

SOP# OM0071.0	EFFECTIVE DATE: 5/6/02	Standard Operating Procedure for Determination of Nitramines and Nitroaromatics in Water by Gas Chromatography
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Appendix A

1. Summary of Procedure

This procedure is suitable for the determination of nitroaromatic and nitramine compounds (explosives) present in water samples at individual compound concentrations of at least 0.01 $\mu\text{g/L}$. This method is applicable to those analytes that are (1) efficiently extracted from water by either toluene or isoamyl acetate as used in the extraction of two separate aliquots of samples, and (2) sufficiently volatile and thermally stable for determination by gas chromatography with electron-capture detection.

2. Scope and Application

2.1 Analytes:

This method is applicable to the following nitroaromatic and nitramine compounds:

<u>Compounds</u>	<u>Method Reporting Limit</u> <u>($\mu\text{g/l}$)</u>
Nitrobenzene (NB)	<0.05
2-Nitrotoluene (2-NT)	<0.2
3-Nitrotoluene(3-NT)	<0.2
4-Nitrotoluene(4-NT)	<0.2
1,3-Dinitrobenzene(1,3-DNB)	<0.05
2,6-Dinitrotoluene(2,6-DNT)	<0.01
2,4-Dinitrotoluene(2,4-DNT)	<0.01
2,3-Dinitrotoluene(2,3-DNT)	<0.01
*3,4-Dinitrotoluene(3,4-DNT)	<0.01
1,3,5-Trinitrobenzene(TNB)	<0.1
2,4,6-Trinitrotoluene(TNT)	<0.01
RDX	<1.0
4-Amino 2,6-DNT(4-Amino)	<0.05
3,5-Dinitroaniline(3,5-DNA)	<0.2
2-Amino 4,6-DNT(2-Amino)	<0.05
Tetryl	<0.1

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* 3,4-Dinitrotoluene is used as the method surrogate

2.2 Applicable matrices: Whole and dissolved water matrices.

2.3 Dynamic Range: 1-150 $\mu\text{g/L}$ without dilution.

3. Safety Issues

Always use proper safety procedures when handling nitroaromatic and nitramine compounds as well as the extraction solvents-toluene and isoamyl acetate. It is essential to read Material Safety Data Sheets (MSDSs) on each compound and solvent prior to performing this method. MSDS's can be found in the safety office for these compounds and solvents.

4. Sample Preservation and Holding Times

Each water sample received for extraction and analysis must be extraction within seven days of collection or four days from receipt at the laboratory. As soon as a sample is received, place it in a dark, explosion-proof refrigerator until it is ready to be extracted.

5. Reagents and Standards

5.1 Reagents: Pesticide grade toluene and methanol

5.2 Standard Types (See SOP for Standard Making for preparation instructions):

Calibration standards-Working standards of the entire group of the individual analytes listed in section 2.1 are prepared at 1,5, 7, 10, 20, 50, 100, 200 $\text{pg}/\mu\text{l}$ in toluene.

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Spiking solutions- The spike solution includes all individual analytes minus 3,4-Dinitrotoluene at around 5000 pg/*ul* in methanol.

Surrogate solution-The surrogate solution contains 3,4-Dinitrotoluene at around 5000 pg/*ul* in methanol.

Third Party Check- The third party check solution is commercially available through Supelco or other vendors and contains Nitrobenzene, 1,3- Dinitrobenzene, 2,4-Dinitrotoluene, and 2,6-Dinitrotoluene at concentrations of 20 pg/*ul* each in toluene. This standard is used to verify the calibration curve for each gc run.

Continuing Calibration Check Solution- The continuing calibration verification solution (CCV) concentration is made at the mid-point calibration range, typically the 20 pg/*ul* standard. A CCV is run every ten samples. It verifies that the initial calibration is still linear.

5.3 Shelf life/Storage requirements: All standards are good for three months from date of preparation. Standards are stored in amber glass vials and kept in an explosion-proof freezer at approximately -10°C to -12°C.

