APPENDIX II

TIER I CHECKLIST

Tier I

This Plan Review Form is #	#	1	of	1	forms completed in the review of this closure plan	
Facility Name	Blue Knel	Industries		Valida	tor/DO	Anyone
ID Number	OHDXXX	123		Date	of Plan	Dec. 2, 2004
Date Review of Plan Completed	Dec. 2, 20	004		Plan i Revis	s: New, Amended, ed	NEW
Document Title:		Industries, e sampling				
Lab Name: GEL Laboratories	Media Typ Waste Wa Solid Was Oil (O):	ater (WW):		TCLP TCLP pH, F	ses Requested: VOC, TCLP Metals; VOC, TCLP Metals; ashpoint, PCBs, Total , Total RCRA Metals	Notes:

Note: The criteria used in the Tier I Data Validation checklist are derived primarily from SW-846 method requirements and U.S. EPA's National Functional Guidelines (NFGs) for Organic and Inorganic Data Review. Criteria from methods are considered preferable as they are specific to that procedure. Where the method is silent, criteria from the NFGs, or other sources when necessary, are adopted. For flashpoint (which uses ASTM methods dictated by the OAC rules), ASTM method criteria are used.

The Tier I data validation manual is the primary reference for this checklist. It explains and gives examples for the questions in this checklist. The Tier II methodology and terminology builds on that established in the Tier I checklist and its associated data validation manual. There is no Tier II manual, only the checklist and completed example checklists. Additional information is also available by referring to the specific methods.

Data Qualifiers and their meanings used throughout the Tier I Checklist			
J	J Estimated		
J+	Estimated High (results are likely reported higher than the true value)		
J-	Estimated Low (results are likely reported lower than the true value)		
R	Rejected		
UJ	Undetected Estimated		
NJ	Tentatively Identified, Quantitation Estimated		

Section 1.0 Report Completeness and Technical Holding Times Г

1.1 Sample Package Completeness and Deliverables				
This section provides a checklist of important component	Completeness ts of data reports. If the report is incomplete, it may be necessary to mation is provided. Please, refer to the Tier I data validation manual			
 1.1.1 Describe any discrepancies between the Chain Of Custody (COC) record and submitted sampling data. Action: If there are discrepancies, contact the laboratory for any missing deliverables and/or an explanation. 	No PCB data is present in the lab report. (Pgs. 44-53 are missing). The lab or the facility should be contacted and a new data report containing the required information should be submitted to the Agency. COC is found on pg. 4			
1.1.2 Is a signed statement from the laboratory present that attests to the validity of the data? Action: Take no further action and contact the facility and have the lab submit a valid data report If no response, qualify all data as unuseable.				
1.1.3 Is a case narrative present that summarizes QA/QC discrepancies and/or other problems' Action: No action is necessary, but this information is useful to focus data validation efforts.	Yes. The narrative is presented as part of the analytical results for each of the requested methods.			
1.1.4 Are COC forms present for all samples? Action: If not contact the facility for replacement of missing or illegible copies	Yes, a COC is present. Page 4.			
 1.1.5 Do the COC forms, sample receipt form, or the case narrative indicate any problems with the sample receipt, condition of samples, analytical problems or special circumstances affecting the quality of the data? Action: Use the information to focus data validation efforts. 	The sample receipt form indicates that the samples arrived in good condition. One except is the lack of results for PCBs. It is not known whether the omission of data is an oversight or if samples were just not analyzed. The data narratives do indicate a variety of problems that may require qualification of data.			
1.1.6 Are custody seals present and intact?Note: In some cases custody seals are not necessary. The data validator should review the project's SAP and DQOs to determine the necessity of custody seals.				

1.1	Sample Package Completeness and Delivera	ibles
req val to o	Is a sample receipt form present? If so, does it contain information on condition of sample containers, proper preservatives used (cross- check with COC) and temperature of the cooler? Note any comments or abnormal conditions: Action may be taken for the following special conditions: te: For waste samples, the temperature puirement is not always necessary. The data idator should review a project's SAP and DQOs determine the proper response.	The Sample receipt form indicated that all samples were in good condition upon arrival. The cooler temperature was 6°C. This is acceptable. The sample receipt form is found on page 5. There is no indication of preservation from the COC or sample receipt form, except for cooling the samples. This is acceptable. The type of preservation is relevant to judging technical holding time criteria.
A.	For samples analyzed for volatiles that were not properly cooled (temperature more than 4 <u>+</u> 2°C), all positive results should be qualified as "J-" and all non-detects qualified as "UJ."	
В.	For all liquid Volatile Organic Compound (VOC) samples or vials with air bubbles (>2 mm), positive results should be qualified as "J-" and non-detects as "UJ" or "R" depending on professional judgment (taking into account other quality control information such as sample cooler temperature and other site -specific data quality objectives).	
C.	If aqueous samples for VOCs were not preserved, check that technical holding times were met (see Technical Holding Times, Table 1). If not, qualify all associated sample results.	
D.	If liquid TCLP samples were preserved, qualify all associated results as rejected and flag the data with an "R."	

Data Validation PRF - Tier I Version 3.0, January 9, 2006

			TA	BLE 1 - Completer	ness and Technical	Holding Times				
Sample ID	Lab ID	Matrix	Sample Date	Date Received by the Lab	Parameter	Extraction Date	Preparation Date	Analysis Date	QA/QC Data Present ^A	Batch ID#
028	115782001	Waste Water	06-23-04	06-25-04	TCLP Metals	07-01-04 #346201	07-02-04 #346201	07-06-04	YES	346202
					TCLP Hg	07-01-04 #345991	07-06-04 #346228	07-06-04	Yes	346229
					TCLP VOCs	07-06-04 #345667	NA	07-16-04	Yes	347359
RO-02	115782002	Solid	06-24-04	06-25-04	TCLP Metals	07-01-04 #345991	07-02-04 #346201	07-06-04	Yes	346202
					TCLP Hg	07-01-04 #345991	07-06-04 #346228	07-06-04	Yes	346229
					TCLP VOCs	07-06-04 (missing)	NA	07-08-04	Yes	347359
002	115782011	Oil	06-24-04	06-25-04	pН	NA	NA	06-30-04	Yes	345466
					Flashpoint	NA	NA	07-21-04	Yes	350989
					PCBs	Not found	Not found	Not found	Not found	Not found
					Total VOCs		07-07-04 #347357	07-08-04	Yes	347358
					Total Metals	NA	07-07-04 #346534	07-06-04	Yes	346537
					Total Hg	NA	06-30-04 #344887	07-06-04	Yes	344889

A: Batch specific QA/QC requirements for Tier I data validation for Organic Data consists of Blank Data Matrix Spike/Matrix Spike Duplicate data, and surrogate data. For Inorganic Data the QA/QC data includes a matrix spike and it's duplicate and blank data. Additional QA/QC data may include ICP serial dilution results and postdigestion spike data.

