

October 25, 1999

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Tien-Yu (Tammy) Chang,

Below are itemized responses to the findings identified during your on-site evaluation of DataChem Laboratories, Inc. (DCL) on October 12, 1999. The responses are listed in the exact order as received in your audit report sent October 20, 1999.

Chromatography (HPLC) Action Items

1. DataChem will develop an SOP for manual integrations that will include the provisions to provide "before" and "after" chromatograms with every data package, a written reason for the manual integration, and signatures of analysts and peer reviewer. This SOP will be completed by November 26, 1999. Additionally, all manual integrations performed on AFCEE projects will contain a "before" and "after" chromatogram with the reason for such, and analyst and peer reviewer signatures included in the data package.
2. DataChem will purchase min/max thermometers for critical refrigeration units and record the temperature extremes detected over the weekend or holidays.

One person (who was not interviewed by the audit team) is responsible to check all refrigerators in the building. When a temperature deviation is encountered the person takes a second reading later, if the unit continues to be out of compliance an attempt is made to bring the unit back into compliance by the next day. If the unit cannot be brought back into compliance then a refrigeration specialist is called to provide same day service to fix the unit.

Exhibit 1 illustrates this process from April 1999. A refrigerator was checked and found to be out of compliance on April 2. Because the refrigerator was empty a second check was not taken that day. The refrigerator was adjusted and checked again on Monday the 5th and found to be back in compliance. However on the 14th the refrigerator was again out of compliance. The refrigerator was discarded and a new one placed in service on the 20th.

3. DCL has ordered several new pipettors for use in the extraction area in order to have a sufficient number to dedicate for each solvent type.
4. DCL will write down the lot number of sodium chloride in the extraction notebook in order to insure traceability for all compounds added to explosive samples from your project. **Exhibit 2** is an example of the extractionist notebook page itemizing the NaCl lot number (circled).
5. Soil samples are dried in an operating fume hood at ambient conditions until, in the judgement of the extractionist, they are dry. This usually takes several hours to overnight, but occasionally takes longer. Because the method requires $2\text{g} \pm 0.1\text{g}$ of sample be weighed for preparation, it is our experience that the difference in weight due to any drying time added onto the extractionists' normal drying time, such as to "verify" constant weight with a second weighing, is much less than the 0.1g error tolerance for weighing out samples for extraction.
6. Acceptance criteria for the top loading balance is written in the balance logbook located with the balance. Analysts has been reminded to inquire and understand the acceptance limits for daily balance check weights.
7. This certificate of analysis was obtained from Aldrich on October 13th.
8. DataChem will perform a 72-hour retention time window study by November 12, 1999. The resulting retention time windows will be used to process data for AFCEE projects.
9. O-cresol is spiked in the sample extract to monitor injection and instrument response consistency for diagnostic and troubleshooting purposes only. It is not required by the method and there are no criteria established for acceptance (see **Exhibit 3**, SOP OL-SW-8330, Determination of Explosives by EPA Method 8330, section 2.6).
10. See 9 above.
11. Solution ID numbers are recorded in the analytical notebook which is included with every data set. (See **Exhibit 4**).
12. Analyst was notified of the error. A one day expiration will be applied to CCV solutions such that they are prepared fresh on the day of use.
13. The SOP is incorrect and will be modified by November 5th to reflect actual practice in the laboratory.
14. Calculation is available in method 8000 for reference. Note, however, that the analyst was under pressure of "many eyes looking on" and, once given 30 seconds alone, was easily able to manually calculate the CCS check, which was shown to the audit team while they were here.
15. Manual integration training is performed during initial analyst training. Notwithstanding, as per item 1. above, DataChem will have an SOP for training and periodic analyst review by November 26, 1999.

16. The second column used for confirmation will be calibrated before the AFCEE project. Continuing Calibration Verification will be performed routinely thereafter to validate the initial calibration.
17. The analyst has *named* the calibration standards \$2, \$10, etc. to reflect the amount of intermediate solution necessary to spike into 1 mL of solvent for standardization. Section 8.1 of the SOP is correct. In addition Table 3 in the appendix to the SOP (see **Exhibit 3**) clearly identifies the name of each standard and its corresponding concentration in various units.
18. Section 9.6.1 of the SOP refers to the secondary confirmation column. Analysts always run a low level standard on the secondary column before each set to be confirmed (See **Exhibit 3**). There is a reference to running a low level standard to validate the RL in section 4.3.2 of the AFCEE QAP v3.0, but no reference that this must be done for every set. DatChem policy is to report the low standard concentration as the RL (or PQL). Results reported between the RL and MDL are flagged per the AFCEE QAP.
19. No sample or set reference was provided, so no response is given.

Sample Receipt, Storage, Preservation, Custody & Disposal Action Items

1. Analyst was reminded that the acceptance criteria for thermometers is $\pm 1^{\circ}\text{C}$.
2. Sample Custodian will record his daily infrared thermometer check when receiving samples for this project.

LIMS Action Items

None.

Facility Security Action Items

None.

Instrument Maintenance and Equipment Monitoring/Calibration Action Items

1. Microliter syringes are simply replaced when they malfunction. No attempt to repair them is made.
2. Out of control events may be attributable to solution problems rather than maintenance problems.

QA/QC Functions Action Items

1. DataChem Quality Assurance Manager Mitchell Peterson will conduct an internal audit of the laboratory by January 1, 2000. It is noted that, because of our participation in several federally sponsored programs, the laboratory averages 12 external audits per year by a wide cross-section of government and commercial firms. Problem prevention/solving of programs and processes within the laboratory is conducted on a continuous basis rather than at specified intervals. Notwithstanding, a yearly audit of the entire facility will be prepared by January 1 of every year going forward as required by our QAP.
2. Data review is documented in SOP XX-DC-023, Peer Review. A copy is included for your review as **Exhibit 5**.
3. The only management level above the Laboratory Director is the President/Owner who does not take an active role in managing the laboratory. The owner maintains an office separate from the laboratory and visits only periodically.

Safety Program Action Items

1. Analysts have been reminded to wear proper protective clothing while working with samples and chemicals.
2. Hallways are used for temporary storage in the loading dock area. When a shipment is received it may take one or two days for the responsible person to distribute/store material shipments.

END of Corrective Actions

Please feel free to call me anytime regarding these responses at (801) 266-7700. Thank you for your time and the efforts you have taken to approve DataChem for this important project.

Sincerely,



B. Mitchell Peterson
Quality Assurance Manager
DataChem Laboratories, Inc.

Enclosures: Exhibits 1-5

CC: Bob DiRienzo, DCL
Kevin Griffiths, DCL
Richard Wade, DCL

Exhibit 1

DCL RECORD OF TEMPERATURE

Unit Number : R-63-2

Correction Factor : 0

Acceptable Temperature Range: 2-6°C

Date of Thermometer Calibration: 5/9/98

Record for Month of: April 1999

Day	T(°C)	Initials /Time	T(°C)	Initials /Time	NCR	Day	T(°C)	Initials /Time	T(°C)	Initials /Time	NCR
-----	-------	----------------	-------	----------------	-----	-----	-------	----------------	-------	----------------	-----

1	3.5	MPS 1402				17					
2	3.0	MPS 1400				18					
3						19					
4						20	3°	TA 1100			
5	5.5	MPS 1500				21	3°	TA 11:00			
6	4.5	MPS 1400				22	4.0	TA 1700			
7	4.5	MPS 1600				23	4.5	MPS 1627			
8	3.0	MPS 1400				24					
9	3.5	MPS 1300				25					
10						26	4.5	MPS 1503			
11						27	4.0	MPS 1649			
12						28					
13	3.0	MPS 1600				29	4.5	MPS 1759			
14	3.0	MPS 1535	15.0	MPS 1725		30	4.0	MPS 1453			
15	19.0	MPS 0800				31					
16	18.0	TA 1000									

MAINTENANCE SCHEDULE

DAY	COMMENTS/ACTION
4/2/99	Temperature gauge adjusted? Fridge was empty during this time
4/14/99	Temp below range
4/20/99	New fridge put here, old one discarded.

Exhibit 3

DATA CHEM LABORATORIES, INC.

STANDARD OPERATING PROCEDURE APPROVAL SHEET

SOP TITLE: Determination of Explosives by EPA Method 8330

DOCUMENT CONTROL NUMBER: OL-SW-8330 Revision 5

EFFECTIVE DATE: July 28, 1998

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APPROVALS:

MANAGER

Richard H. Wade

Date

7/28/98

Q A MANAGER

B. M. Plus

Date

7/28/98

LAB DIRECTOR

[Signature]

Date

7-28-98

STANDARD OPERATING PROCEDURE

DETERMINATION OF EXPLOSIVES BY EPA METHOD 8330

1.0 SCOPE AND APPLICATION

- 1.1 This SOP is applicable to the determination of nitroaromatics and nitramines in soil and water using HPLC analysis with UV detection. This SOP provides procedures and conditions used in performance of EPA Method 8330.
- 1.2 The compounds which are determined by following this SOP are listed below:

<u>Abbreviation or Analyte Code</u>	<u>Compound</u>
HMX	Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine
RDX	Hexahydro-1,3,5-trinitro-1,3,5-triazine
NB	Nitrobenzene
TETRYL	Methyl-2,4,6-trinitrophenyl nitramine
135TNB	1,3,5-Trinitrobenzene
13DNB	1,3-Dinitrobenzene
246TNT	2,4,6-Trinitrotoluene
24DNT	2,4-Dinitrotoluene
26DNT	2,6-Dinitrotoluene
2NT	2-Nitrotoluene
3NT	3-Nitrotoluene
4NT	4-Nitrotoluene
2-Am-DNT	2-Amino-4,6-Dinitrotoluene
4-Am-DNT	4-Amino-2,6-Dinitrotoluene

- 1.3 This SOP is restricted to use by or under the supervision of analysts experienced in the use of HPLC, skilled in the interpretation of chromatograms, and experienced in handling explosive materials. Furthermore, the analyst and extractionist performing this method will demonstrate the ability to generate acceptable data by providing accuracy and precision data which meet the requirements of the method.
- 1.4 The presence of additional compounds with retention times equal to the analytes of interest which absorb at 254 nm will interfere with the analysis and may result in inaccurate qualitative and quantitative results. The interfering compounds may arise from solvents, reagents, glassware, or instrumentation. For this reason, method blanks will be analyzed with each analytical batch to demonstrate the absence of interferences.

2.0 SUMMARY OF METHOD

- 2.1 Method 8330 provides high performance liquid chromatographic (HPLC) analysis and detection of ppb levels of certain explosive residues. A salting-out extraction procedure is used for low level water samples. Direct injection of diluted and/or filtered sample is used for water samples of higher concentration.

