A66 - CCME REFERENCE METHOD FOR THE CANADA-WIDE STANDARD FOR PETROLEUM HYDROCARBONS (PHC) IN SOIL - TIER 1 METHOD

Revision 1.12

May 6, 2020

(Note: Checklist incorporates requirements from Dec 2000 version + Addendum 1)

(CCME CWS PHC and PERCENT MOISTURE)

Laboratory Name: _____

Appendix Name: _____

Appendix Number: _____

Assessor: _____

Date: _____

* NOTE: for assessment of Petroleum Hydrocarbons (PHC) and Percent Moisture in soil by CCME only; for analysis of PHC in soil by other reference methods or PHC in water, please use A03-Appendix to the CALA Rating Guide.

CALA
Trust, measured accurately

TEST SPECIFIC CHECKLIST FOR CCME PETROLEUM HYDROCARBONS

Clause	Requirement	Y N N/A Comments	Y N N/A Comments	Y N N/A Comments
17025: 2017		**(F1) C6 – C10 Hydrocarbons**	**(F2-F4) C10 – C50 Hydrocarbons**	**(F4G) Gravimetric Heavy Hydrocarbons and % Moisture**
	TEST METHOD CURRENCY			
7.2.1.2	The current authorized test method and necessary supporting work instructions are available to the analyst.	❑❑❑	❑❑❑	❑❑❑
	TEST METHOD VERIFICATION/VALIDATION			
7.2.1.5	Data exists for the method as implemented in the lab under assessment against the CCME PHC benchmark method, as per Appendix 2 and Addendum 1.	benchmark CCME PHC method is purge and trap GC/FID	benchmark CCME PHC method is 16-24 hour Soxhlet extraction, rotovap, silica gel, GC/FID	F4G benchmark CCME PHC method is 16-24 hour Soxhlet extraction, rotovap, silica gel, gravimetric. % moisture is gravimetric to constant weight.
		❑❑❑ implemented method comparable within 20% of CCME PHC method	❑❑❑ implemented method comparable within 20% of CCME PHC method	❑❑❑ implemented method comparable within 20% of CCME PHC method

Clause	Requirement	Y N N/A Comments	Y N N/A Comments	Y N N/A Comments
		❑❑❑ accuracy assessed by acceptable recoveries from samples validated by the CCME method	❑❑❑ accuracy assessed by acceptable recoveries from samples validated by the CCME method	❑❑❑ accuracy assessed by acceptable recoveries from samples validated by the CCME method
7.2.1.5	MDL is done on 7 or more replicates at the 99% CI	❑❑❑ MDL for F1 is less than 12 mg/kg (or 20% of Tier 1 guidelines (2008), whichever is higher) using soil spiked with gasoline at 50 to 200 mg/kg.	❑❑❑ MDL for F2 and F3 are done with weathered diesel spiked soil at 20 to 100 mg/kg and is less than 3.9 and 9.0 mg/kg. F4 is done with SAE 30 motor oil and is less than 8 mg/kg or 20% of Tier 1 guidelines (2008), whichever is higher.	❑❑❑ MDL for F4G done using soil spiked with SAE 30-weight motor oil at 2,000 to 10,000 mg/kg. MDL includes silica gel cleanup and is less than 290 mg/kg or 20% of Tier 1 guidelines (2008), whichever is higher.
7.2.1.5	precision at levels greater than 10 times MDL meets:	❑❑❑ C6 to C10 hydrocarbons 30%	❑❑❑ C10 to C50 hydrocarbons 20%	❑❑❑ F4G 30%

Clause	Requirement	Y N N/A Comments	Y N N/A Comments	Y N N/A Comments
	TEST METHOD CONTENT- TEST PROCEDURE			
7.2.1.2	All necessary successive steps in the test procedure (including details on reagent preparation, storage and shelf life) are adequately documented in the test method.	❑❑❑ method is based on CCME PHC Tier 1 Method including 100% poly(dimethylsiloxane) low bleed column using GC/FID	❑❑❑ method is based on CCME PHC Tier 1 Method including 100% poly(dimethylsiloxane) low bleed column using GC/FID	❑❑❑ method is based on CCME PHC Tier 1 Method
		❑❑❑ methanol extraction is done (minimum 2:1 methanol: wet solid)	❑❑❑ 1:1 hexane:acetone is used as extraction solvent	❑❑❑ 50:50 n-hexane:acetone is used as extraction solvent (minimum 20:1 solvent:dry soil ratio)
		❑❑❑ samples are diluted to be less than highest calibration peak	❑❑❑ samples are diluted to be less than highest calibration peak	❑❑❑ if F4G result without silica gel is less than 50% of CWS PHC criteria then report; if higher, then must do silica gel procedure

