant & Edible Matrices (analyzed for STEC)
1.Allow plates (Hardy Diagnostics Compact DryTM EC) to reach room temperature
2.Using one sterile pipet or scoop for each sample, aliquot 1 ml or 1 g of homogenized test sample to a 99 ml pre-filled Dilu-Lok II™ dilution vial to yield a 1:100 dilution. Mix thoroughly by shaking 25 times
3.Transfer approximately 10-15 ml of diluted sample into a sterile centrifuge tube
4.Centrifuge 2 minutes at 5000 rpm or filter with a syringe filter
7.Inoculate sample plates by pipetting 1 ml of each sample dilution directly to the center of a dry plate well
8.Replace plate lids
9.Invert the plates and incubate, upside down with the medium on top, at 35-37 degrees C for 24 hours

See additional space provided. These are abbreviated methods. See attached Standard Operating Procedures Manual for full methods.

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Laboratory Testing of Marijuana and Marijuana Products (3 AAC 306.645):

A licensed marijuana testing facility must meet minimum standards for laboratory testing. Applicants should be able to answer "Agree" to all items below.

The marijuana testing facility applicant has:	Agree	Disagree
Read and understands and agrees to the requirements listed under 3 AAC 306.645	☑	☐

Describe the acceptable range of results for each test the marijuana testing facility will offer:

Potency Testing - a sample has passed if it meets the following criteria:
1. less than 5 mg active THC or Delta 9 in a one serving edible
2. less than a cumulative 50 mg active THC or Delta 9 in a multi serving package
3. or any serving in a multiple serving package is less than 20% from the manufacturer's target

Microbial Testing - the test returns a failed result if one colony forming unit of any analyte (shiga-toxin escherichia coli (STEC), salmonella and aspergillus fumigatus, aspergillus flavus, aspergillus niger) is detected in a one-gram sample.

Residual Solvents - must have concentrations less than values stated below for a passing result:
butanes < 800 PPM
heptanes <500 PPM
benzene <1 PPM
toluene <1 PPM
hexane <10 PPM
total xylenes <1 PPM

Review of analytical results will ensure tests meet the acceptable range of results for the laboratory. Duplicate analyses will be performed on every sample batch in order to ensure consistency of testing accuracy. Results must be within 10% relative standard deviation of the mean to be accepted.

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Section 4 – Employee Qualification and Training

Review the requirements under 3 AAC 306.625 – 3 AAC 306.630, and identify how the proposed establishment will meet the listed requirements.

Proficiency Testing Program (3 AAC 306.625):

Describe how the marijuana testing facility will ensure the scientific director and all testing analysts are proficient in utilizing testing equipment and analyzing samples:

The Scientific Director will receive extensive personal training on the Agilent Technologies GC and LC chromatographs and their accessory equipment. Training is provided by qualified trainers who have been trained on Agilent Technologies equipment.

All analytical staff will go through a standardized training program provided by the Scientific Director. Training includes, but is not limited to, reviewing the Peak Analytical Quality Manual, reviewing the Peak Analytical Standard Operating Procedures, undergo hands on training on all equipment and procedures and finally they will be required to pass a performance evaluation using performance evaluation samples prepared by the Scientific Director. To pass, a performance evaluation test results must be within 6% of the known value of the samples. No analyst will be allowed to perform tests for a customers until they have passed the performance evaluation test.

See attached Peak Analytical Quality Assurance Manual for more specific details on training and quality assurance practices.

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Scientific Director (3 AAC 306.630):

All marijuana testing facilities must employ a scientific director with responsibilities and qualifications set out in 3 AAC 306.630 and 3 AAC 306.640(b).

Name the scientific director and describe how he/she meets the qualifications set out in 3 AAC 306.630(b).

Kara Jurczak, P.E., is proposed to serve as the laboratory's Scientific Director. Ms. Jurczak's qualifications are:
1. Professional Engineer in the State of Alaska,
2. Bachelor of Science Degree, Northern Arizona University, 2003
3. Master of Science Degree, Colorado State University, 2006
4. Five years of post-baccalaureate laboratory work experience as follows:
   a. Laboratory Assistant, R&M Engineering Inc., 2003-2004
   b. Laboratory Instructor, Colorado State University, 2004-2005
   c. Laboratory Assistant, R&M Engineering Inc., 2005-2008

Although Ms. Jurczak's degrees are in Environmental Engineering (BS) and Civil Engineering (MS) that differ slightly from the Scientific Director requirements as stated in 3 AAC 306.630, the intention of the law is met. Environmental engineering is closely related to chemical engineering; the difference being which industry the chemistry principles are applied to. Further, as a Professional Engineer Ms. Jurczak has held a position with the City of Ketchikan requiring a high degree of public trust since 2008. Therefor, this application requests that the Board consider the aggregate of her educational, professional and experience credentials as evidence of her ability to reliably perform the duties of Scientific Director.

Section 5 – Security

Review the requirements under 3 AAC 306.650, and identify how the proposed establishment will meet the listed requirements.

Chain of Custody (3 AAC 306.650):

Describe how the marijuana testing facility will meet the chain of custody requirements as listed in this section:

A chain of custody will be prepared for all samples or sample batches either prior to or upon receipt. Commercial clients must prepare a Transport Manifest through the METRC system and a sampling statement prior to delivery. The chain of custody will contain sections pertaining to:
1. minimum sample requirements for each test offered and each matrix of marijuana or marijuana product
2. storage requirements for samples
3. document condition of packages and seals
4. document condition and amount of samples provided
5. document each person handling the original samples, aliquots and extracts
6. document transfer of samples, aliquots and extracts to another testing facility for additional testing or at the request of the originating facility
7. document the date and amount for disposal of samples, aliquots and extracts

Peak Analytical LLC will maintain a current list of Authorized Personnel on the laboratory server. The facility will be locked and monitored via video surveillance and alarm system during non-working hours. Storage of samples will take place within restricted access areas. Storage of samples will occur at 4 degrees Celsius. A restricted access area will be used to log in and aliquot samples. All sample information and chain of custody will be stored on the laboratory server.

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Section 6 – Reporting and Records Retention

Applicants should review 3 AAC 306.670 – 3 AAC 306.675 and be able to answer "Agree" to all items below.

Reporting, Verification (3 AAC 306.670):

The marijuana testing facility applicant:	Agree	Disagree
Has read, understands, and agrees to the reporting requirements outlined in 3 AAC 306.670(a)	☑	☐
Has read, understands, and agrees to the reporting requirements outlined in 3 AAC 306.670(b)	☑	☐

Records Retention (3 AAC 306.675):

The marijuana testing facility applicant:	Agree	Disagree
Has read, understands, and agrees to the records retention requirements listed in 3 AAC 306.675	☑	☐

I declare under penalty of perjury that I have examined this form, including all accompanying schedules and statements, and to the best of my knowledge and belief find it to be true, correct, and complete.

Signature of licensee

Kara Jurczak

Printed name

Subscribed and sworn to before me this 21 day of December, 2017.

Notary Public
MELINDA ESPOSITO
State of Alaska
My Commission Expires Jun 16, 2018

Notary Public in and for the State of Alaska.

My commission expires: 6-16-18

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(Additional Space as Needed):

Continuation from Page 3

Microbial Testing: Concentrates (Salmonella)
1.Allow plates (Hardy Diagnostics Compact DryTM SL) to reach room temperature
2.Label plates with sample identifiers and "blank"
4.Inoculate the control blank with 1 ml sterile water
5.Inoculate sample plates by pipetting 1 g of each sample directly to the dry plate well, keep sample 1 cm from the edge of the well, being careful not to touch the surface of the matrix with the pipet tip.
a.Use one sterile pipet for each sample
b.When the sample is viscous, pipetting the sample on several points on a plate may be needed
6.Pipet 1 ml of sterilized water at the opposite side of the plate from where the specimen was dropped. Once dispensed, the sterile water will automatically diffuse across the surface by capillary action to form a gel; manual spreading of the inoculum is discouraged
8.Invert the plates and incubate, upside down with the medium on top, at 41-43 degrees C for 20-24 hours

Microbial Testing: Concentrates (Aspergillus)
1.Allow plates (Hardy Diagnostics Compact DryTM YMR) to reach room temperature
2.Label plates with sample identifiers and "blank"
4.Inoculate the control blank with 1 ml sterile water
5.Inoculate sample plates by pipetting 1 g of each sample directly to the dry plate well, keep sample 1 cm from the edge of the well, being careful not to touch the surface of the matrix with the pipet tip.
b.When the sample is viscous, pipetting the sample on several points on a plate may be needed
6.Pipet 1 ml of sterilized water at the opposite side of the plate from where the specimen was dropped. Once dispensed, the sterile water will automatically diffuse across the surface by capillary action to form a gel
8.Incubate inverted plates at 25-30 degrees C for 48-72 hours (+/- 2 hours)

Residual Solvents Testing:
1.Weigh 20 mg of oil, wax or other cannabinoid concentrate to a 20 ml headspace vial
2.Prepare calibration standards, at least one air blank (empty vial) and two DMSO blanks
3.Add the samples to the headspace sampler in this order: air blank, DMSO, standards (high to low for standard curve), sample, DMSO
4.Run the analysis including calibration

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PEAK ANALYTICAL
STANDARD OPERATING PROCEDURES
MANUAL

REVISION 2

May 2018

Peak Analytical LLC
2208 Tongass Avenue
Ketchikan, Alaska 99901

APPROVAL

SCIENTIFIC DIRECTOR-SIGNATURE:

SCIENTIFIC DIRECTOR-PRINT: Kara Jurczak

DATE: 5/1/2018

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SAMPLE PREPARATION

Homogenization

Plant Matrices

1. Obtain 1-2 g and remove all parts which will not be consumed
2. Grind to a fine powder with pestle and mortar

Concentrate: Oil

No homogenization required

Concentrate: Kief, Dry-Sift, Bubble Hash, Honeycomb Wax

Stir until well mixed

Concentrate: Shatter or Brittle/Glassy Items

Grind to a powder

Hard Candy

Grind with pestle and mortar

Baked Goods

Grind with pestle and mortar

Gummy / Caramel Candy

Cut into slim pieces

Chocolate Bars

Chop into small pieces

Carbonated Beverages

Degas for 30-minutes on 3-D rocker

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POTENCY TESTING

Extraction and Cleanup

Plant Matrices and Concentrates

1. Mark extraction vial with sample identifier
2. Tare extraction vial without cap
3. Into extraction vial weigh 0.500 g (+/- 0.050 g) homogenized sample
    a. Concentrates: weigh 0.100 g (+/- 0.010 g) of homogenized sample
        1 Record exact amount weighed, analyst name, date on Sample Preparation Sheet
4. Using a pipette, add 10 mL of MeOH on top of the sample, then cap
5. Vortex for 4 minutes
6. Centrifuge 1 minute at 5000 rpm
7. Filter supernatant with syringe filter into clean transfer vial
8. Write sample identifier on 1.8 ml autosampler vial
9. Dilute at 1:100 dilution:
    a. Use a 1000 µl class-A glass syringe and add 1000 µl of MeOH to the autosampler vial
    b. Use a 10 µl class-A glass syringe to remove exactly 10 µl of MeOH from the autosampler vial
        i. 990 µl is now remaining in the vial
    c. Use a 10 µl class-A glass syringe to transfer 10 µl of the filtered extract, then cap the vial
    d. Mix thoroughly by shaking
10. Use the 1.8 ml autosampler for analysis.