Note: To fill out this table, list one sample ID# then list all analytical parameters on one line each with their associated analysis dates, batch ID#s, etc.(e.g., put mercury on a separate line from the other metals since it will have its own prep. dates, analysis dates, and batch ID#s).

1.2 Technical Holding Times

Table 1

Technical Holding Times for Volatile, Semi-Volatile, Metals and pH Samples

Technical holding time is the time, in days, from sample acquisition in the field to either laboratory preparation or analysis. Technical holding times are established from information contained in the laboratory report, chain of custody, and raw analytical bench sheets (if available). Technical holding times also depend upon whether samples were preserved. The recommended technical holding times for volatile compounds, semi-volatile compounds, metals, and TCLP analyses are listed below.

	Preserved *(see note below)	From field collection to extraction	From extraction to preparation	From extraction to analysis	Max holding times	Common preservative
VOCs (8260B) (aqueous)	Yes	NA	NA	14 days	14 days	Cool to 4 <u>+</u> 2° C, HCl
VOCs (8260B) (aqueous)	No	NA	NA	7 days	7 days	Cool to 4 <u>+</u> 2° C
VOCs (8260B) (liquid waste)	No	NA	NA	14 days	14 days	Cool to 4 <u>+</u> 2° C
VOCs (8260B) (solid/soil/waste)	No	NA	NA	NA	14 days	Cool to 4 <u>+</u> 2° C or no preservative
VOCs (EnCore) (5035/8260B) (solid/soil/waste)	Yes	2 days	NA	12 days	14 days	Encore Sampler
SVOC(8270C)	Yes	7 days	NA	40 days	47 days	Cool to 4 <u>+</u> 2° C
Total Metals (6010B/7000)	Yes	NA	NA	180 days	180 days	Nitric Acid (pH<2- aqueous); cool to 4° C - solid samples
Mercury (7470A)	Yes	NA	NA	28 days	28 days	Nitric Acid (pH<2- aqueous); cool to 4° C - solid samples
TCLP VOCs (1311/8260B)	No	14 days	NA	14 days	28 days	no preservative
TCLP SVOCs (1311/8270C)	No	14 days	7 days	40 days	61 days	no preservative
TCLP Metals (except mercury) (1311/6010B)	No	180 days	NA	180 days	360 days	no preservative
TCLP mercury (1311/7470A)	No	28 days	NA	28 days	56 days	no preservative
pH (9040B)*	No	24 hours (prof. judgement)	NA	NA	24 hours (prof. Judgement)	no preservative

* - Waste (and oil) samples may not need to meet same preservation standards/holding times as aqueous

samples.

1.2 Technical Holding Times

Technical Holding Times

Technical holding times are an important component of assuring that data is valid and not biased from inappropriate handling procedures. Technical holding times are judged by assessing the lapsed time from field sampling to extraction and to analyses. There are specific technical holding time requirements for specific classes of compounds. In addition, holding times may vary due to the presence or absence of preservatives. The validator should refer to specific criteria for holding times listed in Table 1 and in the Tier I Data Validation Manual. Use information on sampling, extraction and analysis dates (examined in section 1.0) to determine whether technical holding times are in compliance with criteria listed in Table 1. Complete the following table to determine if any violations of technical holding time exist, and qualify all associated sampling data.

1.2 Technical Holding Times - Volatile Organic Compounds

	Are samples properly preserved? Check preservation requirements, chain of custody, and sample receipt form for discrepancies.	List impropriety(-ies): Yes. TCLP samples should not be preserved and there is no indication of preservation. All samples were chilled to 6°C. This is acceptable. COC is found on pg. 4. The sample receipt form is found on pg.5
1.2.2	If samples were improperly preserved, or unpreserved, and the technical holding times were exceeded, qualify all positive results for affected samples as "J-" and all non-detected results as "UJ" or "R" based upon professional judgement.	List sample ID(s): NA
1.2.3	If technical holding times are greatly exceeded (> 2x the time requirement) upon analysis or re-analysis then the validator may use professional judgement to qualify all non-detected compounds as "J" or "R" based upon professional judgement and on DQOs.	List sample ID(s): NA

1.2 Te	1.2 Technical Holding Times - Semi-Volatile Organic Compounds				
1.2.4	If technical holding times are exceeded (Table 1), qualify all positive results for affected samples as "J-" and all non-detected results as "UJ."	List sample ID(s): N	IA		
1.2.5	If technical holding times are greatly exceeded (> 2x the time requirement), based on the project's DQOs, the validator may use professional judgement to qualify all non-detected compounds as "R" and all positive results as "J"	List sample ID(s): N	A		

1.2 Technical Holding Times - Inorganic Compounds				
preservation preservation	properly preserved (4°C for solids; acid for aqueous samples)? Check requirements, chain of custody, and ot form for discrepancies.	List impropriety(-ies): Yes. TCLP samples were not preserved. All other samples were chilled to 6°C. This is acceptable.		
Action: Note any ir qualify results.	npropriety, and use the information to			

1.2 Technical Holding Times - Inorganic Compounds				
1.2.7	If samples were improperly preserved or properly preserved and the technical holding times were exceeded (Table 1), qualify all positive results for affected samples as estimated ("J-") and all non- detected results as "UJ." or rejected ("R") depending on DQOs.	List sample ID(s): NA		
1.2.8	If technical holding times are greatly exceeded (> 2x the time requirement), the validator may use professional judgement and the project's DQOs to qualify all non-detected compounds as "R" and all positive results as "J-" or "R" depending on DQOs.	List sample ID(s): NA		