- 2.2 For the analysis of low level water samples, a 770-mL portion of the sample is saturated with sodium chloride and then extracted with acetonitrile. Equal volumes of extract are then thoroughly mixed with a 1% calcium chloride solution containing internal standard. The resulting mixture is analyzed using an isocratic HPLC system equipped with UV detection and a column heater.
- 2.3 For the analysis of soil samples, a 2-g portion of the sample is mixed with acetonitrile and then sonicated for 18 hours. Equal portions of the sample supernatant and 1% calcium chloride containing internal standard are mixed thoroughly. The resulting mixture is analyzed using an isocratic HPLC system equipped with a column heater and UV detection.
- 2.4 Positive results in field samples are qualitatively confirmed on a secondary column. The second column analysis is not quantitative and is used only to confirm the presence of each compound. Only the target compounds which are confirmed in the second column analysis are reported, using quantitative results from the primary column analysis.
- 2.5 The surrogate, 3,4-Dinitrotoluene (3,4-DNT), is added before extraction to monitor extraction efficiency.
- 2.6 The internal standard, ortho-cresol (OC), is added with the calcium chloride solution before analysis. The internal standard is not used for quantitation but is used to determine the consistency of injections and to evaluate instrument performance.

3.0 SAMPLE COLLECTION, PRESERVATION AND STORAGE

- 3.1 Field samples collected for analysis by this method require no preservation other than refrigeration at $4^{\circ}\text{C}\pm 2^{\circ}\text{C}$.
- 3.2 Soil samples collected in the field are transferred into 5-ounce amber, wide-mouth bottles with Teflon-lined caps for transport to the laboratory. Water samples are transferred into two 1-liter amber bottles for transport to the laboratory.
- 3.3 Sample integrity is maintained at the laboratory until analysis can be performed by storing the field samples at $4^{\circ}\text{C}\pm 2^{\circ}\text{C}$.
- 3.4 Holding Times
 - 3.4.1 Field samples for method 8330 have an extraction holding time of seven days for water and 14 days for soil samples from the date of sampling. The extracts from the field samples have an analysis holding time of 40 days from the date of extraction.

4.0 SAFETY PRECAUTIONS

- 4.1 Good laboratory technique dictates the careful handling of all laboratory samples and reagents. Eye protection and a laboratory coat are minimum requirements. The following specific additional safety measures are recommended:
 - 4.1.1 For sample receipt and log-in, a hood should be used until the sample containers are found to be intact. Gloves should be worn while handling the sample containers.

- 4.1.2 For sample transfer and splitting, *etc.*, a hood and gloves should be used.
- 4.1.3 For sample sonication, a hood and gloves should be used.
- 4.1.4 Special hazard warning: The analytes are explosive and therefore should be handled with care at all times, especially when grinding a dry sample. Do not dry the samples in an oven; air dry only.

5.0 INSTRUMENTATION AND EQUIPMENT

5.1 All glassware must be acid washed to prevent decomposition of the explosives.

5.2 Glassware/Hardware – Soil

- 5.2.1 Syringes: 10-, 25-, 250-, and 1,000- μ L - Gas tight
- 5.2.2 Volumetric flasks: 10-, 25-, and 100-mL - Class A
- 5.2.3 10 mL syringe
- 5.2.4 Sample vials
- 5.2.5 Sonicator bath
- 5.2.6 200 mm vials
- 5.2.7 Analytical Balance - Metler
- 5.2.8 Nylon Acrodisk filters - Gelman or equivalent

5.3 Glassware/Hardware – Water

- 5.3.1 Syringes: Various volumes as required - Gas tight
- 5.3.2 Volumetric flasks: Various volumes as required - Class A
- 5.3.3 Disposable Pasteur pipets
- 5.3.4 Graduated cylinders: Various volumes as required - Class A
- 5.3.5 0.45- μ m nylon 66 filters, 47-mm - Gelman or equivalent
- 5.3.6 1 Liter Erlenmeyer flasks
- 5.3.7 15-mL blow-down tubes
- 5.3.8 Blow-down apparatus
- 5.3.9 N₂ blow-down needles, size 13

5.3.10 3" and 1" stir bars

5.3.11 Stir plate

5.3.12 Teflon caps

5.3.13 Pipets: 10-mL and 1-mL - Class A

5.3.14 Timer

5.4 HPLC system consisting of an Hewlett Packard 1050 pump, solvent degasser, autosampler, UV detector, and column heater, or Hewlett Packard 1090 HPLC system or equivalent.

6.0 CHEMICALS, REAGENTS, AND STANDARD SOLUTIONS

6.1 The reagents used in this method are:

6.1.1 Methanol, HPLC grade or equivalent - Burdick & Jackson.

6.1.2 Acetonitrile, UV grade or equivalent - Burdick & Jackson.

6.1.3 Ortho-Cresol Solution: 10 mg of ortho-cresol diluted in 5.0 mL of UV grade acetonitrile (2000 µg/ml).

6.1.4 1% Calcium Chloride/Internal Standard Solution: 1 gram of calcium chloride (reagent grade) and 1.0 mL ortho-cresol solution diluted in 100 mL of ASTM Type II water.

6.1.5 ASTM Type II water.

7.0 SAMPLE PREPARATION

7.1 Soil

7.1.1 Air dry the samples at room temperature or colder. Do not expose the samples to heat or direct sunlight.

7.1.2 Grind and homogenize the air-dried samples in an acetonitrile-rinsed mortar and pestle to pass through a 30-mesh sieve.

7.1.3 Place 2 grams of homogenized sample into a 200 mm test tube, record the weight, add 1000 µL of 40 µg/mL solution of 3,4-DNT in acetonitrile. This provides a surrogate concentration of 20 µg/g for each sample.

7.1.4 Add 1.0 mL of matrix spiking solution to the LCS, MS and MSD samples. The concentration of the matrix spiking solution is specified in Table 5.

7.1.5 Add the appropriate volume of acetonitrile to each sample to bring the final volume up to 10.0 mL. Cap each tube with a Teflon-lined screw cap.

- 7.1.6 Vortex each sample for one minute, then sonicate in a cooled ultrasonic water bath for 18 hours. The temperature of the water bath should be kept at room temperature or below to avoid breakdown of tetryl and possibly other explosives. Check often.

Note: To ensure efficient sonication, fill the bath to a level greater than or equal to the level of the sample and visually check the samples for cloudiness periodically. If samples are not cloudy, investigate the performance of the bath. Poor recoveries are obtained when the ultrasonic water bath is not operating properly.

- 7.1.7 Allow the samples to settle for 30 minutes. Filter 5 mL of the extract through a 0.45- μm Teflon filter, discarding the first 2 mL and collecting the remainder for analysis.
- 7.1.8 Store the extracts in amber glass, acid washed vials with Teflon-lined screw caps. Store at $4^{\circ}\text{C} \pm 2^{\circ}\text{C}$ and protect from light.

7.2 Water

7.2.1 High-Level Method

7.2.1.1 Direct injection following the high-level method is recommended for aqueous samples with concentrations greater than 200 $\mu\text{g/L}$. It is also recommended that process waste samples or other potentially high level samples be screened with the high level method to determine if the low-level method is required. Concentrations less than about 50 $\mu\text{g/L}$ will require the low level method.

7.2.1.2 **Sample Filtration:** Place a 5-mL aliquot of each water sample in a scintillation vial, add 5 mL of acetonitrile and mix. Filter through an acetonitrile rinsed 10-mL syringe equipped with a 0.45 μm PTFE filter. Discard the first 3 mL of filtrate and retain the remainder in a Teflon-capped vial for HPLC analysis. HMX quantitation can be improved with the use of methanol rather than acetonitrile for dilution before filtration.

7.2.2 Low-Level Method (salting out extraction)

7.2.2.1 **NOTE:** Rinse all glassware with acetonitrile.

7.2.2.2 Add a large stir bar then add 325 g of sodium chloride to a 1 L Erlenmeyer flask. Measure out 770 mL of a water sample (using a 1 L graduated cylinder) and transfer it to the flask containing the salt. Spike samples with 1000 μL of a 10.0- $\mu\text{g/mL}$ solution of 3,4-DNT in acetonitrile as surrogate. Add 1000 μL of matrix spiking solution for water 8330/MS-W (see Table 7) to the LCS, MS and MSD samples. Mix contents at maximum obtainable speed on a magnetic stirrer for 15 minutes. **DO NOT STOP THE STIRRING OF THE SALT.**

7.2.2.3 Add 164 mL of acetonitrile (measured with a 250-mL graduated cylinder)

while the solution is being stirred, tightly cap with a Teflon cap. Stir for an additional 30 minutes, this time is critical and must be followed exactly. Turn off the stirrer and allow the phases to separate for 15 minutes.

- 7.2.2.4 Add a small stir bar to a 110-mL volumetric flask and add 84 mL of saturated salt water (425 g NaCl must be added per liter of reagent water to be sure the salt water is saturated).
- 7.2.2.5 Remove the acetonitrile (upper) layer (about 15 mL) with a Pasteur pipet, and transfer it to a 110-mL volumetric flask containing 84 mL of saturated salt water. If the solution is greater than the 6 to 8 mL mark, then another 110-mL flask must be used with 84 mL of saturated salt solution to accommodate all of the acetonitrile from the 1-liter flask. Cap the 110-mL flask with a Teflon cap.
- 7.2.2.6 Pipet 10 mL of fresh acetonitrile to the water sample in the 1-liter flask. Cap and again stir the contents at maximum obtainable speed for exactly 30 minutes followed by 15 minutes for phase to separate.
- 7.2.2.7 Combine the second acetonitrile portion with the initial extract in the first 110-mL flask if it is below the 6 mL mark; or, in the second 110-mL flask if the first one has reached the 6 to 8 mL mark. If the solution is below the 6 mL mark on the second 110-mL volumetric flask, transfer solution from the 1-L flask until such volume is obtained. As much as possible, pipet upper layer of the 1-L solution. Cap and stir the contents on a magnetic stirrer at maximum obtainable speed for 30 minutes followed by 15 minutes for phase separation.
- 7.2.2.8 CAREFULLY transfer the acetonitrile phase to a 40-mL blow-down test tube using a Pasteur pipet. Also, transfer the acetonitrile phase from the second 110-mL volume flask if one was used. There must NOT be any salt water solution transferred with the acetonitrile. The salt water contains a high concentration of an interference which produces a large peak at the beginning of the chromatogram and interferes with the HMX determination.
- 7.2.2.9 Pipet an additional 1.0 mL of acetonitrile to the 110-mL volumetric flask. If the solution is below the 2.0 mL mark on the 110-mL volumetric flask, transfer upper portion of the solution from the 1-L flask until such volume is obtained. Cap and stir the contents of the flask for 30 minutes followed by 15 minutes for phase separation. Combine the second acetonitrile portion with the initial extract in the 15-mL tube. Nitrogen blow-down to 5 mL. Then transfer to a 5- mL volumetric flask. Dilute to volume with acetonitrile.
- 7.2.2.10 If the diluted extract is cloudy, filter it through a 0.45- μ m PTFE filter using an acetonitrile rinsed 10-mL syringe. Discard the first 0.5 mL of filtrate and retain the remainder in a Teflon-capped vial for HPLC analysis.