11. The remaining extracts should be stored until analysis is completed
    b. If analysis will not occur on the same day the analytical sample should be stored at 4 degrees C
    c. After analysis is completed, re-cap the vial with a new cap to avoid sample evaporation and store at 4 degrees C

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Edible Matrices

1. Mark extraction vial with sample identifier
2. Tare extraction vial without cap
3. Into extraction vial weigh 0.500 g (+/- 0.050 g) homogenized sample
    a. Carbonated beverages: add 10 ml degassed sample
4. Add 5 ml of reagent water and hydrate for 30-60 mins using orbital rocker
5. Add 5 ml of a polar solvent: Acetonitrile (ACN containing 1% Acetic Acid) or MeOH
6. Add one QuEChERS Q-sep™ Original Unbuffered Method packet
7. Vortex for 4 minutes
8. Centrifuge for 5 minutes at 5000 rpm
9. Transfer approximately 1 ml of the supernatant to a dSPE tube
10. Vortex for 1 minute
11. Centrifuge for 4 minute at 5000 rpm
12. Dilute at 1:20 dilution:
    a. Use a 1000 µl class-A glass syringe and add 950 µl of MeOH to the autosampler vial
    b. Use a 100 µl class-A glass syringe to transfer 50 µl of the supernatant being careful not to bump the vials during transfer and to avoid any solids, then cap the vial
    c. Mix thoroughly by shaking
12. Use the 1.8 ml autosampler for analysis.

13. The remaining extracts should be stored until analysis is completed
    d. If analysis will not occur on the same day the analytical sample should be stored at 4 degrees C
    e. After analysis is completed, re-cap the vial with a new cap to avoid sample evaporation and store at 4 degrees C

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POTENCY TESTING

Calibration (HPLC)

1. Standard dilutions for calibration curve
2. A four point calibration curve must be diluted according to Table 1 below using Certified Reference Materials (CRM)
    d. Class-A syringes are used
    e. The amount measured must be between 10% and 100% of the syringe volume
    f. Standard MeOH is added to 1.8 ml autosampler vials
3. A Working Stock solution is created by adding the individual cannabinoid standards to achieve a final concentration of 100 µg/ml
    a. The stock solution is created by adding 100 µl of each individual standard with a final volume of 1000 µl in MeOH
        i. Example: if seven cannabinoids are being analyzed then 100 µl each cannabinoid standard is added creating a volume of 700 µl. The remaining volume (300 µl) is MeOH. This results in a final concentration of 100 µg/ml

Table 1

Working Stock (dilution above)	µl (100 µg/ml Working Stock) added	µl MeOH added	Concentration of Standard
100 µg/ml	400	0	100 µg/ml
100 µg/ml	250	250	50 µg/ml
100 µg/ml	100	900	10 µg/ml
100 µg/ml	50	950	5.0 µg/ml

4. Analyze all four standards above from low to high concentration
5. After standards have been analyzed create a calibration curve with correct unit
    a. The calibration is typically linear and forced through zero
6. The correlation coefficient must be > or = 0.990 for each cannabinoid standard. if correlation coefficient is less than 0.990 corrective actions must be taken
    a. Typical corrective action is:
        i. Check baseline integrations for improper integration of peaks
        ii. Backflush column by reversing flow and allowing liquid to flow in waste container

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iii. Replace guard-column
iv. Replace any parts according to manufacturer specifications
v. Re-run calibration curve

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POTENCY TESTING

Analysis of Cannabinoids (HPLC)

1. The sample batch must include a continuing calibration verification standard (CCV) after every 10 samples. The initial calibration is considered as a valid CCV
    a. The concentration of the CCV is a mid-point of the calibration curve
        i. According to Table 1 it can be either the 10 µg/ml or 50 µg/ml standards
    b. CCV criteria: The CCV calculated amount for each cannabinoid must not deviate more than 15% from the known concentration

    $$\text{Percent Deviation} = 100 * \frac{(\mu g/ml\ CCV - \mu g/ml\ known\ concentration)}{(\mu g/ml\ known\ concentration)}$$

    c. If the CCV fails corrective action must be taken:
        i. All samples prior to and after the failed CCV must be re-analyzed with a valid calibration
        ii. Follow the corrective actions specified in 6(a) above
2. Load chromatogram in data analysis program
    a. Check baseline integrations for irregular integration of peaks
    b. Review analysis report and ensure amounts are within linear range
        i. Linear range is defined as the concentration from low to high of the calibration curve. If Table 1 is used, the linear range is from 5.0 to 100 µg/ml
        ii. If the calibration curve is forced through zero with an acceptable correlation coefficient, the amounts can be less than linear range if the signal to noise is greater than 10:1. This is noted in the final report.

<u>Eluent Order</u>
CBV
CBD-A
CBD
CBN
THC
THC-A

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POTENCY TESTING

Interpretation (HPLC)

In ChemStation:

1. In the Sequence Table, enter sample weight used in mg
2. Choose ESTD% report type using Area
    a. This will read out % (by weight) of each cannabinoid
3. THC_{Total} = THC + 0.8770*(THC-A)

General calculation for the percent of each cannabinoid if the concentration is given in µg/ml:

$$\text{Percent \%} = 100 * \text{Dilution Factor} * \frac{\text{ml final volume}}{\text{mg extracted}} * \frac{1\ \text{mg}}{1000\ \mu\text{g}} * \frac{\mu\text{g}}{\text{ml}}$$

Example: For a 500 mg sample that has been extracted in 10 ml MeOH and analyzed at 1:100 dilution with a found concentration of 75.0 µg/ml

$$\textit{Percent \%} = 100 * 100 * \frac{10\ \text{ml}}{500\ \text{mg}} * \frac{1\ \text{mg}}{1000\ \mu\text{g}} * \frac{75.0\ \mu\text{g}}{\text{ml}} = 15.00\%$$

Example: If the same sample is analyzed with a dilution factor of 100 entered into chemstation

$$\textit{Percent \%} = 100 * \frac{10\ \text{ml}}{500\ \text{mg}} * \frac{1\ \text{mg}}{1000\ \mu\text{g}} * \frac{7500.0\ \mu\text{g}}{\text{ml}} = 15.00\%$$

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POTENCY TESTING

Instrumentation Setup (HPLC)

Chromatographic Conditions for Potency Analysis

Apparatus: Agilent Technologies Model 1100 HPLC with quaternary pump, degasser, auto-sampler and thermostatted column compartment.
Column: Restek Raptor ARC-18
Column Temperature: 50 Degrees C
Injection Volume: 5 or 10 µL (more volume might be need for certain analyses)
Wavelength: 230 nm

Mobile Phase:
A – H_20 (0.1% Formic Acid)
B – Acetonitrile (0.1% Formic Acid)
Flow Rate: 1.5 ml/min

Time:	%A	%B
0.00	25	75
7.00	0	100
11.00	0	100
11.10	25	75

Note: There will be a hold time at 100% B to ensure that the column is clean for the next injection. This could take up to 8 minutes.

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HPLC BEST PRACTICES

- Always use clean glass bottles for all solvents used on the instrument.
  Replace all solvents at least every two weeks, depending on solvent type replacement of solvents might need to be more frequent.
- Always use HPLC grade solvents for mobile phase, cleaning, and troubleshooting.
- Make sure samples to be analyzed are soluble in mobile phase being used and are free of particles via filtration.
- Purge the pump before each sample run (purge valve must be open) starting with 100%-line A for approximately 5-10 minutes at 2.5ml/min and then 100%-line B for approximately 5-10 minutes at 2.5ml/min; then switching to starting method gradient and purging for approximately 5-10 minutes at 2.5ml/min.
  Purge is considered sufficient when no bubbles are seen coming out of the purge waste line.
- Ensure that there is a steady baseline and column is conditioned prior to starting analysis. Pump pressure should be consistent prior to analysis and can be achieved by running for at least 20-30 minutes prior to analysis.
  No injections should be done until pump pressure has stabilized.
- Once a week flush all pump channels with 100% water to remove build up, do NOT pump through the column, pump to waste (Only necessary if your mobile phase contains salt or acid).
- Use of needle wash or needle seat backflush can decrease contamination from samples.
- Allow the detector to warm-up for at least 30 minutes prior to starting an analysis.
- When removing columns for storage flush the column in the suggested storage solvent (found in column manual), typically this is either 50/50 methanol/water OR 50/50 isopropanol/water.
  Make sure to seal column ends after removing.
- If powering down the entire system for an extended period, flush the entire system with water (without a column) to remove any buffer build-up.
- After each sequence run a cleaning method with mobile phase without any added buffer for at least 30 minutes (example: if you are running 75% methanol/25% water with 0.1% formic acid, your cleaning method should be 75% methanol/25% water without the 0.1% formic acid).

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MICROBIAL TESTING

Analysis (Standard Plate Counts)

Plant & Edible Matrices (analyzed for STEC)

1. Allow plates (Hardy Diagnostics Compact Dry™ EC) to reach room temperature
2. Using one sterile pipet or scoop for each sample, aliquot 1 ml or 1 g of homogenized test sample to a 99 ml pre-filled Dilu-Lok II™ dilution vial to yield a 1:100 dilution. Mix thoroughly by shaking 25 times
    a. May use Butterfield's phosphate-buffered water in lieu of Dilu-Lok II™
3. Transfer approximately 10-15 ml of diluted sample into a sterile centrifuge tube
    a. Centrifuge 2 minutes at 5000 rpm
    b. Option: may use a syringe filter instead of centrifuging
4. Label plates with sample identifiers and dilution factors
    a. Label one plate as "blank"
5. Remove plate lids
6. Inoculate the control blank with 1 ml buffered water
7. Inoculate sample plates by pipetting 1 ml of each sample dilution directly to the center of a dry plate well, being careful not to touch the surface of the matrix with the pipet tip. Once dispensed, the sample will automatically diffuse across the surface by capillary action to form a gel; manual spreading of the inoculum is discouraged
8. Replace plate lids
9. Invert the plates and incubate, upside down with the medium on top, at 35-37 degrees C for 24 hours
10. After incubation read the plates
    a. Only count STEC colonies
    b. A passing test has less than 1 CFU/g

Concentrates (Salmonella)

1. Allow plates (Hardy Diagnostics Compact Dry™ SL) to reach room temperature
2. Label plates with sample identifiers
    a. Label one plate as "blank"
3. Remove plate lids
4. Inoculate the control blank with 1 ml sterile water
5. Inoculate sample plates by pipetting 1 g of each sample directly to the dry plate well, keep sample 1 cm from the edge of the well, being careful not to touch the surface of the matrix