Section 2.0 VOC Data Validation

2.1 Blank Data Summary Review - Volatile Organic Compounds				
Blank Data Laboratory blanks are used to assess whether contamination from the laboratory, reagents, or other samples exists and whether this contamination can bias sample results. The qualification of sample results will depend upon the magnitude of blank contamination.				
 2.1.1 Is the method blank data present for each batch (method and matrix), including TCLP? Action: If not present, request information from the facility. If the required method blanks were not analyzed, sample results <u>may</u> be qualified as "J," for positive results and "UJ," for non-detected compounds. Qualification should take into account other QA/QC information. 	A TCLP blank for VOCs is present for samples 028 and RO-02 (batch 347359). Page 68. A blank for total VOCs is present for sample 002 (batch 347358). Page 71.			
2.1.2 Is there an indication that the samples associated with that blank were diluted? Note: The dilution factor can usually be found in the data report (a dilution factor of 1 indicates no dilution).	List the dilution factor(s): Yes. TCLP VOCs for 028 and RO-02 are diluted: DF for RO-02 = 100 page 62 DF for 028 and 002 = 50 pages 60 and 64- 65			
2.1.3 Do any method/field/trip/rinsate blanks have any positive results for any volatile target analytes? Was the same target compounds found in the samples? List those analytes and the results that are both found in the blanks and samples. These analytes are subject to qualification.	See next question for analytes			
Note: A list of samples associated with each of the contaminated blanks should be prepared. Field blank results should be used to qualify data. Trip blanks are used to qualify samples based on potential contamination during shipment, and are not required for non- aqueous matrices.				
Action: Follow the directions in the table below to qualify sample results due to blank contamination. Use the largest value from all of the associated blanks. If any blanks are grossly contaminated, all data associated may be qualified as "R", based upon professional and the project's DQOs.				

2.1 Blank Data Summary Review -	Volatile Organic Co	ompounds			
2.1.4 For those analytes identified , follow the directions in the Note: If analytes are detected i in the sample of interest, then is necessary. Use the inform to determine whether a dilution be used to determine qualifica dilution is applied to samples, concentration in the samples a dilution factor, then use the cr Table to qualify blanks and sam	Blank Batch Contaminar 347359 PCE Info found on pages 347358 1,2,4 TME Naphthal Styrene Toluene 2-butanc Info found on pages Based upon the dilu sample results for a be gualified as under	0.013 5 60 (028), 6 3 27.1 lene 31.4 13.5 46.0 one 788 5 64-65 (002 ltion factor a ill the constil	002) and 71-72 and the criter cuents listed	670 121 20.5 159 1390 (blank) ria in Table 3, above should	
For Common Volatile Contaminants: methylene chloride, acetone, 2- butanone, cyclohexane	For Other Contaminants:			Action:	
Sample Conc. > Detection Limit but < 10x Blank Result	-	> Detection Limit Blank Result	Identify	the sample undetecte	

Sample Conc. < Detection Limit	Sample Conc. < Detection Limit	Report the detection limit and qualify
& < 10x Blank Result	& < 5x Blank Result	result "UJ" estimated undetected
Sample Conc. > Detection Limit & > 10x Blank Result	Sample Conc. > Detection Limit & > 5x Blank Result	No qualification is necessary

2.2 Volatile Organic Data Review - Laboratory Control Sample (LCS)

Laboratory Control Sample

An LCS should be included with each batch of samples (approximately 20). The LCS consists of an aliquot of a clean (control) matrix similar to the matrix type of the sample and at the same weight or volume. The LCS is spiked with the same analytes at the same concentration as the matrix spike. When the results of the matrix spike indicate a potential problem due to the sample matrix itself, the LCS verifies that the laboratory can perform analyses in a clean matrix (Method 8260B).

2	QA the be	Was an LCS prepared, extracted, analyzed and reported once per group of 20 samples? This information should be included in the package provided by the lab. If not, contact laboratory and request that the information submitted to the agency. This information build be found in the injection log.	Yes. For sample 028 and RO-02 (batch 347359), the LCS is found on page 67. For sample 002, the results are found on pages 70-71.
	the LCS resi	ion: If LCS information cannot be found, consult facility for re-submittal of the data package. If S information is not present, qualify all positive ults as "J." If warranted, the data validator may act all results as unacceptable.	

2.2 Volatile Organic Data Review - Laboratory Control Sample (LCS)		
2.2.2 Does the LCS contain the following volatile target compounds in addition to the required surrogates:	Yes. The LCS in both batches contains the required compounds.	
1,1-Dichloroethene Toluene Trichloroethene Benzene Chlorobenzene		
Note: Method 8260B calls for the LCS to be spiked at the same level as the matrix spike. When the results of the matrix spike indicate a problem due to sample matrix, the LCS should be checked to determine whether the laboratory can perform the analysis on a clean matrix.		
2.2.3 Do the percent recoveries (%R) meet the QC limits provided by the lab?	List compounds and sample IDs that do not meet QC limits:	
Action: If the LCS recovery is greater than the upper acceptance limit, then positive sample results for the affected compound(s) should be qualified as "J." If the mass spectral criteria are met, but the LCS	For batch 347359 (samples 018 and RO-02), the LCS results are acceptable. Batch 347358 (sample 002): 3 LCS compounds were outside of the QC limits.	
recovery is less than the lower acceptance limit, then the associated detected target compounds should be qualified as "J." and the associated non-detected target compounds should be qualified as "R."	QC LImits Bromomethane %R = 31% 41-163 page 70 Chloroethane %R = 20% 51-145 page 70 N-Butyl Benzene %R = 118% 56-115 page 71	
If more than half of the compounds in the LCS are not within the recovery criteria, then all of the associated detected target compounds should be qualified as "J," and all associated non-detected compounds should be qualified as "R."	The results for bromomethane and chloroethane in the samples were non-detect. These results should be qualified as rejected "R". N-Butyl Benzene was detected in sample 002 at 158 ug/kg. This result should be qualified as 158J+ ug/kg.	
2.2.4 Verify the calculations for at least one %R. %R = found/true X100	Using the LCS result for 2-butanone for batch 347358 (sample 002), the %R was reported as 84%. Page 70-71.	
Action: If the %R is not calculated correctly, verify the other %R calculations and/or contact the lab for re-submittal. If the re-calculated %R values fall within the QC limits, the validator should use professional judgement to determine if the lab should be contacted for re-submittal or if the data should be flagged.	To verify, use the value listed under the NOM heading (LCS spike concentration) and the measured result for 2-butanone. %R = 4200/5000 X 100 = 84%	

2.3 Quality Assurance Summary Review - Matrix Spike/Matrix Spike Duplicates, VOC

Matrix Spike/Matrix Spike Duplicates

Matrix spike and matrix spike duplicates are performed to assess method precision for VOC and SVOC analyses. Matrix spikes and duplicates are required for every batch of samples (every 20 - 30 samples). The validator should be aware that the MS/MSD are batch specific, not sample specific. For example, the MS/MSD information may be any sample in the batch, but not necessarily a sample being validated. Because of this, matrix spike and matrix spike duplicate data alone usually aren't used to qualify results, but the information is used with other QA/QC data to qualify data.