6. QA/QC Requirements

Definitions of Analytical Run Sequence Samples, Acceptance Criteria, and Corrective Action Required

6.1 Wash: An initial injection of clean toluene to determine baseline is stable and instrument is clean.

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6.2 Calibration Standards for Nitroaromatic/Nitroamine Individual Analytes:

These standards contain NB, 2-NT, 3-NT, 4-NT, 1,3-DNB, 2,6-DNT, 2,4-DNT, 2,3-DNT, 3,4-DNT, TNB, TNT, RDX, 4-Amino, 3,5-DNA, 2-Amino and Tetryl at 1, 5, 7, 10, 20, 50, 100 and 200 pg/uL concentrations. The analyst must include the 1, 5, 7, 10, 20, 50 and 100 pg/uL standards in every run. It is best to calibrate with a low curve that contains the 1, 5, 7, 10, and 20 pg/uL standards, and a high curve that contains the 7, 10, 20, 50 and 100 pg/uL standards. To be considered acceptable, the standards must come within 20% of their expected value and the R² value for the linear calibration line must be 0.995 or greater. If a standard seems to be nonlinear, see section 7.4.5 for instructions. The minimum number of standards considered acceptable for a curve is four.

6.3 Third Party Check Standard: The third party check standard is purchased premade from a vendor such as Supelco. The solution should contain at least 4 of the Nitroaromatic/Nitroamine analytes at individual concentrations of about 20 pg/uL. This solution must fall within +/- 30% of this value. If not, it is up to the analyst to determine the cause and rectify the problem.

6.4 Reagent Blank: A Reagent Blank of distilled water is prepared with each set to monitor potential contamination of the samples during the prep procedure. There must be one Reagent Blank prepared for every set of samples extracted. Reportable analytes should not be detected at or above the reporting level; if there are, a supervisor must be contacted to determine the course of action. Ten microliters of the method surrogate solution is added to each blank. Surrogate recovery in the blank must fall within current control limits, an example of which is given in Appendix A, or a supervisor must be contacted.

6.5 Reagent Spike: A Reagent Spike containing all of the individual nitroaromatic and nitramine analytes is prepared with each set to monitor method

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performance. Ten microliters of the method surrogate solution and ten microliters of the method spiking solution is added to the Reagent Spike. There must be at least one Reagent Spike prepared for every set extracted. Surrogate and analyte recovery in the spike must fall within current control limits, an example of which is given in Appendix A, or a supervisor must be contacted to determine the course of action. It is the responsibility of each individual analyst to produce control charts on a monthly basis for surrogate and spike recoveries.

6.6 Samples: Ten microliters of the method surrogate solution is added to each sample prior to extraction. Surrogate recovery is monitored by the analyst and reported to the client as a way of measuring gross method performance. Surrogate recovery must fall within current control limits, an example of which is given in Appendix A, or a supervisor must be contacted to determine the course of action. Sample surrogate recovery should not be the sole criterion for rejecting sample data since individual matrices will occasionally interfere with surrogate recoveries. It is the responsibility of each individual analyst to produce real-time control charts of sample surrogate recoveries monthly.

6.7 CCV: Continuing Calibration Check. This is a mid-range standard (20 pg/ul) that is run every 10 samples to monitor analytical performance and reproducibility. Expected values should fall within +/-30% for all analytes. If the CCV exceeds the acceptable range for an analyte on one column but not on the other, it is acceptable to make calls on the column that is acceptable and confirm calls on the one that is not. If, during analysis, a calibration check standard shows any analyte is outside +/-30% of the expected value on both columns, the samples following the last acceptable CCV are suspect. If suspect samples contain detectable amounts of any of the out-of-control analytes, they will need to be rerun for those analytes. If the suspect samples

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appear to be blank, and the report threshold can be met, then the samples will not require reruns. Document the action taken on the QC Check sheet (see Appendix A).