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with the pipet tip. Once dispensed, The dispensed sample will remain at the point of inoculation and will not spread across the whole plate

a. Use one sterile pipet for each sample
b. When the sample is viscous, pipetting the sample on several points on a plate may be needed

6. Pipet 1 ml of sterilized water at the opposite side of the plate from where the specimen was dropped. Once dispensed, the sterile water will automatically diffuse across the surface by capillary action to form a gel; manual spreading of the inoculum is discouraged
7. Replace plate lids
8. Invert the plates and incubate, upside down with the medium on top, at 41-43 degrees C for 20-24 hours
9. After incubation read the plates
    a. Only count Salmonella colonies
        i. Green or blue-green colonies with or without black centers and on a yellow background are indicative of Salmonella
        ii. Bacteria other than Salmonella may grow and produce a yellow background at the site of inoculation, such as Pseudomonas spp., but will not produce green colonies
    b. A passing test has less than 1 CFU/g

<u>Concentrates (Aspergillus)</u>

1. Allow plates (Hardy Diagnostics Compact Dry™ YMR) to reach room temperature
2. Label plates with sample identifiers
    a. Label one plate as “blank”
3. Remove plate lids
4. Inoculate the control blank with 1 ml sterile water
5. Inoculate sample plates by pipetting 1 g of each sample directly to the dry plate well, keep sample 1 cm from the edge of the well, being careful not to touch the surface of the matrix with the pipet tip. Once dispensed, The dispensed sample will remain at the point of inoculation and will not spread across the whole plate
    a. Use one sterile pipet for each sample
    b. When the sample is viscous, pipetting the sample on several points on a plate may be needed
6. Pipet 1 ml of sterilized water at the opposite side of the plate from where the specimen was dropped. Once dispensed, the sterile water will automatically diffuse across the surface by capillary action to form a gel; manual spreading of the inoculum is discouraged

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7. Replace plate lids
8. Incubate inverted plates at 25-30 degrees C for 48-72 hours (+/- 2 hours)
9. After incubation read the plates
    i. Blue colored colonies indicate yeast
    ii. Cottony colonies with characteristic colors are molds
    iii. Count all colonies to obtain the total yeast and mold count
    iv. Greater than 300 CFU will cause the entire growth area to become green/blue. In this case dilute the sample.

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RESIDUAL SOLVENTS TESTING

Calibration (HS GC-FID Full Evaporation Technique [FET])

Concentrates

1. A 5 point calibration must be performed for every sample set
2. Standard dilutions for calibration curve
3. Prepare standards for a five point calibration curve using 100 µg/ml commercially available standards in 2 ml vials
    a. Prepare standards for all analytes desired (isobutane, n-Butane, n-Heptane, benzene, toluene, n-Hexane, zylenes)
    b. All standard preparations shall use a gas-tight Hamilton syringe or equivalent
    c. The standard should be prepared in a solvent less volatile than the analyte that will not interfere with the solvents of interest
        i. Acceptable solvents are dimethyl sulfoxide (DMSO) and dimethyl acetamide (DMA)
            1. DMSO shall be tested for interference before used in standards
4. For Full Evaporation Technique, the 100 µg/mL (in DMSO) commercially available standard may be aliquoted directly to the headspace vials to achieve the standard points desired according to Table 2 below.

Table 2

µL of Stock	Concentration - µg/mL	Equivalent ppm in 100 mg sample of Concentrate	Equivalent ppm in 200 mg sample of Concentrate
10	100	100	50
5	50	50	25
2.5	25	25	12.5
0.5	5	5	2.5
0.1	1	1	0.5

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RESIDUAL SOLVENTS TESTING

Analysis (HS GC-FID Full Evaporation Technique [FET])

1. Accurately weigh 20 mg of oil, wax or other cannabinoid concentrate to a 20 ml headspace vial
    a. Record the weight and cap tightly
2. Prepare at least one air blank (empty vial) and two DMSO blanks (10 µl in each vial)
3. Add the samples to the headspace sampler in this order: air blank, DMSO, standards (high to low for standard curve), sample, DMSO
    a. Make sure all vials are crimped well to prevent leaks
4. Run the analysis
5. After standards have been analyzed create a calibration curve with correct unit
    a. The calibration is typically linear and forced through zero
6. The correlation coefficient must be > or = 0.990. if correlation coefficient is less than 0.990 corrective actions must be taken
    a. Typical corrective action is:
        i. Check baseline integrations for improper integration of peaks
        ii. Check for interference in solvents
        iii. Backflush column
        iv. Replace guard-column
        v. Replace any parts according to manufacturer specifications
        vi. Re-run calibration curve

7. Identify and integrate the peaks
    a. The elution order is
        i. Isobutene
        ii. Butane
        iii. Methanol
        iv. Pentane
        v. Ethanol
        vi. Acetone
        vii. Isopropanol
        viii. N-hexane
        ix. Chloroform
        x. Benzene
        xi. Heptane

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xii. Toluene
xiii. DMSO or DMA diluent peak
    1. Except the diluent peak, all peaks should elute within 3 to 7 minutes

8. Calculate the average standard area response for the peaks identified or expected in the unknown sample. Calculate the relative standard deviation (RSD) for the standards.
    a. If the RSD is ≤ 15% then the system is suitable for calculation of sample concentration
    b. If the RSD is > 15% troubleshoot the chromatography and prepare a fresh set of standards and samples
9. If system suitability is met, calculate the residual solvent concentrations for the peaks identified based on the linear plot calibration equation produced

   Note: if an increased detection limit is required, as might be the case for butanes and heptanes, option 1) is to increase the sample size which will increase detection limit proportional to the sample increase; option 2) add a 500 ppm and 1000 ppm point to the calibration curve as appropriate for the analyte based on its detection limit

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RESIDUAL SOLVENTS TESTING

Interpretation (HS GC-FID Full Evaporation Technique [FET])

$$y = m * x + b$$

where y = peak area of solvent / peak area of STD
m = slope of the line
x = concentration of solvent / concentration of STD
b = intercept

Solving the equation for the unknown concentration of solvent yields:

$$\text{Concentration of Solvent} = \frac{[y-b] * [\text{STD concentration}]}{m}$$

The linear plot determines the values of m and b, the value of STD concentration is known and included in the method, and the value of y is determined from the sample runs.

For each sample run the instrument will calculate and report the concentration of residual solvent in mg/ml. This value will be used to determine the concentration of residual (mg/ml) in the original gas sample using the following equation:

$$\text{Concentration of solvent} = \frac{[\text{conc gas in extract sample}] * [\text{volume STD}]}{\text{mass of original sample used}}$$

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RESIDUAL SOLVENTS TESTING

Instrumentation Setup (HS GC-FID Full Evaporation Technique [FET])

GC-FID Settings (Agilent 6890)

Column: RXi-624Sil MS, 30 m, 0.32 mm ID, 1.8 µM Cat# 13870
Sample: Residual Solvent mix
Diluent: Dimethyl sulfoxide (DMSO)
Injector Port: 250 °C with 10:1 split
Liner: 2.0 mm ID straight inlet liner
Carrier Gas: Nitrogen using constant flow mode
Linear Velocity: 65 cm/sec
Carrier Flow Rate: 59.1 ml/min
Oven: 35 °C for 1.5 min, ramp to 300 °C at 30 °C /min and hold 2 min.
Detector: FID at 350 °C
Make-up Gas: Nitrogen
Make-up Gas Flow Rate: 45 ml/min
Hydrogen Flow: 40 ml/min
Air Flow: 450 ml/min
Data Rate: 20 Hz

Headspace Sampler Settings (Tekmar 7000) – Method 1

Sample Temp: 140°C
~~Needle Temp: 140°C~~ N/A on this HS
Valve Oven: 160 °C
Loop: 160 °C
Transfer Line: 160 °C
Vial Pressure: 20 psi
GC Cycle Time: 25 min
(time for GC to cool down after a run)
Vial Eq. Time: 30 min
Pressurize Time: 5.0 min
Equilibration Pressure: 0.05 min
Loop Fill Time: 2.0 min
Loop Eq. Time: 0.1 min
Inj. Time: 1.0 min
*(these settings may be modified to adjust instrument response)

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RESIDUAL SOLVENTS TESTING

Regulatory Limits [3 AAC 306.645(B)(3)]

Substance	Acceptable Limit per Gram
Butanes	Less than 800 PPM (µg/g)
Heptanes	Less than 500 PPM (µg/g)
Benzene	Less than 1 PPM (µg/g)
Toluene	Less than 1 PPM (µg/g)
Hexane	Less than 10 PPM (µg/g)
Total xylenes	Less than 1 PPM (µg/g)

CONVERSIONS

PPM = mg/l = µg/ml

PPM = mg/kg = µg/g

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GC-FID BEST PRACTICES

Instrument and Column

- Always keep the instrument powered on; at the end of a sequence set oven temperature to 100°C and maintain constant flows through the inlet, column, and detector.
    - If shutting down for a week or longer, bake out instrument, for at least 20 minutes, with high temperature and proper flows to rid the system of any sample build-up. Once baked-out, power down ChemStation, the GC, and turn off all gas cylinders
- Verify gas flows, pressures, and temperatures of all GC components prior to starting a sequence.
- Check the liner and septa at least once a week (depending on frequency and number of injections).
- Once every two weeks bake out column/oven, inlet, and detector with proper gas flows and pressures and a split ratio of 100:1. (Inlet at 300° C; column/oven at 250°C; detector at 350°C).
- Always wear gloves when handling the column, column nuts, and ferrules.
- When installing a new column, make sure to condition it at 20° C higher than the maximum method temperature for 1 hour.
- When removing a column and replacing with a different column, immediately cap both ends before placing in the column box for storage.
- Make sure there is a clean cut of the column before installing, cut should be straight across.
- Make sure the column is only 4-6mm above the ferrule/column nut on the inlet side.
- Make sure the column is deadened against the jet and then pulled back slightly so that the column isn't resting on the top of the jet on the detector side.
- Check the gold seal to verify there are no particulates stuck to the top of the gold seal.
    - If particulates are seen, replace the gold seal or clean with a cotton-swab and methanol.

Headspace Injection

- Verify gas flows, pressures, and temperatures of all headspace components prior to starting a sequence.
- Verify that the transfer line is at least 10° warmer than the headspace oven and loop.
    - The loop should be the same temperature or warmer than the headspace oven (example: oven 140°, loop 140°, transfer line 150° OR oven 140°, loop 150°, transfer line 160°)

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- Once every two weeks bake out the headspace with proper gas flows and pressures and temperatures at the maximum possible for the instrument.
    - Make sure to bake out headspace and GC at the same time.

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PEAK ANALYTICAL
QUALITY ASSURANCE MANUAL

REVISION 1

March 2018

Peak Analytical LLC
2208 Tongass Avenue
Ketchikan, Alaska 99901

APPROVAL

SCIENTIFIC DIRECTOR-SIGNATURE:

SCIENTIFIC DIRECTOR-PRINT: Kara Jurczak

DATE: 3/1/2018

This Quality Assurance Manual is prepared to be compliant with ISO 17025.