	Is matrix spike/matrix spike duplicate recovery data present? tion: If any matrix spike data is missing, the laboratory build be contacted for a re-submittal.	In this lab report, the matrix spike, matrix spike duplicate is listed as prep-spike, prep-spike duplicate (i.e., PS and PSD) For batch 347359 (samples 028 and RO-02) the QC results are found on pages 67-68. For batch 347358 (sample 002) the QC results are found on pages 73-76.
2.3.2	How many VOC spike recoveries are outside the QC limits?	Record the spike recovery and control limits: For those constituents that are outside of the control limits: Yes, for batch 347358 (sample 002) 4 compounds are out of criteria in the matrix spike and 2 of the same compounds are outside of QC limits in the matrix spike duplicate. PS %R PSD%R Limits 2-Butanone 48 55-149 acetone 24 44-181 bromomethane 29 30 44-163 chloroethane 19 18 51-145 hexachlorobutadiene 52 56-121 Batch 347359 displayed no problems with matrix spike/matrix spike duplicates
wit	How many RPDs for matrix spike and matrix spike duplicate recoveries are outside the QC limits for VOCs? te: The MS/MSD results may be used in conjunction h other QC criteria to determine the need for data alification. Outliers should be identified.	Record the recovery data out of criteria and control limits. Review surrogate and LCS data to determine if qualifiers are necessary: Yes, for batch 347358 (sample 002), 2 compounds had RPDs that exceeded the control limits. Page 75. RPD Limits 2-butanone 40 0-30 acetone 99 0-30 The results for batch 347359 (samples 028, RO-02) were within control limits. Page 67.

2.4 VC	OC Surrogate Recovery			
recove used w	VOC Surrog pate compounds are spiked compounds of ary of surrogate compounds allows an ass with other QA/QC data to qualify sample rea in the data validation guidance document.	known comp essment of ma	atrix interference. VOC	surrogate recoveries are
Su	rrogate Compound	Water *	Soil/Sedir	ment_*
	Bromofluorobenzene	86-115	74-121	
	promofluoromethane	86-118	80-120	
	luene-d ₈	88-110	81-117	
1,2	P-Dichloroethane-d₄	80-120	80-120	
Pe Flu	 P-Dichlorobenzene-d₄ ntafluorobenzene 46 Method 8260B, Table 8. Acceptance cr Are the surrogate recovery data present for each batch (method and matrix), including TCLP? 	Yes, ea		contains information on age 62)
sai	te: Samples may be included in separate mple batches and separate surrogate coveries should be provided.			
	tion: If no, contact the laboratory for explanati d re-submittals.	on		
2.4.2	Were any outliers correctly (based upon the laboratory's criteria)?	E List the	sample ID(s), matrix(-	ces) and parameter(s):
	tion: Mark, circle or highlight the suspected tliers.	Batch 34	17359 page 62 Bromofluorobenzene	%R Limits 118 95-108

2.4 VOC Surrogate Recovery

2.4 V	2.4 VOC Surrogate Recovery			
2.4.3	If any surrogate compound was out of compliance, was re-analysis performed to confirm a matrix interference?	List sample ID(s) for surrogate compounds out of compliance and criteria:		
in qu da	ote: Check the report narrative for an dication of re-analysis. Additionally, lalification may not be appropriate for TCLP ta. Best professional judgement may be used qualify data.	There is no evidence of re-analysis for sample RO-02. The results for this sampe are subject to qualification. (Page 62) In this sample, two surrogates were above the upper control limit. Therefore, all positive results should be qualified as estimated and flagged with a "J+."		
lin Re	tion: If a surrogate is above the upper control nit, all positive results should be qualified as "J+". esults listed as non-detected should not be valified.	Non-detected results should not be qualified.		
cri all	any surrogate recovery is less than the lower teria, but greater than or equal to 10% recovery, detected compounds should be qualified as "J-" d all non-detected compounds as "UJ."			
de ba	any surrogate recovery is less than 10%, all tected compounds should be qualified as "J-" or sed upon best professional judgement and all n-detected compounds as "R."			

Section 4.0

Metals Data Validation

4.0 Metals Analysis Data Validation			
4.1 Blank Data Summary Review - Metals Data Blank Data Laboratory blanks are used to assess whether contamination from the laboratory, reagents, or other samples exists and whether this contamination can bias sample results. The qualification of sample results will depend upon the magnitude of blank contamination.			
4.1.2 Were any samples diluted? Action: Record the dilution factor(s)	No. Batch 346202 (samples 028 and RO-02) Page 30 & 32 Batch 346537 (sample 002) Page 34		
 4.1.3 If metals are detected in the blank, check the sample results and record all analytes and the results detected in both the blank and sample. Note: Use the information from 4.1.2 to determine whether a dilution factor should be used to determine qualification. When a dilution factor is applied to samples, the contaminant concentration in the samples are divided by the dilution factor, then the criteria discussed below is used to qualify sample results. 	Batch 346202 (samples 028 and RO-02): Page 36 Analyte Blank (mg/L) Spl. Result (mg/L) Barium 0.0233 0.28 0.169 RO-02 2.65 2.65 Batch 346537 (sample 002) Page 37 Analyte Blank (mg/L) Spl. Result (mg/L) Arsenic 1.90 002 ND No qualification is necessary (i.e. the results are greater than 5X the blank results).		
Action: Positive sample results that are greater than the detection limit but less than 5 X the blank results (after dilution is accounted for) should be qualified as estimated and flagged with a "U." Sample results greater that 5X the blank results (after accounting for dilution) should not be qualified.			