7. Procedure:

7.1 Comments:

7.1.1 Interferences: This procedure involves two separate extractions using two different solvents on separate-100 ml aliquots of the same sample. The second extraction is performed using isoamyl acetate. Isoamyl acetate must be double distilled prior to extraction to prevent interferences.

7.1.2 Once samples are received by the laboratory, they are logged in and prepared by the method given in section 7.3.

7.2 Labware

7.2.1 Double Distillation of Isoamyl Acetate-Assemble a distillation apparatus like the one below: (figure 7.2.1)

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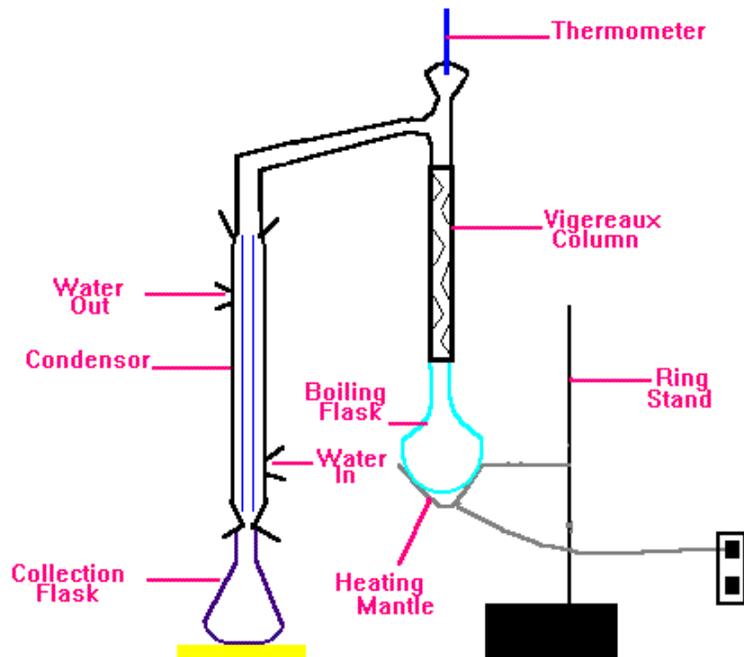


Figure 7.2.1 Distillation Apparatus

Place the undistilled isoamyl acetate in the boiling flask with a couple of boiling chips. Cover the boiling flask and vigreux column with aluminum foil. Plug-in the heating mantle. Dispose of any solvent collected up to about 95°C. Collect any solvent after the solvent is heated greater than 95°C. When the solvent left in the boiling flask is about 2 mls with a tint of yellow, stop the distillation and discard the remaining solvent. Take the solvent collected and perform the same procedure again. After the second distillation, the solvent is ready to use for extraction.

7.3 Sample Preparation

7.3.1 Toluene Extraction:

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Equipment and Consumables

- (1) pesticide grade toluene
- (2) method surrogate and spike
- (3) 10 ul microdispenser
- (4) rinse methanol for microdispenser
- (5) 100 ml volumetric flasks and caps
- (6) 1-inch teflon coated magnetic stir bars (cleaned with alconox detergent, rinsed in acetone, and heated at 260°C for 2 hours.)
- (7) magnetic stir plates
- (8) deionized water
- (9) disposable borosilicate pipets (burned at 480°C for 3 hours)
- (10) amber borosilicate vials (burned at 480°C for 3 hours)

Extraction Procedure

- (1) Rinse all glassware with toluene and allow to dry completely.
- (2) Remove samples from refrigerator and allow them to equilibrate to room temperature.
- (3) Mix each sample well and measure 100ml of the sample into a 100ml volumetric flask.
- (4) Make a blank by measuring 100ml of deionized water into a 100ml volumetric flask. Prepare a spike the same way.

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(5) Carefully add a magnetic stir bar to each volumetric flask. Try to avoid spilling any. Replace with more sample in that case.