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INTRODUCTION

Statement of Management Philosophy
Peak Analytical is committed to providing the highest quality services and customer service to our clients. The execution of this commitment is the responsibility of each employee in the organization. The requirements of this Quality Assurance Manual (QAM) apply to each member of the laboratory at every level.

The Peak Analytical Quality Assurance Manual provides the guidance document to all personnel in fulfilling the responsibilities to provide the highest quality, regulatory defensible data. It serves as the statement of quality and commitment by the laboratory and all the personnel at Peak Analytical.

Peak Analytical insures that no economic incentives will compromise data quality. Management will not place any undue production pressure (including production incentives) on the staff of the laboratory that may compromise data quality.

The Laboratory Scientific Director, with the active support of Laboratory Management is responsible for the implementation of the Quality Assurance Manual. They have the authority, and obligation, to stop work if it fails to meet the exacting criteria defined in this Manual.

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QUALITY SYSTEM

Quality Policy Statement
The objective of the quality system and the commitment of management is to consistently provide our customers with data of known and documented quality that meets their requirements and regulatory requirements. Our policy is to use good professional practices, to maintain quality and uphold the highest quality of service. The laboratory ensures that personnel are free from any commercial, financial, and other undue pressures, which might adversely affect the quality of work. This policy is implemented and enforced through the unequivocal commitment of management, at all levels, to the Quality Assurance (QA) principles and practices outlined in this manual. However, the primary responsibility for quality rests with each individual within the laboratory organization. Every laboratory employee must ensure that the generation and reporting of quality analytical data is a fundamental priority. Every laboratory employee is required to familiarize themselves with the quality documentation and to implement the policies and procedures in their work. The laboratory maintains a strict policy of client confidentiality.

Quality Assurance Manual
This Quality Assurance Manual (QAM) details the operations to provide the highest quality and defensible data at Peak Analytical. It outlines policies with a focus on the Quality Assurance/Quality Control (QA/QC) procedures implemented at the laboratory. These are based on EPA guidelines, State guidelines and industry standard practices; often referred to as good laboratory practice.

This document specifies the operations in the Laboratory to assure the accuracy, precision, completeness, representativeness, and comparability of all analyses performed in the Laboratory. The purpose is to assure that all data being reported are of the highest quality, and all documentation supporting this is available.

Quality Assurance
Quality Assurance is a system for the integration of planning, assessment, and improvement efforts related to quality in all sections at the laboratory. This includes all actions performed by laboratory personnel and documentation of laboratory performance. This is designed to identify and correct problems in the analytical process or demonstrate statistical control of a process in the laboratory. The objective of a quality assurance program is to reduce measurement errors to specified limits and produce results of acceptable and consistent quality.

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Quality Control

Quality Control is daily procedures and actions taken by the laboratory to assure integrity in sample handling, performance of testing and analysis, record maintenance and data processing. It is a systematic approach to inspection, testing and corrective actions applied to processes estimating sample quality and to determine any changes required to maintain or achieve a specific level of quality.

Quality Control practices include the analysis of applicable QC samples with each set of test samples. These include calibration standards, blanks, continuing calibration verification standard (CCV), duplicate sample analysis and spiked samples.

Additional quality control measures include:

1) Strict adherence to Good Laboratory Practices (GLP)
2) Consistent use and application of Standard Operating Procedures (SOP's)
3) Adherence to specific protocols for measurement processes
4) Use of highly trained and qualified personnel
5) Use of reliable and maintained equipment
6) Supervision of operations by senior personnel and/or management
7) Specific acceptance criteria for all Quality Control parameters
8) Procedures for identification and correction of out of control data

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CLIENT CONFIDENTIALITY

Peak Analytical adheres to strict client confidentiality policy. This includes distribution of analytical results and client privileged information only to approved parties. No information is provided over the phone without confirmation as to the recipient's identity. Phone numbers, facsimile numbers and email addresses are confirmed on the chain of custody or verified independently.

All electronic data (storage or transmissions) are kept confidential, based on technology and laboratory limits, as required by client or regulation.

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PERSONNEL, TRAINING AND DATA INTEGRITY

Training Requirements
A vital requirement in quality assurance programs is adequate training of analysts prior to sample analysis being performed. All quality related activities performed in the laboratory will be accomplished by qualified analysts, with documented experience, degrees, training, and education. Activities that require training include laboratory safety, sample receiving, sample preparation, standard or reagent preparation, calibration of instruments or tools, data validation, and record/document management. All analysts will be trained in QA/QC principles in addition to scientific methodology. This will allow achievement of skill levels that meet standards of quality.

Initial Orientation
All analysts undergo initial Quality Assurance training. Included is a discussion of their ethical and legal responsibilities, including potential punishments and penalties for improper or illegal actions, regulatory information and general training on duties and requirements of the analysts. All analysts are required to read the Quality Assurance Manual. Analyst signatures are required to document their understanding and adherence to all Peak Analytical Quality Assurance and ethics policies.

Scientific Training
Prior to beginning sample analysis, analysts must demonstrate knowledge of method requirements by passing performance evaluation requirements. These procedures may be specific methods or laboratory Standard Operating Procedures.

This demonstration includes:
1) Reading and review of the SOP and identification of any method exception criteria applicable.
2) Acceptable preparation and analysis of a Calibration Curve (if required by method), which must meet statistical requirements of the specific method.
3) Acceptable preparation and analysis of a performance evaluation test with samples prepared by the Scientific Director. To pass, a performance evaluation test results must be within 6% of the known value of the samples.
4) Demonstration of understanding of good laboratory practices, HPLC Best Practices and GC-FID Best Practices.

The Scientific Director or Laboratory Manager are responsible for training of analysts.

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Quality Assurance Training
General training in requirements of the laboratory QAM is required of all laboratory personnel. Training will address regulatory requirements, basic and advanced quality control concepts and practices, individual responsibilities of the scientific staff, reporting and correction of non-conformance, performance evaluations and laboratory documentation.

Data Integrity Training
Data integrity training shall be performed for all employees. In addition, all employees will be required to read and sign the Peak Analytical Ethics Agreement during their initial employment orientation.

Data integrity training shall be documented and copies of signed ethics agreements will be kept in each employee's personnel file. The data integrity training shall include definition of ethical behavior, penalties for acting unethically, specific examples of fraudulent behaviors, the obligations of the company and of the employees to maintain an ethical workplace and mechanisms for confidential reporting of ethical breaches.

Code of Ethics
Peak Analytical maintains a program of ethical conduct. All employees must adhere to the following statement of ethical compliance, insuring that all data is of sound quality free from any intentional bias or fraudulent activity.

I understand the high ethical standards required of me with regard to the duties I perform and the data I report in connection with my employment at Peak Analytical. I have read and understand the Laboratory Quality Assurance Manual and will adhere to all Quality Control policies and procedures including the Peak Analytical Code of Ethics.

I agree to the following:
I will not intentionally report data values that are not the actual values observed or measured.
I will not intentionally report dates and times of data analysis that are not the actual times the data analysis was conducted.
I will not condone any accidental or intentional reporting of invalid data by other employees and immediately report the occurrence to my supervisor.
I will immediately report any accidental reporting of invalid data to my supervisor.
I will keep all client data and information confidential.
I understand that improper, unethical or illegal actions are not permitted and possible punishments and penalties include verbal or written reprimands, unpaid time off, dismissal and potentially legal action including fines and incarceration.

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ACCOMMODATIONS AND ENVIRONMENTAL CONDITIONS

Facilities

Peak Analytical occupies 1240 square feet of space, located in Ketchikan, Alaska. Office space occupies approximately 200 square feet of the space. A reception room occupies approximately 130 square feet. The remainder of space is allocated to analytical laboratories and employee facilities.

The laboratory is planned as a "single room" facility, allowing interaction between scientific staff in various areas. This allows peer to peer interaction and provides a forum for discussion of scientific facets of jobs currently in the analytical process.

The environmental conditions of the laboratory such as temperature are maintained and monitored where applicable to the performance of the test methods. The laboratory has been designed to provide adequate and unencumbered bench space for each analysis. The aisles and work spaces have been designed to provide clearance for safe conduct of samples and equipment throughout the lab.

The facility is secure and access to the building is monitored at the front entrance. The building is equipped with smoke and burglar alarm systems, motion detectors and security cameras.

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EQUIPMENT

Inventory
Peak Analytical maintains analytical equipment inventories sufficient to perform analysis requested routinely by clients and as required by State administrative codes.

Up-to-date instructions on the use and maintenance of equipment (including any relevant manuals provided by the manufacturer of the equipment) are readily available for use by laboratory personnel. All equipment is calibrated or checked before being placed into use to ensure that it meets laboratory specifications and the relevant standard specifications. Test equipment, including hardware and software, are safeguarded from adjustments which would invalidate the test results measures by limiting access to the equipment and using password protection where possible.

Equipment that has been subject to overloading, mishandling, given suspect results, or been shown to be defective or outside specifications is taken out of service, isolated to prevent its use, or clearly labeled as being out of service until it has been shown to function properly. If it is shown that previous tests are affected, then procedures for non-conforming work are followed.

Each item of equipment and the software used for testing and significant to the results is uniquely identified and records of equipment and software are maintained. This information includes the following:

1) Identity of the equipment and its software.
2) Manufacturer's name, type identification, serial number or other unique identifier.
3) Manufacturer's instructions, if available, or a reference to their location.
4) Dates, results and copies of reports and certificates of all calibrations, adjustments, acceptance criteria.
5) Maintenance plan where appropriate, and maintenance carried out to date.
6) Documentation on all routine and non-routine maintenance activities.
7) Any damage, malfunction, modification or repair to the equipment.
8) Date received and date placed into service (if available).

Analytical equipment list as of the date of the revision of the QAM is listed below:

- Agilent 6890A Plus Gas Chromatograph
- Agilent 6890 opt. 211 Flame Ionization Detector
- Agilent G1560A Split/Splitless inlet with EPC
- Tekmar 7000 Headspace Concentrator
- Tekmar 14-4401-00, 50k-position vial autosampler

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- Agilent 1100 (G1322A) HPLC with Vacuum Degasser & Solvent Tray Module
- Agilent 1100 (G1312A) Binary Pump
- Agilent 1100 (G1313A) Autosampler
- Agilent 1100 (G1316A) Thermostated Column Compartment
- Agilent 1100 (G1365B) Multi-wavelength Detector (MWD)

Benchtop equipment (support equipment) list as of the date of the revision of the QAM is listed below:

- Microscope- AM Scope SM-1TSZ-144S-5M
- Forced air oven- Quincy Lab 30GC
- Vortex- Benchmark BV 1000 Bench Mixer
- Centrifuge- Benchmark LC-8 5000
- Incubators- (3 each) Benchmark H2200-h
- Mechanical Shaker- Benchmark 3D Shaker
- Autoclave- Revolutionary Science Autoclave Steam Sterilizer
- Balance- Adam Equipment NBL 84e

Maintenance for Analytical Equipment

All equipment is properly maintained, inspected, and cleaned. Maintenance of analytical instruments and other equipment may include regularly scheduled preventive maintenance or maintenance on an as-needed basis due to instrument malfunction and is documented in instrument logs, which become part of the laboratory's permanent records.