Blanks- Mercury			
 4.1.4 Were a method/preparation blanks included with each batch. Action: Consult the lab and if possible have the data submitted. If the data is not available, the data validator may apply best professional judgement to qualify the sample results. 	Yes. Batch 346229 (Hg. samples 028 and RO-02) Page 38 Batch 346537 (Hg sample 002) Page 38		
4.1.5 Were any samples diluted? Action: Record the dilution factor(s)	No. Page 34 Batch 346537 (sample 002) Page 34 Batch 346537 (Hg sample 002) Page 34		

4.1.6	Did the method blank contain mercury above detectable levels? Was mercury also detected in the sample results? If so, these results are subject to qualification.	No detections of mercury were found in either blank.
	te: If mercury is discovered in the method blank ove the detection limit, the lowest concentration of	
any sample in that batch must be <u>10 times</u> the method		
blank concentration (after dilution is accounted for). If this is not the case, all samples in that batch should		
hav	ve been re-digested and re-analyzed.	
	ion: Review the blank data. If the sample results are	
	itive but less than 10 times the concentration in the nk, the results should be qualified as "U".	

4.2 Metal Spike Recovery

Metal Spike Recovery

Spikes are elements of known composition that are added to blanks and to samples that measure accuracy and precision of the analyses. At least one spike (termed a matrix spike or preparation spike) should be included for each batch of samples. Spike recovery criteria listed in this section are determined from U.S. EPA's National Functional Guidelines for Inorganic Data Review. The criteria applied by an individual laboratory may vary. The laboratory should be consulted and it's QA/QC criteria supplied to the data validator. An Laboratory Control Sample (LCS) or preparation spike is a method blank (e.g. a blank consisting of reagents used to prepare a sample) that are spiked with known concentrations of target analytes. The LCS is a batch specific quality control sample that is used to verify the analytical system can accurately measure target analytes using an ideal sample.

4.2.1	1 Confirm that at least one pre-digestion spiked sample was analyzed per batch, matrix type	Yes.	
	sample delivery group?	Hg data for both batches are found on page 38.	
Action: If not present, contact the facility for re- submittal.		Metals by ICP	
			page 36 Page 37
4.2.	2 Are all spike recoveries (except Hg and Ag) within control limits (e.g., 75% to 125%)?	List those elements out of control:	
		Yes. All spike recoveries are within control limits.	
Note: When the spike sample result is less than the instrument detection limit, the percent recovery calculation should use a value of zero (not the detection limit) for the sample result.			
	Action: Is the sample concentration \geq 4 times the spiked concentration? If yes, disregard spike recoveries for analytes whose concentrations in samples are > 4 times the spike added. If no, circle those analytes whose concentration is < 4 times the spike added.		

4.2 Metal Spike Recovery	
4.2.3 Based on the results of 4.2.2, if the sample results were <4x the spike amount and spike recoveries were out of criteria, a post-digestion spike should be analyzed.	NA
Note: Post-digestion spikes are not required for Ag or Hg; however, one typically is run if the LCS was out of control. The post digestion spike confirms a matrix interference and should not be used for qualification	
Action: Contact the facility/laboratory for an explanation if a post-digestion spike was not analyzed. If a satisfactory explanation is available, use professional judgement to qualify sample results.	
 4.2.4 Are any aqueous spike recoveries: 1. Less than 30%? 2. Between 30% and 74%? 3. Between 126% and 150%? 4. Greater than 150%? 	
Note: The TCLP extract should be handled as an aqueous sample.	No
Action: If < 30%, and the sample results are below the detection limit, all data should be qualified as "R."	
If between 30% and 74%, qualify all positive data as "J-" and non-detected data as "UJ."	
If between 126% and 150%, qualify positive as "J+." All undetected compounds are acceptable.	
If > 150% note for possible positive bias. The data validator may qualify data as rejected "R" based on professional judgement and the eventual end use of the data.	
 4.2.5 Are any soil/solid/waste spike recoveries (pre and post digestion): Less than 10%? Between 10% and 74%? Between 126% and 200%? Greater than 200%? 	All percent recoveries are within control limits.
Action: If < than 10%, those elements out of control limits should be qualified as "R."	

If between 10% and 74%, qualify those elements out of control limits as J-".

If between 126% and 200%, qualify positive data, for those elements out of control limits, as "J+".

If > than 200%, qualify all positive data, for those elements out of control limits, as "R."

4.2 Metal Spike Recovery		
post-dige to confiri	If the pre-digestion spike was outside the QC limits for Atomic Adsorption furnace analysis (e.g., SW-846 methods in the 7000 series), was a post-digestion spike performed? Samples should not be qualified based on estion spike results. The results are used m a matrix interference. If a post-digestion as not prepared, the data validator may e data.	NA
lf ·	Based on the results from 4.2.6, were the post-digestion spike recoveries within the quality control range (75% to 125%)? f > 125%, qualify all positive data as "J+". < 75%, qualify both positive and non- ata as estimated and falg this data with	NA

4.3 Quality Assurance Data Review - Inorganic Analysis - AA Analysis

Graphite Furnace Atomic Adsorption QC Atomic Adsorption analyses require specialized QA/QC procedures that may be different than Inductively Coupled Plasma (ICP) Emission Analysis. Commonly, AA analysis is performed for mercury and selenium. Mercury analysis data validation is specifically detailed in the Inorganics Section of the Tier II Checklist. The Tier I Data Validator is directed to the Agency's Data Validation Review Manual and to specific methods detailed in SW-846. In general, external calibration procedures are commonly required by the method. In addition, duplicate injections and multiple concentration post-digestion spikes are required to establish precision and accuracy data. 4.3.1 Were duplicate injection of samples List sample IDs and appropriate method and calculated RPD: performed and if so, were the duplicates within + 20% RPD for samples with NA concentrations above the detection limit? Note: Results are reported based upon the average of duplicate injections. If the acceptance criteria is not met, the sample should have been re-analyzed (i.e., with at least two additional injections). Action: If RSD criteria is not met or the sample was not rerun, qualify all positive data as "J." 4.3.2 If the samples were re-analyzed (i.e., 2 more No. __ injections), do the duplicate injections agree within 20% RSD? . List sample IDs and appropriate method and Yes. calculated RSD. Action: If the RSD criteria is not met, qualify all positive results as "J." 4.3.3 Were Matrix Spike/Matrix Spike Duplicates Yes analyzed at a rate of 1 in 20 or per batch? Action: If no MS/MSD were analyzed, qualify all positive results as "J" and all undetected results as "UJ."