8.0 CALIBRATION

- 8.1 The calibration ranges for each analyte in this method are listed below. The low values represent the lowest standard analyzed during initial calibration and are the PQLs (practical quantitation limits). The PQLs will be used as the lower reporting limit unless project specific reporting limits are provided.

<u>Analyte Code</u>	<u>Soil</u>	<u>Water</u>
HMX	0.20 µg/g to 100 µg/g	0.26 µg/L to 130 µg/L
RDX	0.20 µg/g to 100 µg/g	0.26 µg/L to 130 µg/L
NB	0.20 µg/g to 100 µg/g	0.26 µg/L to 130 µg/L
TETRYL	0.20 µg/g to 100 µg/g	0.26 µg/L to 130 µg/L
135TNB	0.10 µg/g to 50 µg/g	0.13 µg/L to 65.0 µg/L
13DNB	0.10 µg/g to 50 µg/g	0.13 µg/L to 65.0 µg/L
246TNT	0.20 µg/g to 100 µg/g	0.26 µg/L to 130 µg/L
24DNT	0.10 µg/g to 50 µg/g	0.13 µg/L to 65.0 µg/L
26DNT	0.20 µg/g to 200 µg/g	0.52 µg/L to 260 µg/L
2NT	1.00 µg/g to 200 µg/g	0.52 µg/L to 260 µg/L
3NT	1.00 µg/g to 200 µg/g	0.52 µg/L to 260 µg/L
4NT	1.00 µg/g to 200 µg/g	0.52 µg/L to 260 µg/L
2-Am-DNT	0.20 µg/g to 100 µg/g	0.26 µg/L to 130 µg/L
4-Am-DNT	0.20 µg/g to 100 µg/g	0.26 µg/L to 130 µg/L

8.2 Preparation of Calibration Standards

- 8.2.1 Separate primary stock standards are prepared at a concentration of 1.00 mg/mL by dissolving and/or diluting known amounts of each compound in an appropriate amount of acetonitrile (see Table 1). Preparation is recorded in the concentrate stock standard preparation logbook. Primary stock standards are stored at 4°C ± 2°C in glass test tubes with Teflon-lined screw caps. Replace after one year, or sooner if comparison with check standards indicates a problem.
- 8.2.2 A combined standard solution of intermediate concentration is prepared by following the procedures outlined in Table 2. The intermediate solution is stored at 4°C ± 2°C in a glass container with a Teflon-lined screw cap. Replace after six months, or sooner if comparison with check standards indicates a problem.
- 8.2.3 Working standards are prepared by transferring, via syringe, portions of the intermediate solution (from Table 2) to 1 mL volumetric flasks then diluting to final volume with acetonitrile. Refer to Table 3 for the preparation of the working standards.
- 8.2.4 Alternatively, commercially prepared solutions are used if they are certified by the manufacturer or an independent source. They are diluted in an appropriate amount of acetonitrile to yield the concentrations listed in Table 2.

- 8.2.5 Calibration standards are stored at $4^{\circ}\text{C} \pm 2^{\circ}\text{C}$ in glass containers with Teflon-lined screw caps. Replace after two months, or sooner if comparison with check standards indicates a problem.
- 8.2.6 An initial calibration verification (ICV), prepared by a third party (other than QC or the analyst) or purchased from an outside source, is prepared in the same manner and at the same concentration as the calibration standard SW8330/#5 (see Table 3). The ICV is stored at $4^{\circ}\text{C} \pm 2^{\circ}\text{C}$ in a glass container with a Teflon-lined screw cap. Replace after two months, or sooner if comparison with standards indicates a problem.
- 8.2.7 A continuing calibration standard (CCS) is prepared in the same manner and at the same level as the calibration standard SW8330/#5 (see Table 3). The CCS is stored at $4^{\circ}\text{C} \pm 2^{\circ}\text{C}$ in a glass container with a Teflon-lined screw cap. Replace after two months, or sooner if comparison with check standards indicates a problem.

8.3 Initial Calibration

- 8.3.1 The instrument is allowed to warm up for approximately 30 minutes or until a stable baseline has been established. This allows the column and detector to equilibrate.
- 8.3.2 Each of the initial calibration standards listed in Table 3 is analyzed in triplicate. For each analyte, instrument response is plotted versus standard concentration using an average response of the triplicate injections for each concentration level. A linear fit of the calibration data is used to construct the initial calibration curves. The acceptance criteria for the initial calibration curve is a correlation coefficient of 0.995 or higher. Once the acceptance criteria are met, sample results are then quantitated against the initial calibration curves.
- 8.3.3 The ICV solution is analyzed after the initial calibration standards but before any samples are analyzed. The ICV is used to verify the accuracy of the standard solutions used for calibration. The ICV solution quantitated against the initial calibration curve must be within $\pm 25\%$ of the actual concentration of the ICV. Project specific ICV criteria will be used when available.

8.4 Continuing Calibration

- 8.4.1 A Continuing Calibration Standard (CCS) is analyzed in triplicate at the beginning of sample analysis and injected once after every ten samples and at the end of analysis.
- 8.4.2 The initial calibration curve for each analyte is used to quantitate each CCS analyzed and all field samples.
- 8.4.3 The results for each analyte in an CCS must be within $\pm 15\%$ of the target concentration for that analyte.
 - 8.4.3.1 Sample analyses must be bracketed between continuing calibration standards that meet the $\pm 15\%$ criteria.

8.4.4 Following appropriate corrective action, a new initial calibration for an analyte must be prepared if the CCS is not within $\pm 15\%$ of the target concentration for the analyte.

8.5 Retention Time Windows

8.5.1 Retention time windows are calculated by averaging the absolute retention times of the analytes in the continuing calibration standards collected over 72 hours, determining the standard deviation, and setting the window equal to \pm three times the standard deviation.

8.5.2 Adjustments to the retention times, if necessary, will be made based on the absolute retention time of the CCSs. Windows may be expanded based on analyst's judgement, but must be documented.

9.0 ANALYSIS

9.1 The instrument is adjusted to the parameters listed below:

9.1.1 Primary column suggested instrument conditions:

9.1.1.1 Column: 250 x 4.6 mm Phenomenex Ultracarb 5 μm ODS(20)

9.1.1.2 Eluent flow rate: 0.8 mL/minute

9.1.1.3 Column oven temperature: 28.5°C

9.1.1.4 Injection volume: 25 μL

9.1.1.5 UV absorbance wavelength: 254 nm

9.1.1.6 Eluent profile: 47% water, 53% methanol; isocratic

9.1.2 Secondary column suggested instrument conditions:

9.1.2.1 Column: 250 x 4.6 mm Supelco LC-CN with 5- μm particle size packing

9.1.2.2 Eluent flow rate: 1.5 mL/minute

9.1.2.3 Column oven temperature: 35°C

9.1.2.4 Injection volume: 50 μL

9.1.2.5 UV absorbance wavelength: 254 nm

9.1.2.6 Eluent profile: 70% water, 15% methanol; 15% acetonitrile; isocratic

9.1.3 Adjust the run time for primary or secondary column analysis so that all the analytes of interest elute off the column.

- 9.2 The instrument is operated until a stable baseline is achieved (approximately 30 minutes).
- 9.3 Mix the soil or water sample extracts and calibration standards with an equal volume of 1% calcium chloride/ortho-cresol solution. If the sample appears "cloudy," filter again through a 0.45-micron PTFE filter. The filtrate is transferred to a vial and inserted into the autosampler.
- 9.4 Perform initial calibration and continuing calibration according to Section 8.0.
- 9.5 Once calibration requirements have been met, inject the samples into the HPLC for analysis.
- 9.6 Positive results in field samples from the primary column analysis are qualitatively confirmed on the secondary column.
 - 9.6.1 A standard at the low end of the calibration range is analyzed before and after the field samples to update retention times and verify adequate sensitivity.
 - 9.6.2 A method blank is analyzed to verify the absence of interferences on the secondary column.
 - 9.6.3 Confirmation is based on the presence of a peak within an appropriate retention-time window.
 - 9.6.4 Confirmed analytes are reported using quantitative results from the primary column analysis.

10.0 QUALITY CONTROL

- 10.1 Acceptance criteria for evaluation of quality control data may be statistically determined performance based limits or fixed upper and lower acceptance limits specified by a client, contract or analytical method. The type of limits to be used and the use of project specific acceptance limits will be specified in the Project Protocol Worksheet. Refer to DCL SOP QC-DC-001 "Establishing and Updating Control Limits" and DCL SOP XX-DC-018 "Evaluation of Quality Control Data."
- 10.2 Method Blank
 - 10.2.1 One method blank will be extracted with each analytical batch, with a maximum of 20 field samples per method blank.
 - 10.2.2 The method blank must be evaluated for contamination. If any target compound is present in the method blank then follow the procedure in Figure 1.
- 10.3 Laboratory Control Sample (LCS)
 - 10.3.1 One LCS will be extracted with each analytical batch which will not exceed 20 field samples.
 - 10.3.2 The LCS verifies that the method is in control. If the LCS acceptance criteria are not met, the entire analytical batch is reextracted and reanalyzed. For evaluation of LCS

data and corrective action to be taken, follow the procedures in Figure 2.

10.4 Matrix Spike Samples

10.4.1 A matrix spike and matrix spike duplicate will be extracted and analyzed with each analytical batch which will not exceed 20 field samples.

10.4.2 For evaluation of MS and MSD data and corrective action to be taken, follow the procedures in Figure 3. Specific projects may require reextraction of MS and MSD when outside the project specific acceptance criteria.

10.5 Surrogate Standards

10.5.1 All blanks, LCS, MS, MSD as well as all other field samples will contain 3,4-Dinitrotoluene as a surrogate standard.

10.5.2 For evaluation of surrogate data and corrective action to be taken, follow the procedures in Figure 4.

11.0 CALCULATIONS

11.1 The concentration of each analyte in the sample extract is calculated by comparing the instrument responses for sample to initial calibration curves constructed from calibration standards. The final result is reached by applying correction factors for concentration steps and dilution steps.