NOTE: Stir bars must be burned prior to extraction. They should be burned at 260°C for at least an hour.

(6) Add 10 μ l of surrogate solution to each sample, blank, and reagent spike.

(7) Add 10 μ l of spiking solution to reagent spike.

(8) Mix each sample well.

(9) Add, using a volumetric pipet, 1ml exactly of toluene to each sample, blank, and spike.

(10) Place each sample on a magnetic stir plate and extract for 30 minutes. The toluene should be pulled down by the stirring motion in tiny droplets. It should look like the motion of a tornado.

NOTE: Make sure stir bar is stirring in middle of volumetric flask and stirring continuously. Stir bars that spin out of control can break the volumetric flask, losing the sample.

(11) After the 30 minutes, turn off the stir plates and allow the toluene to move back up to the top of the sample. This should take about 15 minutes.

(12) After the toluene has settled, using a disposable pipet, pipet as much as the extract as possible into an amber GC vial without getting any of the water.

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NOTE: Sometimes emulsions occur and must be broken. If they cannot be easily broken, pipet the emulsion into a centrifuge tube and centrifuge for 5 minutes. This should break the emulsion.

(13) Sample is ready for GC analysis.

NOTE: Toluene extracts all of the analytes with the exception of RDX. In addition, it extracts all of the analytes with high recoveries in the spike, except 4-amino, 2-amino, and 3,5-DNA. Iso-amyl acetate extracts RDX and provides higher extraction recoveries of the above mentioned compounds.

7.3.1 Isoamyl Acetate Extraction:

Equipment and Consumables: The equipment and consumables are the same as the toluene extraction with the exception of using the double-distilled isoamyl acetate as the extraction solvent. Also, a new-unextracted aliquot of water is used.

Extraction Procedure-Perform procedure exactly like the toluene extraction, except extract, using isoamyl acetate, a new aliquot of sample.

7.4 Analyzing of Samples:

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7.4.1 After the sample preparation is finished for the sample set, choose the next set to be analyzed by either picking the oldest set or by picking the set with the highest priority.

NOTE: The toluene extracts are run on a different instrument than the isoamyl extracts. The toluene extracts are run on a GC equipped with two 30-m columns, while the isoamyl extracts are run on one equipped with two 15-m columns. This is due to the fact that RDX does not chromatograph well on longer columns and will only be seen on the shorter ones. However, if the analysts knows that there will be no RDX in the sample, both fractions can be run on the same instrument with the longer columns.

7.4.2 Next, write the analytical sequence in Turbochrom (See SOP on Turbochrom Operation) in the order given using sample weights, dilution factors and final volumes indicated on the paperwork. Two sequences need to be created: one for the toluene extracts and one for the isoamyl extracts.

Sample Analytical Run Sequence:

- 1 Wash
- 2 Third-Party Check
- 3 Calibration Standard #1 (1 pg/ul)
- 4 Calibration Standard #2 (5 pg/ul)
- 5 Calibration Standard #3 (7 pg/ul)
- 6 Calibration Standard #4 (10 pg/ul)
- 7 Calibration Standard #5 (20 pg/ul)
- 8 Calibration Standard #6 (50 pg/ul)
- 9 Calibration Standard #7 (100 pg/ul)

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- 10 Calibration Standard #8 (200 pg/ul)
- 11 Reagent Blank
- 12 Reagent Spike
- 13 Sample #1
- 14 Sample #2
- 15 Sample #3
- 16 Sample #4
- 17 Sample #5
- 18 Sample #6
- 19 Sample #7
- 20 Sample #8
- 21 CCV
- 22 Wash

7.4.3 Once the sequence(s) is created in Turbochrom, load a GC autosample tray with the corresponding sample and standard vials as given in the sequence. Check the volumes of the sample extracts. If evaporation has occurred, use the appropriate solvent to bring the volume up to the line marked on the vial.