Page 20

Section 5.0 Characteristic Tests 5.1 TCLP Preparation and TCLP Spike Recovery

Toxicity Characteristic Leaching Procedure The toxicity characteristic leaching procedure (TCLP) is used to determine whether wastes exhibit the toxicity characteristic or whether Land Disposal Restrictions have been met. The TCLP test is specified in OAC 3745-51- 24 and defined in SW-846, Method 1311. TCLP data validation requires specific data concerning extraction preparation in addition to the usual data submitted for organic and inorganic analytical methods. In most cases, a laboratory will have to supply bench sheet data to complete data validation. The validator should consult the Tier I Data Validation Guidance Manual for specific information and examples.		
 5.1.1 Did the laboratory calculate TCLP filterable solids? Based on the percent solid calculations, were the correct analytical procedures followed? Note: TCLP requires that solid waste, semi-solid waste and liquid wastes be prepared based upon the amount of solids in the waste. For waste that has greater than 99.5% solids, the waste is considered solid and a 100 grams of material is extracted with 20 times this weight of extraction fluid. For waste that is equal to or less than 0.5% solids, the waste is considered a liquid and the liquid itself is considered the extract (no additional extraction fluid or tumbling is necessary). If the waste contains both solids and liquids, the solid portion, trapped by filtering, is extracted with 20 times it's weight of extraction fluid and then analyzed. In addition, an aliquot of the liquid is analyzed. The results are then mathematically combined. Alternately, the multiphase components may be physically recombined prior to analysis. Action: If percent solids were not calculated contact the facility for the proper information. If, based on the percent solids calculations, the appropriate preparation methods were not used, qualify analytical results using the following criteria: All positive results above the detection limits but below the regulatory level should be qualified based on professional judgement and the specific circumstances. 	Percent liquids are listed on Page 41. For sample 028 had greater than 0.5 % solids, but the laboratory treated the samples as a liquid. This is incorrect and the data is subject to qualification. Sample 028: A positive result for Cd was recorded at 0.787 mg/L. This result is just below the regulatory limit of 1.0 mg/L. Since metal extraction may be significant in the solids portion of the waste, the results for Cd should be qualified as rejected and flagged with an "R." Barium was also detected in sample 028 (page 30); however, its results are significantly less than the regulatory limit. Based upon professional judgement, the barium result will not be qualified.	
 5.1.2 If any sample(s) contained a liquid portion and a solids portion requiring extraction (as described above), did the laboratory properly recombine the fractions on a volume basis (either physically prior to analysis, or mathematically after analysis)? 	For sample 028, the solids portion was not separated and extracted. Page 41	
Action: If information is not present or results were not properly recombined, contact the laboratory for resubmittal of documented proper procedures.		

5.1 TCLP Preparation and TCLP Spike Recovery	
5.1.2 Was the proper amount of material extracted?	List sample IDs and sample mass(es) used for the extraction:
Note: For waste samples to be analyzed for metals or SVOCs (in the solid portion), a minimum of 100 grams is required. For waste samples to be analyzed for volatile compounds, approximately 20-25 grams of sample is required.	Page 41.
	Batch 346202 (samples 028 and RO-02) 100 g+ of material was extracted for metals analysis. This is acceptable.
Note: Liquid samples are directly analyzed as the TCLP extract, no extraction fluid is added to the sample.	Batch 347359 (sample 028 and RO-02) no information is presented on the volume of material used for VOC extraction.
Action: If improper sample mass is used, qualify analytical results using the following criteria:	The lab should be contacted and the required
All positive results above the regulatory level should not be qualified.	information for TCLP VOCs presented to the Agency. If this information is not available, the data validator should use professional judgement in qualifying
All positive results above the detection limits, but below the regulatory level should initially be qualified as "J" estimated. Based on professional judgement, qualification of data as "R" may be warranted.	sample results. The data validator may wish to reject results until information is presented from the laboratory.
Based on professional judgement, all non-detected results should be qualified as "J" estimated or "R."	
5.1.3 Was the correct extraction fluid used?	List sample IDs and fluid type(s) used for the extraction:
Notes: Fluid # 1 is <u>always</u> used for VOC analysis.	METALS: Page 41 For Sample RO-02, the only solid sample, the initial
Fluid #1 should be used if the final pH of the pre-test sample is below 5.0.	pH was determined to be 8. After acidification, the final pH was 3.567. This indicates that a TCLP fluid #1 is correct. The lab used TCLP fluid #1 for
If the pH is above 5.0, hydrochloric acid should be added to the pre-test sample (refer to the method for specifics).	extraction. This is correct. However, the pH of the buffer solution (pH 5.09) was outside of the methods requirements.
Re-analyze for pH.	For sample 028, no extraction fluid addition was
Fluid #1 should be used if the final pH of the pre-test sample is below 5.0.	necessary because this sample was treated as a liquid.
Fluid #2 should be used if the final pH of the pre-test is above 5.0.	VOCs: For VOCs, no information is presented. TCLP fluid #7
Action: Consult with the facility and have the extraction fluid information submitted. If the improper fluid is used, qualify analytical results using the following criteria:	should always be used for VOC extraction. This information should be requested from the lab. If it is not available, the data validator should use best professional judgement to qualify sample results.
All positive results above the regulatory level should not be qualified.	
All positive results above the detection limits, but below the regulatory level, should initially be qualified as "J." Rejection of data may be warranted if other preparatory procedures are outside of criteria.	
All non-detected results should be qualified as "R."	