11.2 The concentration of each analyte in the sample extract is calculated using a linear regression program. The curve-fit equation is:

$$\text{Conc. in sample extract } (\mu\text{g/mL}) = \frac{\text{Instrument Response} - \text{Y Intercept}}{\text{Slope}}$$

The final result is calculated by the following equation:

$$\text{Final Results } (\mu\text{g/L}) = A \times W \text{ (water concentration factor)}$$

$$\text{Final Results } (\mu\text{g/g}) = A \times S \text{ (soil concentration factor)}$$

Where: A = Concentration in sample extract ($\mu\text{g/mL}$)

$$W = \frac{\text{Total Volume of Extract (mL)}}{\text{Volume of Sample taken for Analysis (L)}} = \frac{5 \text{ mL}}{0.770 \text{ L}} = 6.49 \text{ mL/L}$$

$$S = \frac{\text{Total Volume of Extract (mL)}}{\text{Volume of Sample taken for Analysis (L)}} = \frac{10\text{mL}}{2\text{g}} = 5\text{mL/g}$$

11.3 CCS, surrogate and check standard recovery are calculated as follows:

$$\% \text{ Recovery} = \frac{A \times W \times 100}{T} \text{ or } \frac{A \times S \times 100}{T}$$

Where: T = Target concentration in $\mu\text{g/L}$ or $\mu\text{g/g}$

11.4 Matrix spike recovery is calculated as follows:

$$\% \text{ Recovery} = \frac{(C - D) \times 100}{T}$$

Where: C = $\mu\text{g/L}$ or $\mu\text{g/g}$ result of the spiked sample

D = $\mu\text{g/L}$ or $\mu\text{g/g}$ result of the unspike sample

11.5 Relative percent difference:

$$\% \text{ RPD} = \left(\frac{C_o - C_D}{\frac{C_o + C_D}{2}} \right) \times 100$$

Where: C_o = concentration of original sample

C_D = concentration of duplicate sample

12.0 REPORTING RESULTS

12.1 Results are reported in units of $\mu\text{g/g}$ or $\mu\text{g/L}$ unless specific contracts or QA plans specify alternate reporting units.

12.2 Reporting formats and data deliverables should be in accordance with the specific contract or QA plan governing the sample analysis. See table 9 in the appendix for Explosive Compounds (Soil & Water).

13.0 REFERENCES

13.1 EPA Method 8330, SW846, Revision 0, September 1994. United States Environmental Protection Agency, Office of Soil Waste and Emergency Response.

13.2 "Spiking Solution Control," Tom Jenkins, 1987.

13.3 "Comparison of RP-HPLC Determination for Explosives in Water," Cold Regions Research and Engineering Laboratory, April 1988.

13.4 DCL SOP XX-DC-018 "Evaluation of Quality Control Data."

13.5 DCL SOP XX-QC-001 "Establishing and Updating Control Limits."

APPENDIX

Selected physical properties of the analytes are:

<u>Analyte Code</u>	<u>m.p.</u>	<u>Density</u>
HMX	NA	NA
RDX	203 C	1.82 g/mL
NB	5.7 C	1.20 g/mL
TETRYL	NA	NA
135TNB	122.5 C	1.76 g/mL
13DNB	89 C	1.58 g/mL
246TNT	80.1 C	1.65 g/mL
24DNT	70 C	1.32 g/mL
26DNT	66 C	1.28 g/mL
2NT	-2.9°C	1.16 g/mL
3NT	15 C	1.16 g/mL
4NT	52°C	1.30 g/mL
2-Am-DNT	NA	NA
4-Am-DNT	NA	NA
3,4-DNT	61°C	1.26 g/mL

Table 1. PRIMARY STOCK STANDARDS

<u>Analyte Code</u>	<u>Analyte Weight</u>	<u>Dilution Volume</u>	<u>Name of Concentrated Stock</u>	<u>Concentrated Stock Standard Concentrations</u>
HMX	5.00 mg	5.00 mL	SW8330-HMX/A	1,000 µg/mL
RDX	5.00 mg	5.00 mL	SW8330-RDX/A	1,000 µg/mL
135TNB	5.00 mg	5.00 mL	SW8330-135TNB/A	1,000 µg/mL
13DNB	5.00 mg	5.00 mL	SW8330-13DNB/A	1,000 µg/mL
TETRYL	5.00 mg	5.00 mL	SW8330-TETRYL/A	1,000 µg/mL
246TNT	5.00 mg	5.00 mL	SW8330-246TNT/A	1,000 µg/mL
NB	5.00 mg	5.00 mL	SW8330-NB/A	1,000 µg/mL
24DNT	5.00 mg	5.00 mL	SW8330-24DNT/A	1,000 µg/mL
26DNT	5.00 mg	5.00 mL	SW8330-26DNT/A	1,000 µg/mL
2NT	5.00 mg	5.00 mL	SW8330-2NT/A	1,000 µg/mL
3NT	5.00 mg	5.00 mL	SW8330-3NT/A	1,000 µg/mL
4NT	5.00 mg	5.00 mL	SW8330-4NT/A	1,000 µg/mL
34DNT	5.00 mg	5.00 mL	SW8330-34DNT/A	1,000 µg/mL
2A46DT	5.00 mg	5.00 mL	SW8330-2A46DT	1,000 µg/mL
4A26DT	5.00 mg	5.00 mL	SW8330-4A26DT	1,000 µg/mL

Note: Solvent of dilution is acetonitrile

Table 2. STANDARD SOLUTIONS OF INTERMEDIATE CONCENTRATION

<u>Name of Source Solution</u>	<u>Volume of Source Solution</u>	<u>Flask Volume</u>	<u>Name of Intermediate Solution</u>	<u>Analyte Code</u>	<u>Concentration of Intermediate Solution</u>
SW8330-HMX/A	200 µL	10mL	SW8330/I	HMX	20.0 µg/mL
SW8330-RDX/A	200 µL			RDX	20.0 µg/mL
SW8330-135TNB/A	100 µL			135TNB	10.0 µg/mL
SW8330-13DNB/A	100 µL			13DNB	10.0 µg/mL
SW8330-TETRYL/A	200 µL			TETRYL	20.0 µg/mL
SW8330-246TNT/A	200 µL			246TNT	20.0 µg/mL
SW8330-NB/A	200 µL			NB	20.0 µg/mL
SW8330-24DNT/A	100 µL			24DNT	10.0 µg/mL
SW8330-26DNT/A	200 µL			26DNT	20.0 µg/mL
SW8330-2NT/A	400 µL			2NT	40.0 µg/mL
SW8330-3NT/A	400 µL			3NT	40.0 µg/mL
SW8330-4NT/A	400 µL			4NT	40.0 µg/mL
SW8330-34DNT/A	200 µL			34DNT	20.0 µg/mL
SW8330-2A46DT	200 µL			2A46DT	20.0 µg/mL
SW8330-4A26DT	200 µL			4A26DT	20.0 µg/mL

Note: ¹ For water samples, all analytes, except NB/A, are added to the volumetric flask and taken to dryness under a stream of nitrogen. NB/A is then added, and the solution is taken to volume with methanol.

² Soil samples, the dilution solvent is acetonitrile.

³ A is 135TNB, 13DNB, and 24DNT.

⁴ B is RDX, NB, 246TNT, Tetryl, HMX, 26DNT, 2A46DT, and 4A26DT.

⁵ C is 2NT, 3NT, and 4NT.

Table 3. INITIAL CALIBRATION WORKING STANDARDS

<u>Name of Source Solution</u>	<u>Volume of Source Solution</u>	<u>Final Volume</u>	<u>Name of Working Standard</u>	<u>Analyte Code</u>	<u>Initial Calibration Standards Concentration</u>		
					<u>µg/mL</u>	<u>µg/g (soil)</u>	<u>µg/L (water)</u>
SW8330/I	2.0 µL	1.00 mL	SW8330/\$2	A	0.020	0.10	0.13
				B	0.040	0.20	0.26
				C	0.080	0.40	0.52
SW8330/I	10 µL	1.00 mL	SW8330/\$10	A	0.100	0.50	0.65
				B	0.200	1.00	1.30
				C	0.400	2.00	2.60
SW8330/I	50.0 µL	1.00 mL	SW8330/\$50	A	0.500	2.50	3.25
				B	1.00	5.00	6.5
				C	2.00	10.	13.0
SW8330/I	200 µL	1.00 mL	SW8330/\$200	A	2.00	10.0	13.0
				B	4.00	20.0	26.0
				C	8.00	40.0	52.0
SW8330/I	1000 µL	1.00 mL	SW8330/\$1000	A	10.0	50.0	65.0
				B	20.0	100.0	130.0
				C	40.0	200.0	260.0

Note: ¹ A is 135TNB, 13DNB, and 24DNT.

² B is RDX, NB, 246TNT, TETRYL, HMX, 26DNT, 34DNT, 2A46DT, and 4A26DT.

³ C is 2NT, 3NT, and 4NT.

⁴ Solvent of dilution is acetonitrile.

Table 4. CONCENTRATED STOCK STANDARDS FOR LCS AND MATRIX SPIKE SAMPLES

<u>Analyte Code</u>	<u>Analyte Weight</u>	<u>Dilution Volume</u>	<u>Name of Primary Stock</u>	<u>Concentrated Stock Concentration</u>
RDX	60.0 mg	3.00 mL	SW8330-RDX/C	20.0 mg/mL
135TNB	50.0 mg	5.00 mL	SW8330-135TNB/C	10.0 mg/mL
246TNT	60.0 mg	3.00 mL	SW8330-246TNT/C	20.0 mg/mL
NB	60.0 mg	3.00 mL	SW8330-NB/C	20.0 mg/mL
24DNT	50.0 mg	5.00 mL	SW8330-24DNT/C	10.0 mg/mL
2NT	120 mg	3.00 mL	SW8330-2NT/C	40.0 mg/mL
2A46DT	60.0 mg	3.00 mL	SW8330-2A46DT	20.0 mg/mL

Note: Solvent of dilution is acetonitrile.

Table 5. LCS AND MATRIX SPIKING SOLUTION – SOIL

<u>Name of Source Solution</u>	<u>Volume of Source Solution</u>	<u>Flask Volume</u>	<u>Name of Matrix Spiking Solution</u>	<u>Analyte Code</u>	<u>Matrix Spiking Solution Concentrations</u>
SW8330-RDX/C	250 μ L	100 mL	SW8330/MS-S	RDX	50.0 μ g/mL
SW8330-135TNB/C	250 μ L			135TNB	25.0 μ g/mL
SW8330-246TNT/C	250 μ L			246TNT	50.0 μ g/mL
SW8330-NB/C	250 μ L			NB	50.0 μ g/mL
SW8330-24DNT/C	250 μ L			24DNT	25.0 μ g/mL
SW8330-2NT/C	250 μ L			2NT	100.0 μ g/mL
SW8330-4A26DT/C	250 μ L			2A46DT	50.0 μ g/mL

Note: Solvent of dilution is acetonitrile.