7.4.4 Replace the septum and liner in the GC and check all gas flows as specified in the SOP on GC Maintenance and Operation. Start the GC running the analytical sequence as described in the SOP on Turbochrom Operation.

7.4.5 Once the Standards for the Nitroaromatic and Nitroamine analytes have been run, update the retention times in the sample table if needed (See Turbochrom Operation SOP). Check to see if the standard curve is acceptable as previously defined (Section 6.2). If the standard curve is not acceptable, look at the curve to see if one standard or a single injection has affected the curve. As a general rule, a bad calibration

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point is not automatically removed from the curve to achieve an acceptable R^2 . The analyst must first determine what caused the errant calibration point, such as a bad injection or a poorly sealed vial. If every indication shows that the GC-ECD is operating properly, the bad calibration point was caused by a one-time problem such as poor vial sealing and there are at least four remaining points, then the point may be dropped from the calibration curve. Document on the QC check sheet the removal of any standards from the curve. If multiple points are bad, it is not acceptable to drop more than one point. The emergence of more than one bad point most likely indicates a systemic error that necessitates more investigation. The samples will need to be rerun after the problem is found and remedied.

7.4.6 Examine the Reagent Blank chromatograms. If there is an indication of blank contamination, contact the supervisor (see Section 6.4). Record the surrogate recovery.

7.4.7 Examine the Reagent Spike chromatograms. Record the individual analyte and the surrogate recoveries.

7.4.8 Run the rest of the analytical sequence. Look at the CCVs. If they meet the acceptable limits given in Section 6.6, analyze the samples. If not, take the action described and process all sample data that do not require reinjection.

7.4.9 To process the sample data, first get the prep sheet for the selected sample and confirm that the sample weight and sample number matches the ASR form. Next, process the sample data taking the precautions outlined in Section 7.7, or justify each peak that is not a call using the acronym suggested in that section. Record the surrogate values from the sample on the Final Report Sheet (See Appendix B) for the sample along with all properly confirmed calls.

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7.4.10 Fill out the QC Check Sheet for the set (See Appendix A). Record as much information as possible on the QC check sheet for present and future reference.

7.4.11 File the hard copies of the Method, Sequence, CCVs, Samples, Reagent Blanks, Reagent Spikes and the QC Check Sheet in an expanding folder as described in Section 8.6. Put a copy of the sequence along with any notes on machine maintenance in the Instrument Logbook.

7.4.12 Have the packet checked by a peer analyst and have this analyst initial the appropriate boxes in the QC Check Sheet after review.

7.4.13 Fill out a spreadsheet (Appendix C) with all results from the samples in the set. Then create a custom letter to accompany the spreadsheet (Appendix D). Give both to the Organics secretary to be mailed to the district.

7.4.14 Fill out a D-M form for the samples (Appendix E) and submit to ADP, so that the samples are removed from the backlog.

7.4.15 Copy the electronic files from the PC hard-drive to the file server ("O" or "Organics Drive" to be saved to tape). Once confirmation is received that the files have been saved to mag. tape on two occasions, delete the file from the "O" Drive.

7.5 Instrumentation

7.5.1 Initial Start-up:

Not Applicable

7.5.2 Calibration and Performance Documentation:

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See Section 6

7.5.3 Example Output:

See Appendix F for example Chromatogram Report Sheets and example Chromatograms for both channels.

7.5.4 Shut-down:

Not Applicable

7.5.5 Maintenance, maintenance records, and responsibilities:

All maintenance performed on the instrument will be logged in detail in the Instrument Logbook.

For explanation of maintenance to be performed, see the SOP of GC Maintenance

7.5.6 Apparatus:

Hewlett Packard 5890 or Perkin Elmer Autosystem dual capillary column gas chromatograph with dual electron capture detectors (or equivalent). The two columns are connected via a "Y" splitter and a 5 meter, 0.53 mm uncoated guard column with a 0.32 mm ID.