Support Equipment

Support equipment includes the list above and various sample preparation devices.

Procedures for calibration and control of support equipment are contained in a separate SOP. All support equipment is maintained in proper working order and records are kept of all repair and maintenance activities, including service calls. All raw data records are retained to document equipment performance. These records include logbooks, data sheets, or equipment computer files. Support equipment is checked with a NIST traceable reference if applicable, to ensure they are operating within the expected range for the application for which the equipment is to be used. The results of calibration of support equipment are within specifications or (1) the equipment is removed from service until repaired, or (2) records are maintained of correction factors to correct all measurements.

Equipment Calibration

Calibration is the standardization of a measurement or instrument by use of a standard or standards, or another instrument to adjust to any variances in accuracy of measurement.

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Calibration Procedures

Calibration of instruments is performed using either internal or external standard calibration. In each case the following process occurs:

1) Calibration standards are prepared to encompass the linear range of the instrument, or the working range for the analysis being performed. The low standard is at or below the reporting limit, unless defined in the method SOP. Multiple points, covering the range selected, are analyzed. Calibration acceptance criteria are defined in each method or SOP, but typically require a 3 or 5 point calibration curve, which may include the blank for certain analysis. The curve must have a relative standard deviation (RSD) better than or equal to that required in the method, or have a correlation coefficient *r* of 0.990 or greater. If the RSD or *r* criteria are not met, the linear range is too large or re-calibration is required.
2) A second source, or a standard of known value, prepared or purchased from a source different from the calibration standards, must be analyzed. Its value must be within 20% of true value, or within the acceptance range given on the standard or within method acceptance criteria.
3) Continuing Calibration Verifications (CCV) must be analyzed at the frequency required by the method or the SOP. At a minimum, one must be run prior to analyzing a sample batch, and must be re-analyzed in accordance with the method. With the exception of Internal Standard calibrations, they must be analyzed at the end of the run batch to verify instrument response stability.
4) If CCV analysis does not meet method criteria, instrument maintenance may be performed. After maintenance, the CCV may be re-analyzed. If it fails to meet acceptance criteria again, the instrument must be calibrated again, and all samples analyzed from the last passing CCV must be re-analyzed.

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TEST METHODS AND METHOD VALIDATION

Test Methodologies
Specific test methods or SOPs are located in the Peak Analytical Standard Operating Procedure Manual. SOPs are reviewed annually and revised as needed; revision dates and summary of changes are documented in each SOP.

Method Variances
Occasionally, due to matrix interference or other influences, typical methods may be ineffective in providing acceptable quality data. Parameters such as detection limits, precision, specificity of individual components, etc., may be affected. In order to provide data meeting client specifications, variances to the method may be implemented by the laboratory. Documentation will describe the variance, document the reasons for the variance, and show that the conditions for the laboratory variance are similar to the expected conditions in the approved method.

Method Validation
A method is validated before it is put into use. Methods that are developed or modified in-house are detailed in method SOPs and are validated to the degree appropriate for their intended use. In general, the procedure is summarized below.

Performance Testing
Performance testing will be used to verify each analyst's ability to generate data of acceptable accuracy and precision. The laboratory confirms that it is capable of generating data of acceptable accuracy and precision on all methods before employing them. Performance testing is documented and kept in the personnel files for each analyst. Performance testing is performed for each analyte whenever the method, analysts, analytes, or instrument type is changed. The Scientific Director certifies that scientific staff members in their area of expertise are trained and authorized to perform all tests.

On-Going (or Continued) Proficiency
After the initial performance test is completed, on-going proficiency is maintained and demonstrated annually through the analysis of either single-blind samples, performing another performance test, or use of four consecutive laboratory control samples compared to pre-determined acceptance limits for precision and accuracy. This is documented in the personnel file of each analyst.

Estimation of Uncertainty
Estimation of uncertainty consists of the sum (combining the components) of the uncertainties of the numerous steps of the analytical process, including, but not limited to,

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sample plan variability, spatial and temporal sample variation, sample heterogeneity, calibration/calibration check variability, extraction variability, and weighing variability. The laboratory estimates uncertainty using the standard deviation calculated from routine quality control samples.

Laboratory-Developed or Non-Standard Method Validation
Laboratory developed, modified standard methods, and non-standard methods require method validation. Method Validation is the confirmation by examination and the provision of objective evidence that the particular requirements for a specific intended use are fulfilled. Where applicable, the laboratory validates non-standard methods, laboratory-designed/developed methods, standard methods used outside their published scope, and amplifications and modifications of standard methods to confirm that the methods are fit for the intended use.

Initial Test Method Evaluation
For chemical analyses, the initial test method evaluation involves the determination of the Limit of Detection (LOD), confirmation of the Limit of Quantitation (LOQ), an evaluation of precision and bias and an evaluation of the selectivity of the method.

Control of Data
All calculations and all relevant data are subject to appropriate checks in a systematic manner. Commercial off-the-shelf software (e. g. word processing, database and statistical programs) used within the designed application range is considered sufficiently validated when in-house programming is not used. The laboratory assures that computers and software are protected, maintained, and secure through measures such as documentation, locked access, and control of the laboratory environment.

Method Detection, Quantification, and Reporting Limits
Limit of Detection (LOD)
The LOD is an estimate of the minimum amount of a substance that an analytical process can reliably detect. An LOD is analyte and matrix specific and may be laboratory dependent.

Limit of Quantitation (LOQ)
LOQ is an estimate of the minimum amount of a substance that can be reported with a specified degree of confidence.

Method Detection Limit (MDL)
MDLs are defined as the minimum concentration of an analyte that can be identified, measured, and reported with 99% confidence that the analyte concentration is greater than zero. It also refers to the minimum concentration of an analyte that a method can detect reliably in either a given matrix or blank. MDL is calculated as:

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MDL = t(99%) (S)
where: t(99%) = students t value for 99% confidence limit, and n-1 degrees of freedom.
(S) = standard deviation of replicate analysis based on a minimum of 7 replicate analysis.

Limits of Quantification (LOQ)
LOQs are determined after MDL calculations are performed. Limits of Quantification are defined as the concentration of an analyte where quantification within specified limits of accuracy can occur. This is normally a multiple of the MDL (2.5 to 10X), and is commonly rounded for ease of reporting and use. PQL's are "assumed" factors applied to the MDL's to simulate real world matrix effects. These vary by matrix from 10X for groundwater to 10,000X for sludge and Hazardous Waste.

Reporting Limit (RL)
Reporting Limits are the standard concentration level at which the laboratory provides "less than" or Not Detected (ND). These levels are above the MDL, and represent the concentration that meets the laboratories LOQ and accuracy requirements. These limits are commonly grouped into ranges for ease of use of the data. Reporting limits are always above the MDL.

Frequency of Method Detection Limit Studies
MDL determinations, and evaluation of LOQ and Reporting Limits, are performed at:
1) Initial validation of a method in the laboratory, or upon validation of a specific instrument in the laboratory.
2) Upon validation of an analyst utilizing that instrument or performing a specific method.
3) Anytime there is a change in instrument sensitivity (normally defined as 35% change in sensitivity or response of an instrument).
4) At a minimum, biennially for each instrument and method.
5) These determinations can be simultaneous, i.e., both an analyst and an instrument can be validated at once. MDL's must be determined on each instrument the method will be performed on.

Procedures to be followed for the determination of MDL's are found in 40 CFR part 138 App. B. This procedure specifies the level for spiking MDL's at, and allows accurate determination of signal/noise levels. This determination is critical to accurate determination of MDL's. Spiking concentrations that are too large will lead to artificially low MDL's due to higher precision at levels above the signal noise. This is one of the most common reasons for non-reproducible MDL determinations. Spiking levels for MDL's should be 0.5-5X the calculated MDL, and should never exceed 10X the calculated MDL. Calculated MDL's exceeding the above guidelines need re-determinations prior to determinations of PQL or RL's.

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MEASUREMENT TRACEABILITY

Calibration is the standardization of a measurement or instrument by use of a standard or standards, or another instrument to adjust to any variances in accuracy of measurement.

Analytical Standards
Analytical Standards are utilized for calibration and preparation of quality control samples. These standards must be traceable to standard reference materials (SRM). Acceptable materials are:

1) EPA CRADA traceable materials.
2) NIST reference standards.
3) Standards made by vendors traceable to one of the above.

If none of the above are available, duplicate analysis of standards prepared separately is acceptable.

All standards must be stored under conditions that provide the largest amount of protection from deterioration and change. Traceability of the standards is established through statistical evaluation of the control sample, or analytical standard relative to NIST or CRADA reference materials. Criteria for acceptability of a new standard solution is: Triplicate analysis of a standard yields a Relative Percent Difference (RPD) of less than 15% when quantified against a SRM from one of the above sources.

Expiration Dates of Standards
All standards obtained from commercial sources as well as SRM's are dated upon receipt. This will be written in indelible ink upon the container received, as well as any container the standard may be transferred to. This information will also be recorded in the standards logbook. The expiration date of the standard is also noted and recorded as above. If no information as to expiration date is available, establishment of an expiration date based upon working knowledge of the standard is acceptable. Standards must be protected from degradation, deterioration and contamination by strict adherence to proper storage and handling procedures.

Stock and working standards are prepared fresh as required to maintain stability and are checked prior to every use for signs of contamination or deterioration (color formation, precipitation, concentration changes, additional peaks and leakage).

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Standards prepared as stock or working standards are labeled with name of compound or mixture, concentration, solvent, date and expiration date. All requirements for storage of the primary standard will be adhered to with the working or stock standard.

Inorganic Standards
Stock standards shall conform to the expiration date of the commercial standard. Working standards are prepared at a minimum of every 6 months, when standards show signs of degradation or when other factors require preparation.

Organic Standards
Liquid or solid stock standard holding times are the manufacturers recommendation or 6 months if no expiration date is available. Commercially prepared standard holding time is the manufacturers recommendation or 1 year if no expiration date is available.

Standard Preparation Equipment
Only Class A volumetric glassware, certified or calibrated pipets, and American Chemical Society grade solvents/reagents at a minimum, will be used for dilutions of primary standards.