Table 6. LCS AND MATRIX SPIKING SAMPLES - SOIL

<u>QC Sample</u>	<u>Name of Source Solution</u>	<u>Volume of Source Solution</u>	<u>Sample Weight</u>	<u>Analyte Code</u>	<u>QC Sample Concentrations</u>
LCS	SW8330/MS-S	1000 µL	2 g	RDX	25.0 µg/g
				135TNB	12.5 µg/g
				246TNT	25.0 µg/g
				NB	25.0 µg/g
				24DNT	12.5 µg/g
				2NT	50.0 µg/g
				2A46DT	25.0 µg/g
MS/MSD	SW8330/MS	1000 mL	2 g	RDX	25.0 µg/g
				135TNB	12.5 µg/g
				246TNT	25.0 µg/g
				NB	25.0 µg/g
				24DNT	12.5 µg/g
				2NT	50.0 µg/g
				2A46DT	25.0 µg/g

Note: ¹ Solvent of dilution is ASTM Type II water.

² Two SPK/HIGHS are prepared for each sample lot.

Table 7. LCS AND MATRIX SPIKING SOLUTION - WATER

<u>Name of Source Solution</u>	<u>Volume of Source Solution</u>	<u>Flask Volume</u>	<u>Name of Matrix Spiking Solution</u>	<u>Analyte Code</u>	<u>Matrix Spiking Solution Concentrations</u>
SW8330-RDX/C	96.3 μ L	200 mL	SW8330/MS-W	RDX	9.63 μ g/mL
SW8330-135TNB/C	96.3 μ L			135TNB	4.81 μ g/mL
SW8330-246TNT/C	96.3 μ L			246TNT	9.63 μ g/mL
SW8330-NB/C	96.3 μ L			NB	9.63 μ g/mL
SW8330-24DNT/C	96.3 μ L			24DNT	4.81 μ g/mL
SW8330-2NT/C	96.3 μ L			2NT	19.3 μ g/mL
SW8330-4A26DT/C	96.3 μ L			2A46DT	9.63 μ g/mL

Note: Solvent of dilution is acetonitrile.

Table 8. LCS AND MATRIX SPIKING SAMPLES – SOIL

<u>QC Sample</u>	<u>Name of Source Solution</u>	<u>Volume of Source Solution</u>	<u>Sample Weight</u>	<u>Analyte Code</u>	<u>QC Sample Concentrations</u>
LCS	SW8330/MS-W	1000 µL	770 mL	RDX	25.0 µg/L
				135TNB	12.5 µg/L
				246TNT	25.0 µg/L
				NB	25.0 µg/L
				24DNT	12.5 µg/L
				2NT	50.0 µg/L
				2A46DT	25.0 µg/L
MS/MSD	SW8330/MS-W	1000 µL	700 mL	RDX	25.0 µg/L
				135TNB	12.5 µg/L
				246TNT	25.0 µg/L
				NB	25.0 µg/L
				24DNT	12.5 µg/L
				2NT	50.0 µg/L
				2A46DT	25.0 µg/L

Note: ¹ Solvent of dilution is ASTM Type II water.

² Two SPK/HIGHS are prepared for each sample lot.

Table 9: Explosive Compounds (Soil & Water)

Compound	Soil ug/g		LCS	MS/MSD	RPD
	MDL	PQL	Control Limit	Control Limit	Control Limit
1,3,5-Trinitrobenzene	0.0970	0.10	65-125	65-125	35
1,3-Dinitrobenzene	0.0626	0.10	65-125	65-125	35
2,4,6-Trinitrotoluene	0.133	0.20	65-125	65-125	35
2,4-Dinitrotoluene	0.0721	0.10	65-125	65-125	35
2,6-Dinitrotoluene	0.130	0.20	65-125	65-125	35
2-Amino-4,6-dinitrotoluene	0.132	0.20	65-125	65-125	35
2-Nitrotoluene	0.209	0.40	65-125	65-125	35
3-Nitrotoluene	0.253	0.40	65-125	65-125	35
4-Amino-2,6-dinitrotoluene	0.119	0.20			
4-Nitrotoluene	0.191	0.40	65-125	65-125	35
HMX	0.0830	0.20	65-125	65-125	35
Nitrobenzene	0.0584	0.20	65-125	65-125	35
RDX	0.133	0.20	65-125	65-125	35
Tetryl	0.145	0.20	65-125	65-125	35
3,4-Dinitrotoluene	0.182	0.2	65-125		

Compound	Water ug/L		LCS	MS/MSD	RPD
	MDL	PQL	Control Limit	Control Limit	Control Limit
1,3,5-Trinitrobenzene	0.0557	0.65	82.2 - 117.6	74.5 - 123.1	15
1,3-Dinitrobenzene	0.0507	0.65	90.7 - 121.3	78.0 - 134.0	15
2,4,6-Trinitrotoluene	0.0797	0.26	87.7 - 112.3	75.2 - 124.4	17
2,4-Dinitrotoluene	0.0521	0.65	78.3 - 116.7	68.7 - 125.7	15
2,6-Dinitrotoluene	0.0957	0.26	82.5 - 121.5	76.2 - 127.8	14
2-Amino-4,6-dinitrotoluene	0.116	0.26	89.0 - 121.0	77.5 - 130.5	15
2-Nitrotoluene	0.349	0.52	89.8 - 112.2	78.7 - 119.9	14
3-Nitrotoluene	0.446	0.52	85.5 - 113.1	77.7 - 119.1	16
4-Amino-2,6-dinitrotoluene	0.153	0.26			
4-Nitrotoluene	0.296	0.52	87.5 - 116.5	79.6 - 122.4	16
HMX	0.0991	0.26	75.4 - 115.6	71.8 - 116.8	14
Nitrobenzene	0.0670	0.26	89.7 - 112.3	78.4 - 120.6	13
RDX	0.0599	0.26	78.6 - 116.2	65.5 - 122.2	14
Tetryl	0.0993	0.26	87.8 - 120.2	64.1 - 143.9	17
3,4-Dinitrotoluene	0.159	0.26	50-150		

The values given in this table are currently in use by DataChem Laboratories, Inc. These values are subject to change due to specific project requirements and/or updates by the laboratory.

FIGURE 1
ORGANIC METHOD BLANK

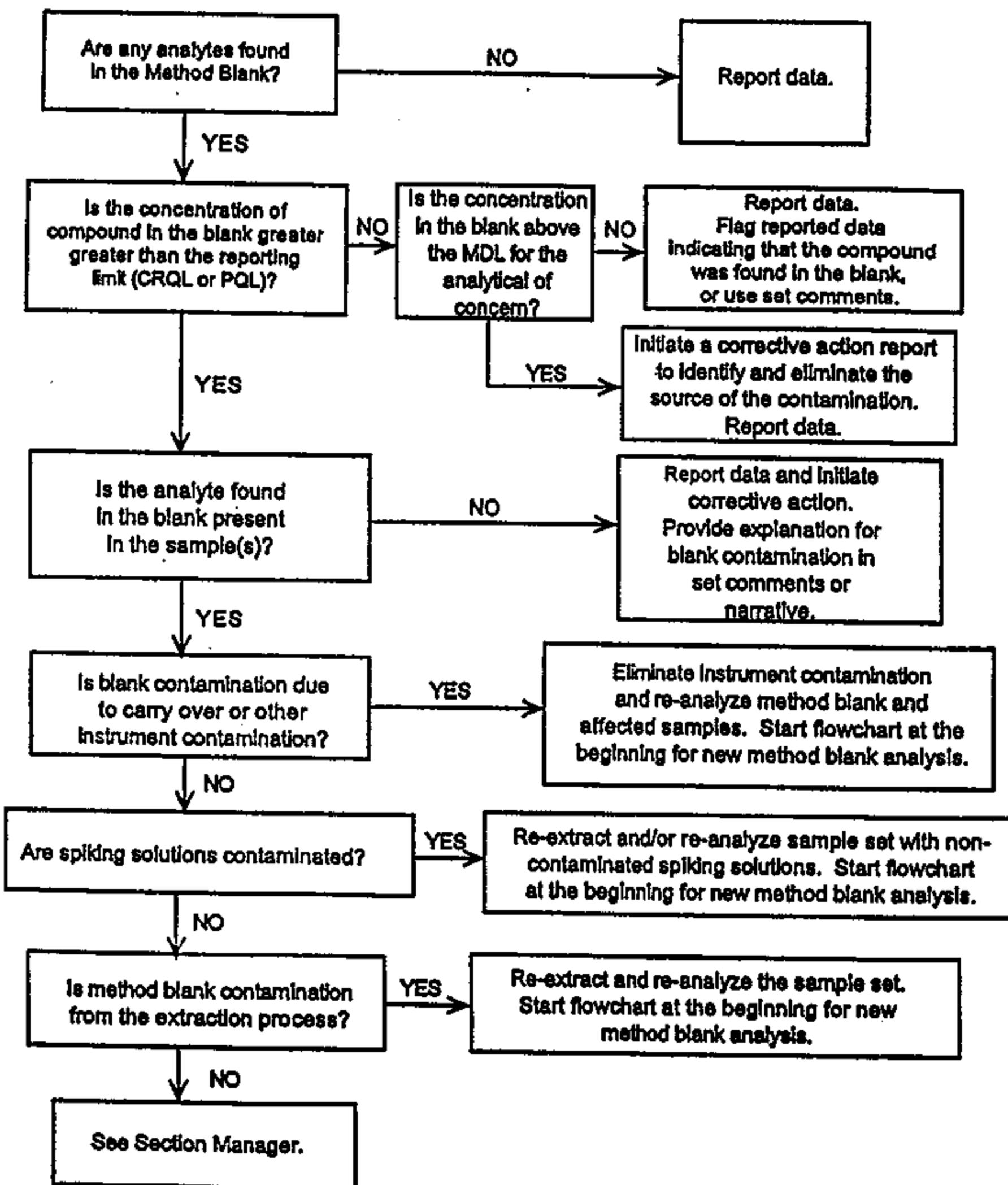
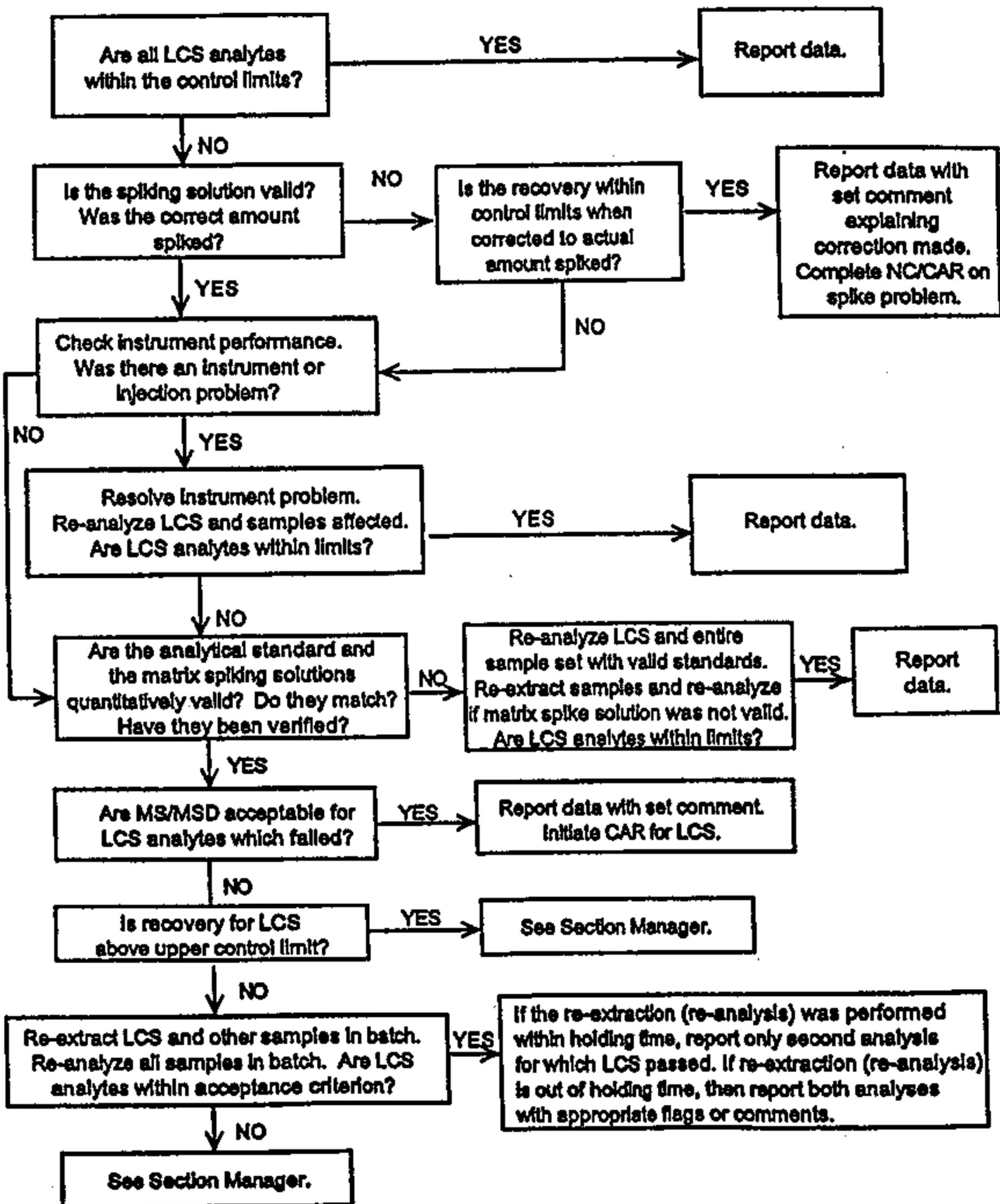