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Toluene Extract Instrument

Column A: 30-m/0.25 mm ID, 95% dimethyl-5% diphenyl polysiloxane (Rtx-5) Restek, or equivalent.

Column B: 30-m/0.25 mm ID, 14% cyanopropyl-86% methyl polysiloxane (Rtx-1701) Restek, or equivalent.

Iso-Amyl Extract Instrument

Column A: 15-m/.25 mm ID, 100% dimethylpolysiloxane (Rtx-1) Restek, or equivalent.

Column B: 15-m/.25 mm ID, 95% dimethyl-5% diphenyl polysiloxane (Rtx-5) Restek, or equivalent.

Liners: A Perkin Elmer Cyclo-liner or HP Deactivated 4 mm liners, or equivalent.

7.5.7 Oven Temperature Program (See SOP on Turbochrom Operation to describe how to set the software to run the oven temperature program):

NOTE: All Rates are in Degrees Celsius per Minute, All Temperatures are in Degrees Celsius, All Times are in Minutes

Toluene Fraction Instrument

Initial Temperature: 60

Initial Hold Time: 0

Rate Temp Hold

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Ramp 1 20.0 140 0.0

Ramp 2 5.0 230 13.0

Ramp 3 10.0 250 0.0

Run time: 37 minutes

Iso-Amyl Fraction Instrument

Initial Temperature: 60

Initial Hold Time: 0

Rate Temp Hold

Ramp 1 5.0 140 5.0

Ramp 2 5.0 230 10.0

Run time: 49 minutes

7.6 Calculations:

7.6.1 Calculation of Sample Concentrations (See U.S.G.S. OFR Report,82-1004):

Response Factor:

$$RF = A/C \times V$$

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RF = Response Factor

A = Integrated peak area of identified component in the Calibration Standard, in area

C = Concentration of the Standard, in pg/ul

V = Volume of Standard injected, in ul

Sample Concentration (ug/l)

$$SC = A \times FV \times D / I \times W \times RF$$

SC = Sample Concentration

A = Integrated Peak Area of identified Sample Component

FV = Final volume of Sample Extract, in ml

I = Volume of Sample Extract Injected, in ul

W = Volume of sample in ml

D = Dilution Factor

7.7 Data Analysis (Evaluation):

7.7.1 Look at each fraction separately for every compound. By the end of processing, every compound must be identified on the chromatogram report sheet as a call by circling it, or have an acronym written next to it that explains why it is not a call.

Acronym :

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NT - Not a Target Compound

NC - Not Confirmed on Other Channel

BRL - Below Reporting Limit

WRT - Wrong Retention Time

WF - Wrong Fraction

RRL - Raise Reporting Limit (Specify new Reporting Limit)

Transfer all values from the chromatogram report sheets to the Final Report Form. If the amount is at or above the reporting limit and below the dilution threshold, report a call.

Also, correct baseline integrations as necessary using the technique given in the Turbochrom Operation SOP.

7.7.2 If the analyst makes a positive identification of a compound that is confirmed on both channels, but the compound is below the detection limit, they have the option of reporting a "Trace" on the spreadsheet that goes to this district. The Trace call is more of a side note for the benefit of the District who receives the hardcopy.

7.7.3 Dilutions: If a compound has a raw amount (concentration before the dilution factor is taken into account) of greater than the high standard it must be diluted and reshot. Estimate a dilution factor that will bring the sample's raw amount into the mid-range of the standard curve.

7.7.4 Coelutions: Some compounds will show irreproducible calibrations due to coelutions on one column, but not the other. In this case, make the call on the quantifiable channel and confirm on the coeluting one.