Guidelines for Standard Preparation
1) Analytical reagent grade materials, in solution or neat, are utilized in the preparation of analytical and control standards. When possible, guaranteed assay materials with supporting documentation (chromatograms, assay results, etc.) are requested and filed.
2) Solvents utilized for dilution of standards are checked for contamination.
3) Analytical measuring tools (balances, pipettes, syringes, etc.) will be calibrated prior to use to assure accurate and acceptable measurements.
4) All data generated is documented immediately in standard preparation logbooks.
5) A sequential log number is assigned to the newly prepared standard. This number must be noted in the standard log, in preparation books, and on any storage vessels used for any aliquot.
6) Standards are analyzed prior to use on the instrument the standard is to be used on.
7) A standard obtained from another approved source is used as the traceability standard.
8) To maximize the precision of the result, both the new standard and the reference standard are analyzed on the same instrument, within the same batch, and as close in time as feasible.
9) Once the standard has passed QC evaluation, it is stored at the required temperature and condition until required for use.

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QUALITY OF TEST RESULTS

Quality Assurance Objectives

The purpose of Data Quality Objectives (DQO's) is to establish specific targets for accuracy (precision and bias). The initial DQO's for a method is to achieve improved precision and accuracy levels than specified in the method reference. Once sufficient data is produced, control charts are established for each analytical parameter appropriate, and the statistics produced are used as the new DQO's for the method, i.e., the mean and standard deviations become the DQO's for that test, provided they are better than the initial DQO's.

Peak Analytical's Quality Assurance Policy has the objectives to verify the production of high quality, defensible, and cost-effective data to its clients. While specific quality assurance plans and items will be required for particular projects, general guidelines are:

1) Data should be accurate within method specifications, and agree within limits with reference values.
2) Data should agree among individual measurements under similar conditions (comparability).
3) Data should be complete in terms of valid data produced as compared to planned (completeness).
4) Data should be comparable to prior data for evaluation, whether produced by Peak Analytical or not.
5) Data should be representative of the population of parameters measured.
6) Data should be reproducible under similar conditions at any time.
7) Quality Assurance programs should continually upgrade the quality of the laboratory performance.

The goal of the laboratory QAP is to produce data of defined and consistent quality. Guidelines are provided for the assessment and reporting of data quality parameters, and for the incorporation of such assessments into databases for evaluation and tracking.

Guidelines

1) Control of sample receiving, login of samples, and tracking of samples throughout the analytical process is maintained to ensure the integrity of the sample.
2) Documentation of instrument performance and preventative maintenance is maintained to allow review of factors effecting data produced during sample analysis.
3) Determinations of the quality of analytical work through analysis of quality control reference samples, duplicate analysis, matrix spike/spike duplicate, and laboratory control samples are routinely performed, in compliance with method or laboratory requirements.

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Quality Control Definitions

Accuracy - Accuracy is evaluated in two fashions, either as measurement accuracy or analytical accuracy. Measurement accuracy is measured by analysis of QC reference materials, and analytical accuracy is determined by spiking known concentrations into samples prior to preparation and analysis. Measurement accuracy is independent of matrix effects, while analytical accuracy includes any effects from sample matrices upon the recoveries of the analytes. Accuracy is affected by both systematic and random errors.

Measurement data is evaluated by comparing the percent recoveries of QC reference materials of known and evaluated concentration, independent of routine calibration. These reference materials can be used as prepared, or diluted into an inert matrix. Control limits are determined by control charting the recoveries and performing statistical analysis upon the results. Laboratory Control Samples (blank spikes) are also utilized to evaluate laboratory performance of methods. This is also independent of matrix effects.

Analytical accuracy is evaluated by comparing the percent recoveries of analytes, which have been added to samples at a known concentration. This is evaluated using matrix spikes and spike duplicate samples. Spike samples are analyzed at 5% frequency (1 in 20 samples), or per analytical batch, whichever is more frequent. Recoveries are assessed to determine the method efficiency and matrix interference effects upon analyte recovery.

The equation used to calculate percent recovery of an analyte is:

$$\% \text{ Recovery} = \frac{(\text{Spike Sample Result} - \text{Sample Result})}{\text{Amount of Sample Spiked}} \times 100$$

Quality Control Samples

The following QC samples are analyzed with client samples:

1) <u>Calibration Standards:</u> Solution(s) of known analyte concentration, used in the calibration (standardization) procedure to determine the relationship between concentration and analytical response. A minimum 3 point calibration is required, not including the calibration blank if used. A valid calibration must have a correlation coefficient of > or = 0.990, or a RSD of < 20% for the calibration curve.
2) <u>Blank:</u> A reagent blank (if applicable) is analyzed with every set of samples that is extracted or digested. This reagent blank includes any and all reagents that are used in the analytical process and is carried through the entire process, including extraction and filtering or digestion.
3) <u>Continuing Calibration Verification Standard (CCV):</u> At least one CCV is analyzed with each set of samples and every 10 additional samples. The CCV is made with certified

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reference materials. Samples run with a CCV that falls outside the control limit are reanalyzed, including digestion or extraction if necessary. The use of data outside of warning limits is at the discretion of the Scientific Director or reviewing analyst.

4) Duplicates: At least five percent of samples are analyzed in duplicate to yield data on the precision of the analysis.
5) Matrix Spike Samples: Sample fortification or spikes are used to verify accuracy of tests requiring extensive sample manipulation (such as acid digestion) or for non-standard type samples. When applicable, matrix spike samples shall be run per matrix and/or concentration level or per batch.

Data Review

During data review, data is compared to historical data, review of sample preparation and data from sample analysis is evaluated. Corrective actions are minimized by implementation of routine system controls. Analysts are provided with, and comply with, specific criteria for procedure, operation, and measurement systems.

Analysts are responsible for data review, check compliance with QC criteria, and checking instrument calibration, blank reviews, review of raw data (including peak identification), and calculations of raw data. Analysts review 100% of data, whether hand or instrumental calculations are performed. Upon completion of this step, secondary review is performed at a minimum of once per month. This step emphasizes data acceptability relative to QC indicators and acceptance criteria, data consistency and completeness.

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DOCUMENT MANAGEMENT

This Section describes procedures for document management, which includes controlling, distributing, reviewing, and accepting modifications. The purpose of document management is to preclude the use of invalid and/or obsolete documents.

Documentation
The Scientific Director controls revision, storage and availability of all Standard Operating Procedures (SOPs). All original documents are signed and dated by the Scientific Director and scanned into read only electronic (.pdf) files. These files are placed on the laboratory server and are made accessible to all staff members. The Scientific Director will maintain master copies of all SOPs in a secure location or file folder. It is the responsibility of each employee to verify that any SOP in their possession, either paper copy or electronic copy, is the most current revision available on the laboratory server. Suggested revisions to electronic documents are presented to the Scientific Director for review and approval. Where practicable, the altered text or new text in the draft is identified during the revision or review process to provide for easy identification of the modifications.

Standard Operating Procedures (SOPs) are used to ensure consistency of application of common procedures, are written procedures that describe in detail how to accurately reproduce laboratory processes. The SOPs are reviewed annually to ensure their contents are suitable and in compliance with the current quality systems requirements, compliance with State regulations and accurately describe current operations. SOPs do not have to be formal documents with predefined section headings and contents. They can be less formal descriptions of procedures described in the Quality Manual or other documents. Each SOP indicates the effective date, the revision number, and the signature(s) of the Scientific Director.

Test Method SOPs
The laboratory has SOPs for all test methods within its scope and for procedures that are part of the Quality System that accurately reflect how the analytical process is performed. Where equipment manuals or published methods accurately reflect laboratory procedures in detail, a separate SOP is not required.

Any deviation from a test method is documented, including both a description of the change made and a scientific justification. The deviation from a test method is reported to the client.

Each Test Method SOP includes or references (as applicable) the following:
1) Identification of the test method.

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2) Applicable matrix or matrices.
3) Detection limit.
4) Scope and application, including components to be analyzed.
5) Equipment and supplies.
6) Reagents and standards.
7) Quality control, including acceptance criteria.
8) Calibration and standardization.
9) Procedure.
10) Data analysis and calculations.
11) Any tables, diagrams, flowcharts and validation data.

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CONTROL OF RECORDS

Records Management
The purpose of records management is to ensure all records for the laboratory are accountable and traceable, legible, retrievable and protected from deterioration or damage including chain of custody (COC) records, logbooks, graphs, raw data, and other required items for verification of data and as required by State codes.

The laboratory maintains and controls records of laboratory data sheets and logs, instrument logbooks, standards logbooks, and records for data reduction, validation, storage, and reporting. Archived records are protected against fire, theft, loss, environmental deterioration, vermin, and in the case of electronic records, electronic or magnetic sources.

All electronic records are backed-up daily. Access to protected records is limited to licensees, laboratory management or their designees, AMCO personnel or law enforcement to prevent unauthorized access or amendment.

In the event that the laboratory transfers ownership or goes out of business, records are maintained or transferred according to the clients' instructions and State regulations.

Documentation of client samples begins when samples are received, assigned unique laboratory numbers, and reconciled with COC forms. Each shipment has a unique sample ID number that is carried throughout the sample analysis and reporting process. The information about sample receipt is contained in the Sample Check-In Log and COC which contains information on sample receipt, matrix, parameters, containers, and documentation.

Additional required information is contained in the instrument logs, standard logbooks and analyst's test data sheets.

All sample and analytical data is retained in accordance with 3 AAC 306.755.

Logbook Requirements
Requirements for logbooks used in the laboratory are as follows:

1) Bound logbooks with numbered pages are preferred over loose sheets. Sheets must be affixed to a logbook page, signed and dated both on the sheet and running onto the logbook.
2) Only assigned logbooks can be used for record keeping.

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3) Logbooks are uniquely named and numbered (example GC Logbook 001).
4) All writing must be legible and in black or blue ink. Corrections can only be made by drawing a single line through the entry and initialing the change.
5) Complete information must be entered.
6) Any data invalidated must be noted as to the cause.
7) All data relevant to the information must be included. This may include reagent lots, preparation information, instrument conditions, etc.
8) When work is continued to another log, the number of the first log is entered on the first page of the second log.

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CONTROL OF NON-CONFORMING WORK

Non-conformances
A nonconformance is a deficiency in characteristic, documentation, or procedure sufficient to make the quality indeterminate or unacceptable. An out of control event is a sub-category of non-conformance.

When either a nonconformance or out of control event occurs, it will be categorized as either a deficiency or an observation.

- Deficiencies are defined when a specific requirement of a program, process or procedure has been violated.
- Observations are defined when there is recognition of an activity or action that may be improved, but is not in violation of a specific requirement. Observations may degrade to a deficiency if not corrected.

Out of Control Events
Out of control events are defined as any occurrence failing to meet pre-established criteria.

Criteria for Out-Of-Control Events
Situations that require a *non-conformance report* include:

1) Failure or suspected failure to comply with test method, or other laboratory SOPs such that the integrity of the test results may be jeopardized.
2) Calibration results or maintenance activities that indicate an equipment malfunction has occurred and may have impacted test results.
3) Test results do not meet the regulatory or client defined acceptance criteria.
4) Results of quality control samples that are outside of established ranges.
5) Performance testing program results that are outside of expected ranges.
6) Client complaints.

Any of these situations may require investigation and corrective actions. Some factors can be assessed through use of control charts. Control charts can yield information on trends, biases, and shifts in the operation of the analytical system.