FIGURE 2
 ORGANIC LCS



**FIGURE 3
ORGANIC MS AND MSD**

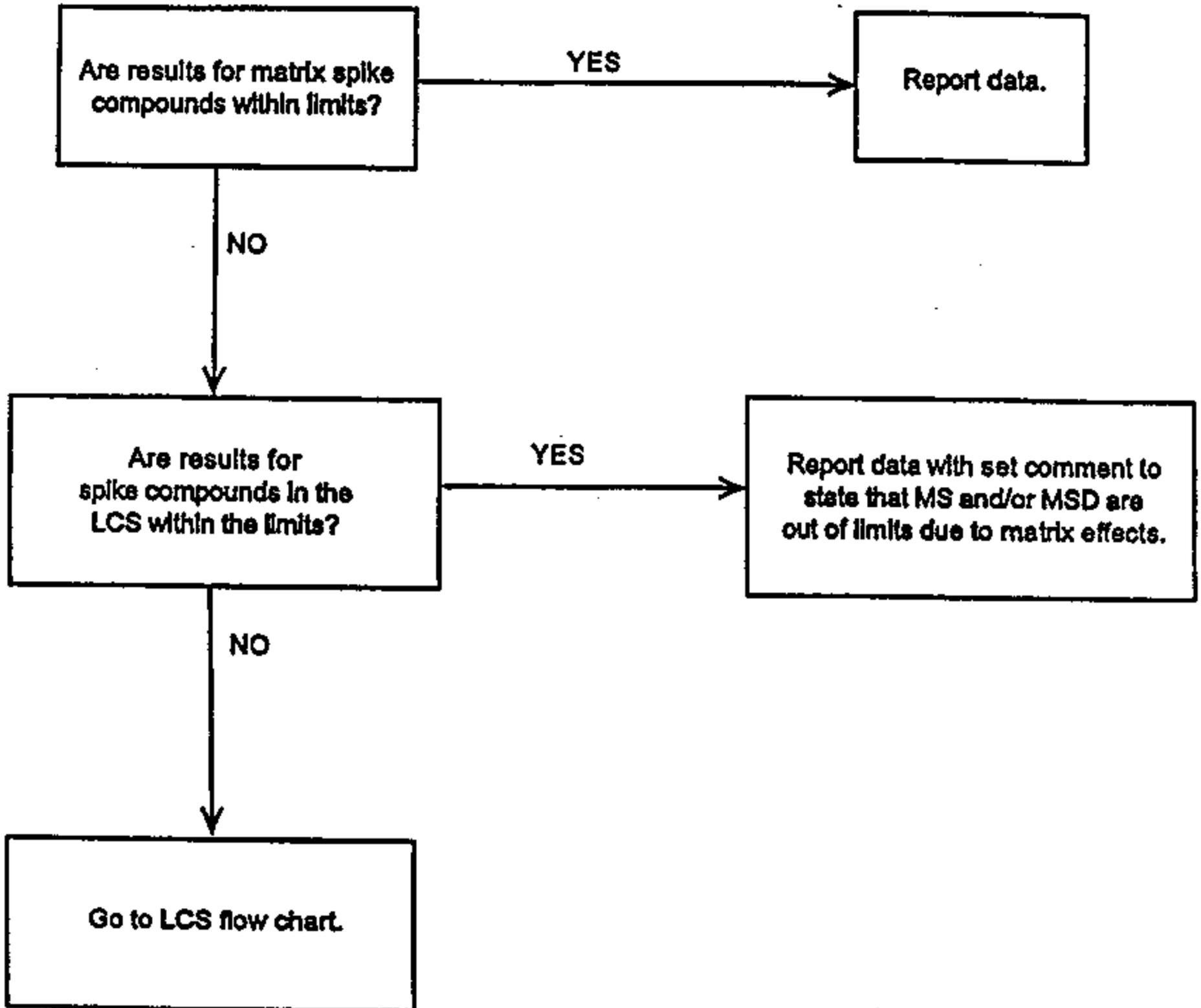
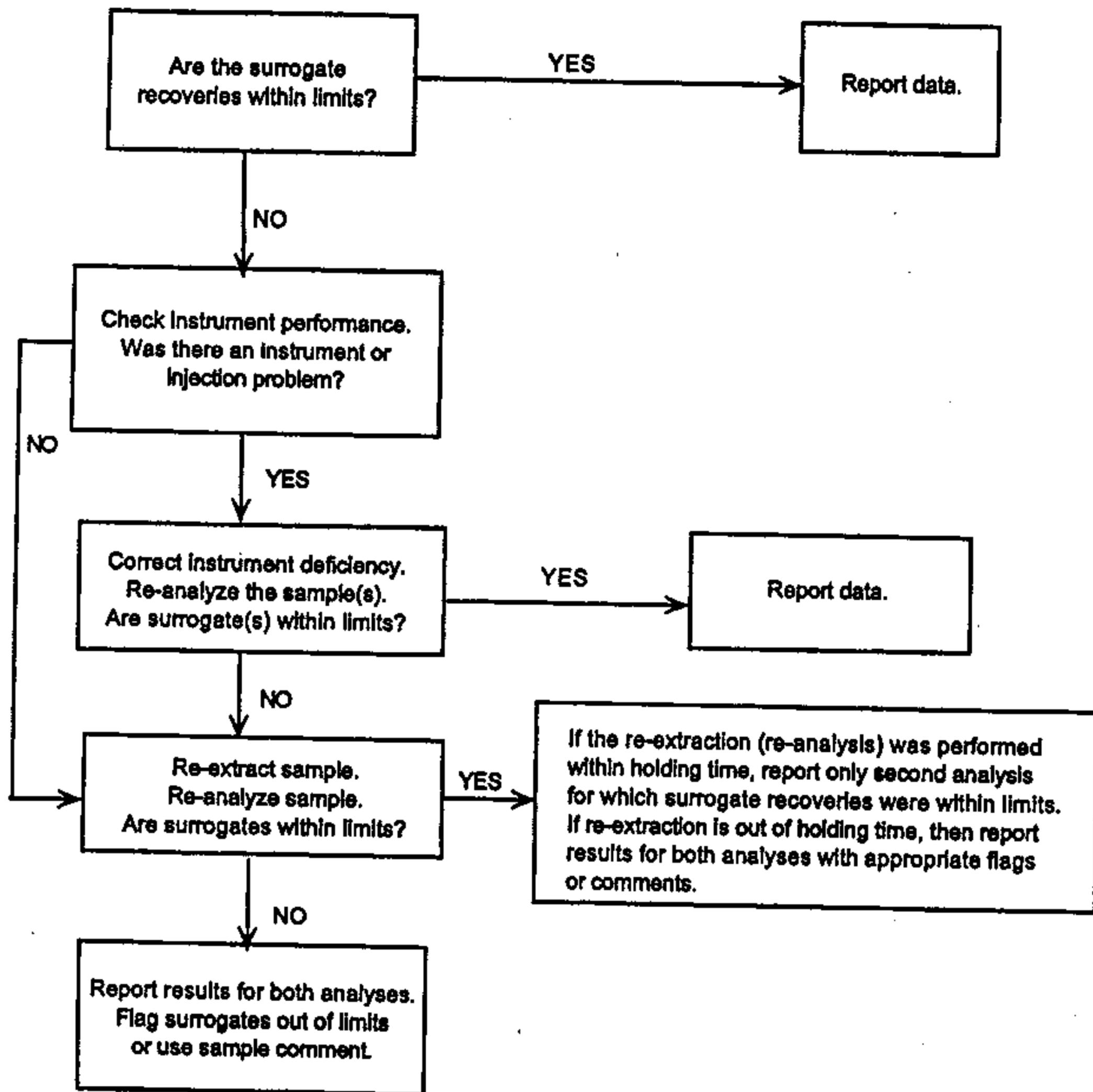