5.1 TCLP Preparation and TCLP Spike Recovery

5.1.4 Did the extraction fluid have the proper pH?	List incorrect fluid pH(s):
Fluid #1 has a pH range of 4.88 to 4.98 Fluid #2 has a pH range of 2.83 to 2.93.	No.
Action: If an improperly prepared extraction fluid is used, qualify analytical results using the following criteria:	For sample 028, the pH of the buffer solution was reported as 5.09. This is outside of the acceptance range for TCLP fluid #1. Page 41.
All positive results above the regulatory level should not be qualified. All positive results above the detection limits, but below the regulatory level, should initially be qualified as "J." Rejection of data may be warranted if other preparatory procedures are outside of criteria. All non-detected results should be qualified as "R."	All positive results below the regulatory limit should initial qualified as estimated "J-". All data below the detection limit should be rejected and results flagged with an "R." Upon review, this qualification may be changed and all positive results can be rejected.
 5.1.5 Was the correct weight of extraction fluid used? Laboratory bench sheets may be needed to complete this section. Action: If the extraction fluid weight is not more than ± 15% of the correct value (2000 grams for metals; 500 grams for VOCs), qualify all results as estimated "J" or "UJ". These values may be re-qualified if additional problems with TCLP preparation exist. If the extraction fluid weight is less than 70% of the proper weight, qualify all results as "R." If the extraction fluid weight is more than 30% greater than the proper weight, qualify all non-detect compounds and positive results below the regulatory level, as "R." All positive results above the regulatory limit will not be qualified. 	Yes. Greater than 2000g was used for TCLP metals (i.e. 20 X 100+ grams) Page 41. There is no information on VOCs.
5.1.6 Was a TCLP blank analyzed with every batch of samples?	List IDs of affected samples: Yes.
Note: TCLP blanks should be prepared using the same extraction fluid as is used for the associated sample's extraction. Action: Contact the facility for submittal of missing data. If no blank was analyzed, qualify all positive results as "R." If	TCLP VOCs blank Page 68 TCLP blank metals Page 37-38 TCLP blank Hg Page 38
data is available, qualify TCLP data as designated in Section 4.0 Blank Data Summary Review	
5.1.7 Was the tumbling time within 18 +/- 2 hours?	
Note: Tumbling time (evaluated based on the day and time tumbling begins/is completed) should be noted on the bench sheets. The laboratory should be contacted if this information isn't present.	16 hours Page 41
Action: If the tumbling time is not within 18 +/- 2 hours, qualify all data as "J."	

5.1 TCLP Preparation and TCLP Spike Recovery		
5.1.8 Was the tumbler speed within 30 +/-2 RPM?	Tumbling speed was 30 RPM Page 41	
Note: Tumbler speed should be noted on the bench sheets. The laboratory should be contacted if this information isn't present.		
Action: If the tumbler speed is not within 30 +/-2 RPM, qualify all data as "J."		
5.1.9 Was the room temperature during the extraction $23^{\circ}C$ + $2^{\circ}C$?	Yes.	
Note: Data would not be rejected using this criterion except in extreme cases (e.g., very cold temperature	Starting temperature was 23°C; ending temperature was 23°C.	
with detectable TCLP compounds).	Page 41	
Action: "J" qualify the data for extractions outside this range or when temperature was not recorded.		

VOC, SVOC and Metal results from the TCLP test should meet the sample QA/QC criteria outlined in Sections 1.0 through 4.0.

5.2 Ignitability

Ignitability

The two testing methods that may be used to determine this hazardous waste characteristic are SW-846 Method 1010 (Pensky-Martens Closed Cup, also ASTM D93-90 or D93-77) and Method 1020 (SETAFlash Closed Cup and ASTM D3828-93). Method 1020A is used for liquids that have lower viscosities and flashpoints between 0 °C and 110 °C.

Method 1010 is the flashpoint method most often used by DHWM. It is used for "fuel oils, lube oils, suspensions of solids, liquids that tend to form a surface film under test conditions, and other liquids." For us, this includes things like paint wastes, parts cleaners, etc. To test the flash point, "the sample is heated at a slow, constant rate with continual stirring. A small flame is directed into the cup at regular intervals with simultaneous interruption of stirring. The flash point is the lowest temperature at which application of the test flame causes the vapor above the sample to ignite." Method 1010 has two options, termed "A" and "B". Method A, the basic procedure, is used unless the material being tested is a suspension of solids or a highly viscous material. Those materials require the use of Method B. There are specific requirements and apparatus for method 1010 that are not included in this check list. These items include recording of barometric pressure, thermometers, stirrer rates, wind shields and drying of wastes that contain free water. If necessary, specific testing requirements that are used should be discussed with the laboratory and appropriate qualifications of the data should be made.

5.2 Pensky-Martens (SW-846, Method 1010) Method A		
5.2.1 Was p-xylene used to calibrate the instrument?	Its not known. p-Xylene is not listed. The LCS information is for 77°F. This is sometimes listed in MSDS sheets for p-xylene.	
5.2.2 Was the flashpoint for the calibration standard p-xylene within 81 <u>+</u> 6 °F?	Record the p-xylene calibration flashpoint(s): The chemical used to calibrate the instrument had a flashpoint of 77°F. If p-xylene, the flashpoint is within the criteria.	
 5.2.3 If the calibration standard was outside of this range (see 5.2.2), was corrective action taken? Action: If no corrective measures were performed, determine whether a significant bias has been imparted to the samples and qualify the results using professional judgement. If sample is still available, notify the laboratory. Consult Tier II evaluator regarding request re-analysis. 	Record IDs of samples that are qualified: NA	
 5.2.4 Based on 5.2.3, if corrective measures were taken, was the p-xylene calibration flashpoint within 81± 2 °F? Note: Corrective measures should have continued until this flashpoint calibration range was attained. Action: If these procedures were not followed and documented, contact the laboratory for an explanation. Lack of an adequate explanation may justify qualifying the data. 	NA	