The detection of one of these conditions is an indication the analytical system may be out-of-control. The out-of-control value is placed on a control chart, circled, and documented through the use of a non-conformance report. The Scientific Director and/or Laboratory Manager are notified, and determine whether the incident is random, or reflects a process that is out-of-control.

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Control Charts
Measurement system performance can be demonstrated by the measurement of homogeneous and stable control systems. The data compiled is plotted, and a control chart generated to indicate whether the system is providing process control. It is also used to notify the laboratory of developing method performance issues, by identifying systematic errors, drifts, or other problems.

Use of control charts:
1) Provide graphical assessment of accuracy and precision for the measurement of each analyte.
2) Allow observations of recovery trends for individual analytes, and allow self-evaluation of analytical output.
3) Provide assessments of analytical capability of the scientific staff.

A system must be verified as being in control, in order to be maintained in control. A system is not in control if it is observed to produce unexpected data more than once every 20-25 runs. Control limits usually become tighter once a process is under a controlled protocol.

Control Chart Types
Control charts that are used to monitor the performance of the methods are:
1) Matrix Spike/Matrix Spike Duplicate (MS/MSD) Recoveries per matrix type.
2) Blank or Laboratory Control Sample (LCS) Percent Recoveries.

Preparation of Control Charts
For methods which quality control acceptance limits are not specifically determined control charts are used to evaluate lab performance. Control charts, when used, will be prepared for each analysis and matrix type separately. The charts will consist of a centerline, two warning limits, and two control limits. This process is described in HAZWRAP DOE/HWP-65/R1 and EPA Handbook on Analytical Quality Control in Water and Wastewater Laboratories. Control charts require a minimum of 20 points to begin charting, so method recommended recoveries shall be used until sufficient data points have been gathered.

Interpretation of Control Charts
Representative concentrations of compounds as described in the method or SOP are spiked into the required percentage of samples (method required frequency for Laboratory Control Samples (LCS) (or Blanks) and MS/MSD, all samples for surrogates when utilized). Recovery information is gathered for these points. The mean (x) and standard deviation (s) is calculated, and from this information warning and control limits are calculated.

These are:
1) Warning limits are defined as x +/- 2s.

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2) Control limits are defined as x +/- 3s.

The %R of each surrogate recovery, MS/MSD recovery, or QC sample is plotted on a control chart and compared with the control and warning limits.

Control charts can also be used to evaluate data precision (RPD) on duplicate or spike duplicate samples. The calculations for warning and control limits are the same as above.

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CORRECTIVE ACTION

Need for corrective actions come from several sources, equipment malfunction, failure of QA/QC checks, follow-up of performance or system audit findings, and QA non-compliance.

Any of the following incidents causes an out-of-control corrective action to be performed:
1) One or more points outside the control limit (3s).
2) Two or more consecutive points outside the warning limit (2s).
3) Seven or more consecutive points on same side of mean recovery bar (x), indicating data trends or shifts.
4) Cyclic or non-random patterns occurring in the data points plotted.
5) Runs of 6 or more points in the same direction.

When instrumental or analytical methods fail QA/QC, the failure will be brought immediately to the attention of the Scientific Director and/or the Laboratory Manager. Corrective measures taken will depend on the analysis, the type of error, and the extent of the failure. If an analysis fails batch QA/QC (MS, LCS, Blank or duplicate) criteria and the batch cannot be reanalyzed (due to insufficient sample) or matrix interference is suspect all failures will be flagged as out of control and reported to the end data user. Corrective actions can be extensive or simple. Each case requiring a corrective action will be determined on a specific basis.

Equipment malfunctions will be handled by segregating the equipment, until repairs can be performed, and precision and accuracy on the instrument restored.

Failure of performance audits will require Peak Analytical to identify the problem in analysis or document traceability. A step-by-step analysis of the root causes of the analytical problem is instituted, and corrective actions are taken.

All incidents of QA failure and corrective actions will be documented fully. Any actions will be taken quickly, and the process will include actions to prevent re-occurrences of the event.

Procedures for Corrective Action
When an out of control event occurs, the analyst will consult the appropriate Method SOP for proper corrective action. If corrective action is not possible (i.e. insufficient sample volume for reanalysis or suspect matrix interference) and the data is out of control or nonconforming, the analyst writes a case narrative (sub-section of the non-conformance report) describing the conditions of data analysis. The report form is filled out and the situation is brought to the attention of the Scientific Director or laboratory manager for

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evaluation. The Scientific Director or laboratory manager will determine the validity of the data and will contact the client to determine if data is acceptable to the client or additional action is necessary (i.e. re-sampling). All conversations with the client are documented. A case narrative is written to describe the nonconformity and is included with the final non-conformance report. All associated data must be flagged as Non-Conforming (NC) and all QC parameters that are out of control and associated with the data are flagged and included in the final non-conformance report.

The Scientific Director is responsible for review of non-conformance reports and will suggest improvements, alternative approaches, and procedures where needed, documenting the actions taken in response to the nonconformity.

Procedures for Halting Analysis

When the analytical system is out-of-control, corrective action processes are initiated by the analyst. If the problem is instrumental or specific to a particular batch analysis, any samples prepared after the event are re-processed after re-calibration, providing holding times are met.

If the batch or sample is still out-of-control after re-analysis, all method related activities are stopped. A detailed investigation to the causes is conducted, and any process problems are corrected. All actions taken are documented in the project file related to the sample(s).

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PREVENTIVE ACTION

Preventive action, rather than corrective action, aims at minimizing or eliminating inferior data quality or other non-conformance through scheduled maintenance and review, before the non-conformance occurs.

Preventive action includes, but is not limited to, review of QC data to identify quality trends, discussion of quality trends and issues at staff meetings and annual managerial reviews. The laboratory's management periodically conducts a review of the laboratory's quality system and testing activities for continuing suitability and effectiveness, and to introduce necessary changes or improvements.

Routine and periodic maintenance schedules for equipment and instruments are followed in the lab.

All employees are encouraged to make suggestions to improve processes throughout the lab and are empowered by management to implement those improvements upon approval by the Scientific Director or laboratory manager.

Trend analysis is used throughout the laboratory to identify systematic and systemic issues that may affect the quality of results or efficiency of processes. Examples of types of systemic issues and measurement tool and useful records that are employed in trend analysis include, but are not limited to, audit reports, control charts, maintenance records, COC discrepancies, hold time violations, calibration reports and method QC failures.

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DATA VALIDATION

Data validation is the process of reducing, reviewing, and accepting (or rejecting) data produced by the laboratory against a set of criteria. It is a systematic procedure of reviewing a body of data against the criteria to provide assurance of validity prior to use by a client or regulatory agency.

Scientific staff will document sample preparation activities in prepared laboratory notebooks or bench sheets. Sample tracking and numbering systems, and the use of quality control criteria, will be used to reject or accept data. These are the primary record of preparation activities in the laboratory, and for data reduction occurring after analysis. Data for GC and LC methods are generated by stand-alone computer systems. Results from each analysis are transferred only to analytical forms specific to the particular analysis. Data is checked for accuracy and precision at the bench and instrument analyst level.

Validation is accomplished through routine audits of data collection and flow procedures, and by monitoring QC sample results. The process consists of data screening, checking, auditing, verification, certification, and review. Upon request, Peak Analytical will certify in writing that the data has been validated in accordance with defined laboratory processes.

Review of QC Data
When an analytical data set is complete, the results will be reviewed to establish the validity of the data.

Minimum Validation Requirements:
1) A minimum 3 point calibration, not including the calibration blank if used.
2) Blank Evaluations - Review of method, instrument, and rinse blanks for background and solvent/reagent contamination. Results of Blank samples are evaluated for % recovery values, and compared with acceptance criteria.
3) Continuing Calibration Verification Standard Evaluation - Review to determine linearity, range, and verify sample values within control limits.
4) Duplicate Sample Evaluation - Review of precision using RPD criteria for duplicates within set, if used in method.
5) Matrix Spike Sample Evaluation - Review of precision and accuracy for the spike results for tests requiring extensive sample manipulation (such as acid digestion) or for non-standard type samples.

Data checks used to validate precision and accuracy of parameters measured, and to support the representativeness, comparability, and completeness include:
1) Correlation coefficient of > or = 0.990, or a RSD of < 20% for the calibration curve.

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2) Documentation of traceability of the instruments standards.
3) Documentation of methodology (analytical and QC) from the SOP.
4) Routine maintenance performed.
5) Documentation of sample transportation and preservation.

Documentation supporting data validation includes:
1) Documentation of calibration.
2) Documentation of instrument checks (noise levels, drift, and linearity).
3) Documentation of traceability of instrument standards, samples and data.
4) Documentation of analytical methodology and QC requirements.
5) Documentation of control taken to minimize and verify interference in the method (reagent blank analysis for accuracy and precision).
6) Documentation of routine maintenance.
7) Documentation of sample preservation and transport.

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REPORTING OF RESULTS

Reporting of Results
The result of each test carried out is reported accurately, clearly, unambiguously, and objectively and complies with all specific instructions contained in the test method. Data are reported without qualification if they are greater than the lowest calibration standard, lower than the highest calibration standard, and without compromised sample or method integrity.

Test Reports
The report format has been designed to accommodate each type of test performed and to minimize the potential for misunderstanding or misuse.

Each test report generated contains the following information, as appropriate:
1) Name and contact info of client.
2) Date sample received.
3) Sample harvest name.
4) Sample strain.
5) Sample matrix.
6) Sample ID.
7) METRC ID.
8) Transport ID.
9) Identification of test method used.
10) Analysis date.
11) Quality control results (pass/fail).
12) Test results.
13) Comments.
14) At the laboratory's discretion, a statement that the report shall not be reproduced without full written approval of the laboratory.

Supplemental Test Report Information
When necessary for interpretation of the results or when requested by the client, test reports may include the following additional information:
1) A Case Narrative with deviations from, additions to, or exclusions from the test method, information on specific test conditions, such as environmental conditions, and any non-standard conditions that may have affected the quality of the results, and any information on the use and definitions of data qualifiers.
2) A statement of compliance/non-compliance when requirements of the quality systems are not met, including identification of test results that did not meet standard sample acceptance requirements, such as holding time, preservation, etc.

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3) Where applicable and when requested by the client, a statement on the estimated uncertainty of the measurement.
4) Where appropriate and needed, opinions and interpretations.
5) When opinions and interpretations are included, the basis upon which the opinions and interpretations are documented. Opinions and interpretations are clearly marked as such in the test report.
6) Additional information which may be required by specific methods or client.
7) Qualification of results with values outside the working range.

Amendments to Test Reports

Material amendments to a test report after it has been issued are made only in the form of another document or data transfer. All supplemental reports meet all the requirements for the initial report and the requirements of this Quality Manual. Amended test reports are titled to assure they can be differentiated from other test reports. When it is necessary to issue a complete new report, the new report is uniquely identified and contains a reference to the original that it replaces.