FIGURE 4
ORGANIC SURROGATE



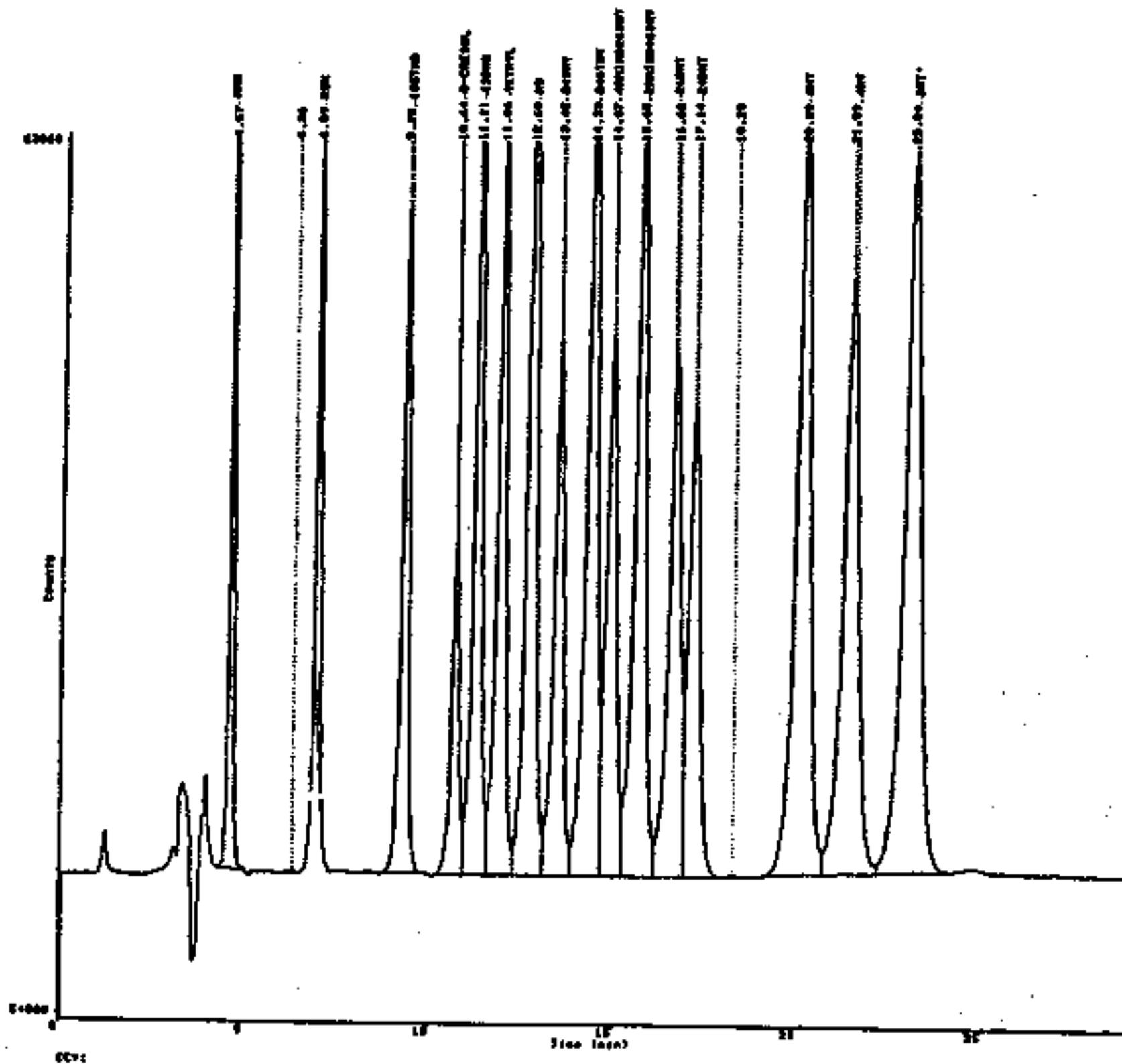


Exhibit 4

Project No.
Book No. 8353

DATA CHEM 38

E BPP6

Page No.	99W-0205-08	99E-0496-01	99E-0505-01						
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DCL HPLC Analysis Notebook

Client: SAIC, DCL, Am. West

CTIOC File: SW8330-14

Account: 8101,8001

Tecon Method(s): 8330TV (Uayo)

Method: 8330

Calibration ID: C98C7000

Matrix: Soil

Sequence(s): BPPF,

Channel/Instruments: 7/LC-8

Lot/sols: ^{ES10/9} BPPF, ESJ 10/9

Sample Preparation: Samples were extracted and prepared according to SW-846 Method 8330 which specifies 2.0 g sample and a final volume of 10mL. Extracts were mixed 1:1 with 1% CaCl₂ containing 20 ug/mL o-cresol prior to analysis (500ul CaCl₂ soln. and 500ul sample into each autosampler vial). The CCV solution is prepared by adding 100ul of CCV intermediate to 500ul CaCl₂ soln and 400 ul of ACN. Conversion is 5 mL/g for samples and QC.

Standards: CCV solution 188WS37508

Instrument conditions:

Column: Ultracarb ODS(20) 250 x 4.4mm
Eluent flow: 0.8 mL / min Inj. Volume: 25 ul
Column Oven Temp: 28.5 C UV abs. Wavelength: 254 nm
Eluent profile: 46% H₂O / 54% MeOH. Isocratic.

Confirmation analysis: For any analyte found on the primary column a confirmation analysis was performed using the conditions to the right:

Channel/Instrument/Conditions

Column: 250X4.6mm Supelco LC-CN with 5µm particle
Size packing.
Eluent flow: 1.5 mL/minute
Column oven temp: 35° C
Injection Vol: 50µl
UV absorbance wavelength: 254 nm
Eluent profile: 70% water, 15% Acetonitrile, 15% methanol
Isocratic.

Confirmation performed on channel 20, Instrument number LC9.

2.5 µl standard 188WS37511 run with confirmation.

Miscellaneous Comments: The retention time window for analytes of individual runs may be expanded to allow for RT drift of analytes in CCV's of that run.

1) Dilution for 99E03812 in separate run
~~ESJ 10/10~~

10/13
 2) Surrogate recovery high in 99E03812

To Page No. NA

Witnessed & Understood by me, <u>Vicki Tran</u>	Date <u>10/11/99</u>	Invented by <u>NOT APPLICABLE</u>	Date <u>10/9/99</u>
		Recorded by <u>Elle Jenkins</u>	

Exhibit 5

DATA CHEM LABORATORIES, INC.

STANDARD OPERATING PROCEDURE APPROVAL SHEET

SOP TITLE: Peer Review

DOCUMENT CONTROL NUMBER: XX-DC-023 Revision 1

EFFECTIVE DATE: July 30, 1998

UNCONTROLLED COPY
SUBJECT TO REVISION WITHOUT NOTICE

APPROVALS:

MANAGER

BMP

Date 9-14-98

Q A MANAGER

BMP

Date 9-14-98

LAB DIRECTOR

[Signature]

Date 9/11/98

STANDARD OPERATING PROCEDURE

PEER REVIEW

1.0 SCOPE AND APPLICATION

- 1.1 This Standard Operating Procedure (SOP) summarizes the DCL peer review process used to verify data from the laboratory for accuracy. These procedures are used by those individuals responsible for peer review of data.
- 1.2 This SOP process does not supersede any client, method or other SOP QC requirements. Criteria listed in this SOP should be used when method, SOP or client criteria is unavailable.
- 1.3 This SOP does not include any Data Package Review. Refer to SOP XX-DC-020 or the specific project protocol worksheet (PPW).

2.0 RESPONSIBILITY

- 2.1 It is the responsibility of each section manager that peer reviewers in their operational section are trained in the procedures outlined in this SOP.
- 2.2 It is the responsibility of the peer reviewer to check data in accordance with Section 4.0 of this SOP. It is also their responsibility to ensure that appropriate evaluation criteria are used as defined in the PPW. The evaluation criteria are prioritized as follows:
 - 2.2.1 As defined by the client.
 - 2.2.2 As defined by the DCL method SOP.
 - 2.2.3 As defined by a published or promulgated method.
 - 2.2.4 As defined by this SOP.

3.0 DEFINITIONS

- 3.1 Peer Review is defined as a checking procedure by a peer chemist or analyst who is knowledgeable of the analytical requirements for the specific method being employed.
- 3.2 Acronyms
 - CCB = Continuing Calibration Blank
 - CCC = Calibration Check Compounds
 - CCV = Continuing Calibration Verification

COC	=	Chain of Custody
GC	=	Gas Chromatography
GC/MS	=	Gas Chromatography/Mass Spectrometry
HPLC	=	High Pressure Liquid Chromatography
ICB	=	Initial Calibration Blank
ICV	=	Initial Calibration Verification
ICP	=	Inductively Coupled Plasma
LCS	=	Laboratory Control Sample
LOD	=	Limit of Detection (MDL)
LOQ	=	Limit of Quantitation (PQL)
MD	=	Matrix Duplicate
MDA	=	Minimum Detectable Activity
MDL	=	Method Detection Limit (LOD)
MS	=	Matrix Spike
MSD	=	Matrix Spike Duplicate
PPW	=	Project Protocol Worksheet
PQL	=	Practical Quantitation Limit (LOQ)
SPCC	=	System Performance Check Compounds
QC	=	Quality Control

4.0 GENERAL PROCEDURES

4.1 The following procedures should be used to review all data prior to submission of the final report. For each operational section, specific review procedures are listed in Sections 5.1 to 5.7 of this SOP.