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7.8 Data Packet Organization:

All data is to be organized in an accordion file wallet that should be labeled with the date the GC first began acquiring data written in the left hand corner, "NA Waters" written in the center, and the "packet number" written in the right hand corner. The packet number includes the instrument identification letter followed by the current year and the sequential packet number and should correlate with the Turbochrom file number (See Turbochrom Operation SOP, part 6.7.1). An example of a packet number is S95001; where S is the instrument S, 95 is the year the file was acquired, and 001 is the first packet. In addition, a copy of the run sequence should be glued or taped to the front of this folder. This wallet should then be subdivided with five manila file folders labeled as given and containing the following:

Sequence and Method: This folder should contain a copy of the sequence, copies of all of the instrument methods as well as calibration reports for both channels (See Turbochrom Operation SOP for how to produce these reports in Turbochrom).

Retention Times: This folder should contain the Retention Time Summaries for both channels (See Turbochrom Operation SOP for how to produce these reports in Turbochrom).

Standards: This folder should contain the QC Check Sheet , the GC report sheets and GC chromatograms for each of the standards.

Blanks and Spikes: This folder should contain the final report sheets (Appendix B), GC reports sheets, GC chromatograms as well as the ASR sheets for the Blanks and Spikes.

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Samples: This folder should contain the final report sheets, GC report sheets, GC chromatograms as well as the ASR sheets for the Samples.

7.9 Data Review:

All data must be thoroughly reviewed by an experienced analyst before it is submitted to the District. The reviewing analyst must review the analyses of the samples to ensure no analyst oversights. The reviewer must check the samples for transcription errors that may exist on the final report sheet. Also, after thoroughly reviewing the samples, the reviewer must initial the sample's final report sheet. Finally, the reviewer must review and initial each point on the QC Check sheet after checking to ensure high quality data.

7.10 Reporting:

7.10.1 Reporting units

Micrograms per Liter (ug/L)-Used for Analyte Values

Percent recovery used for spike and surrogate recoveries

7.10.2 Reporting level - see MDL (Appendix G)

7.10.3 Significant figures - Follow U.S.G.S. NWQL protocol for reporting significant figures.

7.10.4 Deletion Reporting Codes

D-U Deleted - Due to Interference

D-R Deleted - Sample Ruined

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7.10.5 The final report sheet for the Samples, Reagent Blanks, and Reagent Spikes is filled out by the analyst, reviewed and signed by a qualified reviewer, and sent (hard copy) to the district.

8. Archiving:

Sample extracts are held for a minimum of 90 days after results are sent to district. Data packets with final report sheets are held indefinitely at this point in the warehouse. Electronic copies of the raw data is archived onto DAT tapes and kept on file with the Computer Services Unit.

9. References

9.1 U.S.G.S. SOPs Referenced:

SOP for NWQL Standard Operating Procedures, SOP Team, 1994, SOP #QX0001.0

SOP for the Analysis of Organochlorine Compounds in Sediment (Schedule 1325), Dawn Hrinko, 1994, no SOP number.

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SOP on Naming, Documenting and Verifying Standards, author unknown, no SOP number.

SOP on the Preservation of Water and Sediment Samples submitted for Organic Analysis, Jeff Stewart, 1994, SOP #OM0017.0.

SOP of Data Reporting and Worksheets, author unknown, no SOP number.

SOP of Organic Standards Preparation, Dennis Markovchick, 1994, no SOP number.

SOP of Turbochrom Operation, Leslie Merten , 1994, no SOP number.

SOP for GC Maintenance and Operation, Max Stroppel, 1994, no SOP number.

9.2 Additional References:

Standard Operating Procedure-Analysis of Nitroaromatics in Ground and Drinking Water, 1989, DOE and AEHA.

The Determination of Nitroaromatics and Nitramines in Ground and Drinking Water by Wide-Bore Capillary Gas Chromatography, U.S. Army Environmental Hygiene Agency, Organic Environmental Chemistry Division, Aberdeen Proving Ground, Maryland.

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St. Louis Laboratory Standard Operating Procedure-Extraction and Analysis of Nitroaromatic and Nitramine Explosives by GC/ECD, 1994.

10. Key Words:

Gas Chromatograph, GC Vials, Turbochrom