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AUDITS AND MANAGEMENT REVIEW

System audits, performance audits and QA/QC audits of Peak Analytical will be performed on a periodic, on-going basis. These audits will be included in all laboratory audit files, and analyst specific information will be contained in the analyst certification/training files.

System Audits
System audits will be performed internally and may include reviews performed by an outside entity. System audits may also be performed entirely by an outside entity. Internal system audits would be performed similarly to outside audits, but be performed by the Scientific Director or laboratory management. The laboratory will be audited at a minimum of biennially, and include sample management and document control as well as analytical procedures.

Performance Audits
Performance audits would include any performance evaluation samples analyzed by Peak Analytical. These will be documented, and copies of relevant information will be included in analyst certification/training files. These will include, as a minimum, all performance evaluation samples analyzed by the laboratory, including single-blind samples initiated by the Scientific Director or laboratory manager. They would also include all double-blind performance evaluation samples analyzed as part of maintenance of accreditation or certification by regulatory agencies, if applicable. Information on corrective actions should be included. All analysis performed by the laboratory, for which a performance evaluation sample can be obtained, will be analyzed on an annual basis. This information will be recorded in the annual audit report and relevant analyst training files.

Performance audit reports will summarize information on laboratory performance. They will include periodic audits performed by Laboratory personnel, laboratory performance on performance evaluation samples, both internal and external, information on activities to improve precision and accuracy of the analysis and summary information on outside entity audits. They should include responses to performance evaluation results exceeding acceptance limits, and responses to comments contained in internal and external audits. Performance audits will be performed at a minimum of annually.

QA/QC Audits
Daily audits of data collection and flow will include:
1) Review of sample documents for completeness.
2) Review of instrument logs, QC sample results, and analyst bench sheets.
3) Review of performance indicators.

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4) Random calculation checks at a minimum of 10%.
5) Review of all reports subsequent to data entry.

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ACRONYMS

A list of acronyms used in this document and their definitions are:
CCV – Continuing calibration verification
COC – Chain of custody
NC – Non-Conforming
CRADA – Cooperative Research and Development Agreement (EPA CRADA)
DQO – Data Quality Objectives
EPA – Environmental Protection Agency
GC – gas chromatography
GLP – Good laboratory practice
LCS – Laboratory control sample
LOD – Limit of detection
LOQ – Limit of quantitation
MDL – method detection limit
MS – matrix spike
MSD – matrix spike duplicate
NIST – National Institute of Standards and Technology
PE – Performance evaluation
PQL – Practical Quantitation Limit
QA – Quality assurance
QAP – Quality Assurance Plan
QC – Quality Control
QAM – Quality Assurance Manual
RL – Reporting level
RPD – Relative percent difference
RSD – Relative standard deviation
SOP – Standard operating procedure
SRM – Standard reference material

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GLOSSARY

Accuracy
The degree of agreement between an analytical result and an accepted reference value or true value. The accuracy of a result is affected by both systematic errors (i.e., bias) and random errors (i.e., imprecision). Some analysts improperly use accuracy to denote only systematic error. (See "bias" and "precision.")

Action limit
A control limit on a control chart, which, if exceeded, requires corrective action to be taken. Action limits are usually placed at +3 standard deviations from the expected or mean value. (See "Control Limit" and "Warning Limit" below.)

Analyte
That which is analyzed for in chemical, but not physical or biological, determinations.

Analytical error
The error, E, of an analytical result, R, is defined as:
$E = R - T$
where T is the true value.

Analytical Method
Written instructions describing an analytical procedure followed to obtain a numerical estimate of the concentration of a determinant (analyte) in a sample or samples.

Analytical Response
A numerical observation obtained when a sample is presented to a measurement sub-system (e.g., spectrophotometric measurement of the absorbance of a solution). The magnitude is related to the concentration of the determinant (analyte) in the sample.

Analytical Result
A numerical estimate of the concentration of a determinant (analyte) in a sample, obtained by carrying out once the procedure specified in an analytical method. A method may specify analysis of more than one portion of a sample to produce one analytical result. The result can also be thought of as the final value reported to the user.

Analytical System
A combination of analyst, analytical method, equipment, reagents, standards, laboratory facilities, any other components involved in carrying out an analytical procedure.

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Batch
A group of environmental samples prepared and/or analyzed together using the same process, personnel and reagents.

Prep Batch
A batch of samples from 1-20 of the same matrix that are processed together. The maximum time between the start of processing the first and last sample in the batch is 24 hours.

Analytical Batch
A batch of prepared samples (extracts, digestates or concentrates) which are analyzed together as a group. The analytical batch may contain samples derived from more than one matrix and more than one prep batch.

Bias
The part of inaccuracy of an analytical result caused by systematic error.

Blank
A sample used to estimate the analytical response attributable to all factors other than the determinant (analyte) in the sample. Blanks are analyzed identically to samples, but do not contain the determinant (analyte) (e.g., in water analyses, pure water would be analyzed to determine the blank). Some analysts use the term method blank with the same meaning given here.

Calibration Standards
Solution(s) of known analyte concentration, used in the calibration (standardization) procedure to determine the relationship between concentration and analytical response.

Certified Reference Material
A substance, one or more property values of which are certified by a Scientifically valid procedure accompanied by or traceable to a material certificate or other document issued by a certifying body.

Check Standard
A solution of known concentration used to check the precision analyses (and bias due to calibration). When used in conjunction with a control chart, the check standard is called a control standard.

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Control Limit
A value on a control chart used to make decisions as to whether the results or control tests are acceptable. Two kinds of control limits are usually used: warning limits and action limits.

Criterion of Detection
The smallest concentration, which can be distinguished from a blank with no more than a 5% chance of reporting a false positive.

Data Quality Objectives
Qualitative and quantitative statements of the quality of data. Qualitative statements may be made concerning completeness, defensibility, representativeness, and accuracy, as well as other factors. Quantitative statements are usually made concerning acceptable bias and precision (which together, influence accuracy).

Degrees of Freedom
A whole number expressing the amount of information available for an estimate. The whole number is generally the number of independent results less the number of constraints. Constraints are what else needs to be estimated from the same set of results.

Determinant
That which is to be determined. Covers chemical, physical, biological or other analytical determinations. (See "Analyte" above.)

External Standard Calibration
The use of independently prepared standards to determine the relationship between response and concentration, run separately from the sample(s). Also called external standardization.

Interference
Systematic error (bias) in the analytical result caused by the presence of a substance in the environmental sample (or added to the sample during analysis).

Internal Standardization
A calibration and analysis procedure in which the responses of analytes are determined relative to an internal standard (standards added to every sample). Two solutions-calibration and spiking-are required.

Isotope Dilution
An internal standardization procedure in which the internal standards are isotopically labeled analogs of target analytes. Isotope dilution techniques are characterized by very low bias due to calibration.

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Laboratory Control Standard
See Check Standard.

Limit of Detection
The smallest concentration of an analyte for which there is at least a 95% chance the analyte, if present, will be detected (i.e., there is only a 5% chance of obtaining a false negative).

Matrix Spike/Matrix Spike Duplicate
See Spike.

Percent Recovery
That percent of a known amount of material 'spiked' or added to a sample being analyzed which is reported at the end of the analysis. See "spike," "analytical recovery," and "physical recovery."
% Recovery = 100(R2 - Rl)/A
where R, is the result for the sample without the spike, and R2, the result for the spiked sample, and A is the equivalent concentration added in the spiked sample.

Population
The collection of all possible analytical results.

Precision
A qualitative term used to denote the scatter of results. Precision is said to improve as the scatter among results becomes smaller. Random error is also referred to as imprecision. Usually measured as standard deviation.

Quality Assurance
The total integrated program for assuring the reliability of monitoring and measurement data.

Analytical Quality Control
The routine application of statistically based procedures to evaluate and control the accuracy of results from analytical measurements.

Random error
Errors indicated when repeated analyses of identical portions of a homogeneous sample do not give identical results. Random results differ among themselves and are more or less scattered about some value. They are termed random because the sign and magnitude of the error of any particular result vary at random, and cannot be predicted with confidence.

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Analytical Recovery
An estimate, usually expressed in percent, of an analytical result in comparison with a true or reference value for the analyte, (e.g., an analytical recovery of 95% for compound X means that the result was 95% of the true or reference value for X in the sample).

Physical Recovery
An estimate, usually expressed in percent of the amount of standard or analyte present at the final stage of analysis (e.g., final extract), compared with the amount present in the original sample. Physical recovery can be an indirect indication of analytical performance.

Reference Material
A material or substance, one or more properties of which are sufficiently well established to be used for the calibration of an apparatus, the assessment of a measurement method, or for assigning values to materials.

Relative Percent Difference
The difference between duplicate results for analyses of a sample, relative to the mean value of those results and expressed as a percent.
RPD = 100(di - d2)/E(dl + d2)/2
= 200(d1 - d2)/(dl + d2)
where d1, is the result of the first analysis, and d2 the second.

Relative Standard Deviation
The standard deviation relative to the mean. Also called coefficient of variation. RSD is calculated as either s/x-bar or lOOs/x-bar. The latter is sometimes referred to a percent relative standard deviation, or %RSD. RSD indicates the magnitude of imprecision.

Spike
A known amount of analyte added to a sample for the purpose of judging, from the analytical percent recovery, whether there is bias due to interference present in the sample. Also referred to as fortification of the sample. See "percent recovery."

Standard
A solution of known concentration. There are two types of standards: (1) check (or control), and; (2) calibration.

Standard Operating Procedure
A detailed, written description of a procedure designed to systematize the performance of the procedure.

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Standard Deviation
A statistical constant which describes the width of the normal distribution or spread of results.

$$S = \sqrt{\frac{\Sigma(X - \overline{X})^2}{N}}$$

where S = the standard deviation of a sample,
Σ means "sum of,"
X = each value in the data set,
$\overline{X}$ = mean of all values in the data set,
N = number of values in the data set.

Standard Reference Material
A sample of known concentration, also called a certified reference material, issued by the National Institute for Standards and Technology (NIST).

Statistical Sample
The results of one or more determinations from the sample population of all possible results.

Statistics
Certain single values computed from the results, which characterize the distribution of the results. Each statistic has its own frequency distribution which is defined by a particular mathematical function.

Surrogate Standard
A type of check standard added to each sample for certain types of analyses (e.g., trace organic), in a known amount and at the start of processing. The surrogate is not one of the target compounds for the analysis, and is not expected to be present in environmental samples, but should have analytical properties similar to those compounds.

Systematic Errors
Errors indicated by a tendency of results to be greater or smaller than the true value. Usually bias can be considered to be equivalent to systematic error.

Target Compound
A compound which is expected to be in an environmental sample or for which the analysis is being conducted. Also referred to as 'analyte.'

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Warning Limit
A control limit on a control chart, usually +2s distant from the expected or mean value. Action is required when results fall outside the warning limits too frequently. A single value outside a warning limit does not necessarily require action, but should alert one to a possible problem.

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