4.1.1 Method performed with modifications/deviations noted.

4.1.2 Instructions in Project Protocol Worksheet are followed.

4.1.3 Analytical Report form completed and signed by analyst.

4.1.4 Notebooks reviewed and signed.

4.1.4.1 Sample Preparation/Extraction Logs

4.1.4.2 Standards Logs

4.1.4.3 Instrument Logs

4.1.5 LOD/LOQ entered and meet requirements from client.

4.1.6 Instrument QC in compliance with deviations noted.

4.1.6.1 Calibration, CCV, ICV, CCC, SPCC, and Internal Standards

4.1.6.2 GC/MS Tuning

4.1.6.3 Radiochemistry - efficiencies and background

4.1.7 Method QC in compliance with deviations noted.

4.1.7.1 Method Blanks, Laboratory Control Samples, Surrogates and Tracers

4.1.8 Matrix QC in compliance with deviations noted.

4.1.8.1 Matrix Duplicate

4.1.8.2 Matrix Spike and Matrix Spike Duplicate

4.1.9 Holding times met.

4.1.10 Solutions and standards expiration checked.

4.1.11 Units and conversions clearly defined.

4.1.12 Sample Results:

4.1.12.1 Example calculations provided and checked.

4.1.12.2 Relative Retention Times checked.

4.1.12.3 Confirmation analysis run.

4.1.12.4 Manual integrations checked.

4.1.13 Internal COC is complete.

5.0 OPERATIONAL SECTION PROCEDURES

5.1 Inorganic Chemistry Technical Peer Review

Note: It is the peer reviewer's responsibility to ensure that appropriate criteria are used as defined in the PPW. The evaluation criteria are prioritized as per Section 2.2 of this SOP. If the evaluation criteria have not been defined by the PPW, published method, or SOP then the criteria below enclosed in parentheses should be used unless otherwise directed by a laboratory supervisor. The asterisked items are only required for Federal projects, unless otherwise defined in the PPW.

- Solutions standardized if required by method
- Calibration standards analyzed

- *
 - Standards traceability checked
 - Standard Curve Coefficient (> 0.995)
 - ICV analyzed and meet acceptance criteria ($<15\%$)
 - CCV analyzed and meet acceptance criteria ($<15\%$)
 - ICB, CCB analyzed and meet acceptance criteria
 - CCB/CCV frequency met (≤ 10)
 - Method/Preparation Blank analyzed and meet acceptance criteria when performed
 - MS, MSD, and/or MD analyzed and meet acceptance criteria when performed
 - LCS analyzed and meet acceptance criteria when performed
 - Method deviations and reanalysis noted when performed
 - Preparation and analysis Holding Times met
 - Dilution factors noted
 - Notebook pages - transcription accuracy and completeness
 - Calculations checked
 - Report Forms are complete and accurate
 - LOD/LOQ entered and meet requirements
- *
 - Internal COC completed

5.2 Atomic Absorption Technical Peer Review

Note: It is the peer reviewer's responsibility to ensure that appropriate criteria are used as defined in the PPW. The evaluation criteria are prioritized as per Section 2.2 of this SOP. If the evaluation criteria have not been defined by the PPW, published method, or SOP then the criteria below enclosed in parentheses should be used unless otherwise directed by a laboratory supervisor. The asterisked items are only required for Federal projects, unless otherwise defined in the PPW.

- Calibration Standards analyzed and checked
- *
 - Standards traceability checked
 - Standard Curve Coefficient (advisory > 0.995)
 - ICV analyzed and meet acceptance criteria
 - CCV analyzed and meet acceptance criteria
 - ICB, CCB analyzed and meet acceptance criteria

- CCB/CCV frequency met (≤ 10)
- Method/Preparation Blank analyzed and meet acceptance criteria
- MS, MSD, and MD analyzed and calculations checked, applicable action based on recoveries has been taken
- LCS analyzed and meet acceptance criteria
- Method QC recoveries meet acceptance criteria for those performed
- Method deviations and reanalysis noted when performed
- Preparation and analysis Holding Times met
- Sample dilution factors noted on reports
- Notebook pages - transcription accuracy and completeness
- Preparation and Analysis Calculations checked
- Report Forms are complete and accurate
- Precision of injections checked
- Reanalysis checked, documented, and reported
- LOD/LOQ entered and meet requirements
- * • Internal COC completed

5.3 ICP Technical Peer Review

Note: It is the peer reviewer's responsibility to ensure that appropriate criteria are used as defined in the PPW. The evaluation criteria are prioritized as per Section 2.2 of this SOP. If the evaluation criteria have not been defined by the PPW, published method, or SOP then the criteria below enclosed in parentheses should be used unless otherwise directed by a laboratory supervisor. The asterisked items are only required for Federal projects, unless otherwise defined in the PPW.

- Calibration Standards analyzed and checked
- * • Standards traceability checked
- Standard Curve Coefficient (advisory > 0.995)
- ICV analyzed and meet acceptance criteria
- CCV analyzed and meet acceptance criteria
- ICB, CCB analyzed and meet acceptance criteria
- CCB/CCV frequency met (≤ 10)
- Method/Preparation Blank analyzed and meet acceptance criteria
- MS, MSD, and MD analyzed and calculations checked, applicable action

based on recoveries has been taken

- LCS analyzed and meet acceptance criteria
- Method QC recoveries meet acceptance criteria for those performed
- Method deviations and reanalysis noted when performed
- Preparation and analysis Holding Times met
- Sample dilution factors noted on reports
- Notebook pages - transcription accuracy and completeness
- Calculations checked
- Report Forms are complete and accurate
- Precision of injections checked
- Reanalysis checked, documented, and reported
- Check dilutions for interferences
- Check Serial Dilutions
- LOD/LOQ entered and meet requirements
- * • Internal COC completed

5.4 Ion Chromatography Technical Peer Review

Note: It is the peer reviewer's responsibility to ensure that appropriate criteria are used as defined in the PPW. The evaluation criteria are prioritized as per Section 2.2 of this SOP. If the evaluation criteria have not been defined by the PPW, published method, or SOP then the criteria below enclosed in parentheses should be used unless otherwise directed by a laboratory supervisor. The asterisked items are only required for Federal projects, unless otherwise defined in the PPW.

- Calibration Standards analyzed
- * • Standards traceability checked
- Standard Curve Coefficient (advisory >0.995)
- ICV analyzed and meet acceptance criteria (10% from true)
- CCV analyzed and meet acceptance criteria (10% from true)
- ICB, CCB analyzed and meet acceptance criteria
- Retention Time Windows (3 x RSD with analysts judgement)
- CCB/CCV frequency met (≤ 10)
- Method/Preparation Blank analyzed and meet acceptance criteria
- MS, MSD, MD analyzed and meet acceptance criteria when performed

- LCS analyzed and meet acceptance criteria
- Method deviations and reanalysis noted when performed
- Preparation and analysis Holding Times met
- Dilution factors noted
- Notebook pages - transcription accuracy and completeness
- Calculations checked
- Report Forms are complete and accurate
- LOD/LOQ entered and meet requirements
- * • Internal COC completed
- Manual Integration checked

5.5 Chromatography (GC and HPLC) Technical Peer Review

Note: It is the peer reviewer's responsibility to ensure that appropriate criteria are used as defined in the PPW. The evaluation criteria are prioritized as per Section 2.2 of this SOP. If the evaluation criteria have not been defined by the PPW, published method, or SOP then the criteria below enclosed in parentheses should be used unless otherwise directed by a laboratory supervisor. The asterisked items are only required for Federal projects, unless otherwise defined in the PPW.

- Calibration Standards analyzed
- * • Standards traceability checked
- Initial Calibration within method or project criteria
- ICV analyzed and meet acceptance criteria
- CCV analyzed and meet acceptance criteria
- ICB, CCB analyzed and meet acceptance criteria
- Retention Time Windows (3 x RSD with analysts judgement)
- Surrogate recoveries checked and appropriately addressed
- All samples bracketed by valid CCV
- MS, MSD, MD recoveries checked and appropriately addressed
- LCS analyzed and meet acceptance criteria
- Analysis deviations and reanalysis noted when performed
- Preparation and analysis Holding Times met
- Dilution factors noted
- Notebook pages and spreadsheets - transcription accuracy and

completeness

- Calculations checked
- Report Forms are complete and accurate
- Preparation deviations and reprep noted when performed
- LOD/LOQ entered and meet requirements
- * • Internal COC completed
- Manual integrations checked

5.6 GC/MS Technical Peer Review

Note: It is the peer reviewer's responsibility to ensure that appropriate criteria are used as defined in the PPW. The evaluation criteria are prioritized as per Section 2.2 of this SOP. If the evaluation criteria have not been defined by the PPW, published method, or SOP then the criteria below enclosed in parentheses should be used unless otherwise directed by a laboratory supervisor. The asterisked items are only required for Federal projects, unless otherwise defined in the PPW.

- GC/MS Tuning passed criteria (BFB or DFTPP)
- * • Standards traceability checked
- Initial Calibration passed criteria
- Continuing Calibration passes criteria
- Instrument Blank analyzed and meet acceptance criteria
- Relative Retention Time (within 0.060 min.)
- Internal Standard (> 50% CCB area counts and RT within \pm 30 seconds)
- Surrogate recoveries checked when performed
- Sample Frequency - within 12 hours of successful tune
- Method/Preparation Blank analyzed and meet acceptance criteria
- MS and MSD analyzed and meet acceptance criteria
- LCS analyzed and meet acceptance criteria
- Method deviations and reanalysis noted when performed
- Preparation and analysis Holding Times met
- Dilution factors noted
- Notebook pages and spreadsheets - transcription accuracy and completeness
- Calculations checked

- Report Forms are complete and accurate
- Preparation deviations and reprep noted when performed
- LOD/LOQ entered and meet requirements
- * • Internal COC completed
- Manual integrations checked

5.7 Radiochemistry Technical Peer Review

Note: It is the peer reviewer's responsibility to ensure that appropriate criteria are used as defined in the PPW. The evaluation criteria are prioritized as per Section 2.2 of this SOP. If the evaluation criteria have not been defined by the PPW, published method, or SOP then the criteria below enclosed in parentheses should be used unless otherwise directed by a laboratory supervisor. The asterisked items are only required for Federal projects, unless otherwise defined in the PPW.

- Background Checks current
- Daily efficiencies analyzed
- Tracers are compliant
- Method/Preparation Blank below MDA when performed
- MD analyzed and meet acceptance criteria when performed
- LCS analyzed and meet acceptance criteria when performed
- Method deviations and reanalysis noted when performed
- Dilution factors noted
- Notebook and Spreadsheet - transcription accuracy and completeness
- Calculations checked
- Report Forms are complete and accurate
- MDA entered and meet requirements
- * • Internal COC completed

6.0 REFERENCES

- 6.1 DCL SOP XX-DC-020 "Deliverable and Data Package Preparation and Review."
- 6.2 DCL SOP XX-DC-006 "Chain of Custody and Laboratory Tracking."
- 6.3 DCL SOP QC-DC-006 "Nonconformance/Corrective Action Report (NC/CAR) Procedures."
- 6.4 DCL SOP XX-DC-001 "Analytical Data Record Keeping."

6.5 DCL SOP XX-DC-019 "Standards Purity, Preparation, Traceability and Verification."

6.6 DCL SOP WR-DC-001 "The Acquisition, Preparation, and Use of Radioactive Standard Reference Materials."