5.2 Pensky-Martens (SW-846, Method 1010) Method A	
 5.2.5 Was method 1010 - Procedure A or Procedure B used? Note: Method 1010A is for non-viscous samples and 1010B is for viscous samples. It may be difficult to discern which procedure is being used by a lab. Method 1010B uses a stirring device and if RPM measurements are listed it can be assumed that this version of the test is being used. If in doubt, the data validator should contact the lab for an explanation. Action: Proceed to section 5.2 if 1010A is used or section 5.3 if 1010B is used. 	1010 Procedure A We assume that this is the correct method because no information on barometric pressure corrections are included in the data report. In addition, the temperature rise is consistent with 1010 - Procedure A. However, the laboratory should be contacted and the assumption verified. Pages 11 and 16.
 5.2.5 If a sample has an expected flashpoint, based on field/facility information, measurements should begin at least 30-50° F below the expected flashpoint of the material. If the expected flashpoint is unknown, the initial measurements should begin at the ambient temperature of the laboratory. Action: If these procedures were not followed and documented, contact the laboratory for an explanation. Lack of an adequate explanation may justify qualifying the data. 	The result for the sample was 138°F. Assuming a room temperature of 75°F, the laboratory was in compliance. Page 14 or 16.
 5.2.6 Was heat applied so as to raise the temperature of the sample at a rate of 9-11 °F per minute? Note: To determine if this temperature rate is proper, the time and temperature at the beginning of heating should be recorded, and the time and temperature when flash occurred (or when analyses ended) should be recorded. Action: If these procedures were not followed and documented, contact the laboratory for an explanation. Lack of an adequate explanation may justify qualifying the data. 	There is insufficient information. The laboratory should be contacted. If no information is available, the review may qualify data based upon best professional judgement and the project's DQOs.
 5.2.7 Were duplicate analyses performed? Action: If no duplicates exist, the data validator may qualify all results as "J." If additional QA problems exist, the validator may justify rejecting the results if the flashpoint determination is over 140°F. Results should not be rejected for data under 140°F. If duplicate results exist, and the flashpoint range is under 220°F, the results should agree within ± 4°F. If the results do not agree then qualify all results as "J." If additional QA problems exist, the validator may justify rejecting the results if the flashpoint determination is over 140°F. 	Yes, duplicates were performed. Page 16

5.4 pH Determination and Corrosivity Tests	
	pH groundwater monitoring and for determining if a waste rrosivity determinations, OAC 3745-51-22 specifies that test.
5.4.1 Were the pH tests performed as soon as practically possible?	
Note: SW-846 Method 9040B does not specify a maximum technical holding time for pH. However, it does state that all tests must be performed as soon as possible. The Ohio EPA expects that most laboratories can perform the pH test within 24 hours of sample receipt.	
Action: If analyses were performed within 24 hours, no action is necessary. If analyses were performed after 24 hours, but before the end of 7 days after sample receipt, all sample results between a pH of 2.05 and 12.5 will be flagged as "J." If the results are equal to or less than a pH of 2 or greater than or equal to a pH of 12.5, the results will not be flagged.	
If analyses were performed 7 days or more after sample receipt, all sample results between a pH of 2.05 and 12.45 will be flagged as "R." If the results are equal to or less than a pH of 2 or greater than or equal to a pH of 12.5, the results will not be flagged.	YES
5.4.2 Was a yearly NIST certification of the analytical instrument performed?	
Note: This information must be part of the Laboratory's QAPP. Check the QAPP or request information for the facility or laboratory.	
Action: If a yearly certification was not performed, flag all results between a pH of 2.05 and 12.5 as "J." All results meeting the regulatory criteria for corrosivity will not be flagged.	No information was provided.Data should be qualified as estimated
5.4.3 Were the calibration buffers within their expiration date?	
Note: Have the laboratory provide a photocopy of the expiration date, and the buffer batch ID?	
Action: If the expiration date is exceeded, flag all results between pH 2.05 or 12.45 as "R." Initially, results meeting the regulatory criteria for corrosivity will not be flagged; however, the data validator may qualify results based upon professional judgement and the data quality objectives for the data.	No data was provided . Data should be rejected

5.4.4 Was the instrument calibrated correctly using at least two buffers that bracket the expected pH of the sample?	No. Only 1 buffer was used. Page 10 shows the calibration log. Page 8 shows the sample result.
Note: For corrosivity determinations, the calibration buffers must include a pH 2 buffer and a pH 12 buffer. Review the calibration log for information or request information from the laboratory.	The pH of the sample 2.66. This is close to the regulatory limit. An additional pH buffer (pH 2.0) should have been used. Since the value is close to the regulatory limit, the results should be rejected and the data flagged with an "R".
Action: If an insufficient number of buffers were used (i.e., one) or if incorrect buffers were used (buffers did not include a pH of 2 or 12 for corrosivity determinations), flag all results between a pH of 2.05 and 12.45 as estimated, "J." All results meeting the regulatory criteria for corrosivity will not be flagged. If the pH of the waste is within 1.5 pH units of the regulatory criteria for corrosivity (3.0 or 11.0) and a pH 2 or 12 buffer was not used, the results may be questionable and additional analyses using the correct buffers standards may be necessary.	
5.4.5 Was continuing calibration performed?	
Note: If continuing calibration was performed, the pH of the continuing calibration buffer must be within 0.5 pH units of the buffer pH. Information on the continuing calibration standard and results must be requested from the laboratory.	Yes. See page 10. The data is within the 0.5 pH units of the continuing calibration buffer.
Action: If continuing calibration was performed and the results were within 0.5 pH of the calibration buffer, no action is necessary. If continuing calibration was performed, and the results were greater or less than 0.5 pH units of the correct reading for the calibration buffer, then the analysis must have been terminated and the instrument recalibrated. If recalibration was necessary, but not performed, flag all results between a pH of 2.05 and 12.5 as estimated, "J." Initially, results meeting the regulatory criteria for corrosivity will not be flagged; however, the data validator may qualify results based upon professional judgement and the data quality objectives for the data.	
 5.4.6 Were the temperatures of the sample and the calibration buffers within 2°C of each other? Note: request the information from the laboratory. If the sample and the calibration buffers were not within 2°C, then temperature compensation must have been performed. Request information from the laboratory on manual temperature compensation procedures compensation procedu	A run log is found on page 10 which continuing calibration verification standard s and temperature. However, there is no information on the initial calibration of the instrument or whether a device using a an ATC was used. In addition, there is no information on the sample temperature at the time of analysis. The lab should be contacted for clarification. The data should be qualified as estimated and the data flagged with a "J." Based upon the review of other QC data associated with pH analysis, the data validator may wish to
or whether an automatic temperature compensation was used.	reject sample results.
Action: If temperature compensation was required but not performed, flag all results between pH 2.05 or 12.45 as estimated, "J." Initially, results meeting the regulatory criteria for corrosivity will not be flagged; however, the data validator may qualify results based upon professional judgement and the data quality objectives for the data.	

5.4.7 If the sample pH was above 12.0, was the temperature of the sample maintained at 25 $\pm 1^{\circ}$ C?	NA
Action: If the temperature was maintained at $25 \pm 1^{\circ}$ C, then no action is necessary. If the temperature was not maintained at $25 \pm 1^{\circ}$ C, but the results meet the regulatory criteria of corrosivity, then the results will not be flagged. If the temperature was not maintained, then reject, "R," all results between 12.0 and 12.5.	