Clause	Requirement	Y N N/A Comments	Y N N/A Comments	Y N N/A Comments
		❑❑❑ integration is from the beginning of the nC6 peak to the apex of the nC10 peak	❑❑❑ chromatogram returns to baseline before C50; if it does not, then F4G required	❑❑❑ if polar/nonpolar separation is required, sample is redissolved in 50:50 Hexane:DCM
		❑❑❑ methanol extraction is done within 48 hours of arrival at lab and analyzed within 40 days	❑❑❑ all samples are cleaned with 0.6 grams per gram dry sample of 100% activated 60-200 mesh silica gel	❑❑❑ sample is stirred or shaken with 0.6 grams per gram dry sample of 100% activated 60 – 200 mesh silica gel for minimum 5 minutes, one time only
			❑❑❑ toluene is added before evaporation, evaporation avoids nC10 loss	❑❑❑ F4G is not added to GC data
			❑❑❑ integration is from apex to apex for C10-C16, C16-C34 and C34-C50	❑❑❑ F4G is dried at 100°C - 110°C to constant weight
			❑❑❑ extracted within 14 days and analyzed within 40 days	❑❑❑ % moisture is dried at 100°C - 110°C

Clause	Requirement	Y N N/A Comments	Y N N/A Comments	Y N N/A Comments
				overnight or to constant weight
	TEST METHOD CONTENT-SAMPLE HISTORY			
7.4.1	Sample history requirements are adequate, documented and readily available.	❑❑❑ samples are stored at 4°C, minimal headspace or preservation	❑❑❑ samples are stored at 4°C, minimal headspace or preservation	❑❑❑ samples are stored at 4°C, minimal headspace or preservation
		❑❑❑ sample is not dried with Na_2SO_4	❑❑❑ sample is not dried with Na_2SO_4	❑❑❑ sample is not dried with Na_2SO_4
		❑❑❑ sample label is complete	❑❑❑ sample label is complete	❑❑❑ sample label is complete
		❑❑❑ chain of custody	❑❑❑ chain of custody	❑❑❑ chain of custody
		❑❑❑ BTEX is sampled from the same bottle, if required	❑❑❑ PAH and GHH are sampled from the same bottle, if required	❑❑❑ PAH, % moisture and C10-C50 are sampled from the same bottle, if required
		❑❑❑ separate 5g samples are taken	❑❑❑ separate 5g samples are taken	❑❑❑ separate 5g samples are taken for both GHH and % moisture

Clause	Requirement	Y N N/A Comments	Y N N/A Comments	Y N N/A Comments
	TEST METHOD CONTENT-METHOD CALIBRATION			
6.4.6	Method calibration is appropriate, documented and implemented.	❑❑❑ calibration and retention time marking with toluene, nC6 and nC10 in methanol	❑❑❑ calibrate with C10, C16 and C34 in toluene with separate nC50 as retention time marker, not for quantitation	❑❑❑ balance is calibrated regularly
		❑❑❑ nC6 separates from solvent peak	❑❑❑ nC10 separates from solvent peak	
			❑❑❑ calibrate using average response factor for C10, C16 and C34; response factors for C10, C16 and C34 must be within 10% of the average response for the three.	

Clause	Requirement	Y N N/A Comments	Y N N/A Comments	Y N N/A Comments
		❑❑❑ nC6 and nC10 response factors are within 30% of toluene	❑❑❑ nC50 response factor must be no less than 70% of average of C10, C16 and C34 response factors.	
		❑❑❑ appropriate petroleum product is used as 2nd standard	❑❑❑ appropriate petroleum product is used as 2nd standard	
		❑❑❑ chromatographic linearity demonstrated with products (such as gasoline) within 15% for the range and single compounds within 10%.	❑❑❑ linearity is demonstrated with products (such as diesel or motor oil) with single compound calibration standards and is within 15% for each range and for single compounds, within 10%	
		❑❑❑ 3 point cal curve with toluene plus a blank	❑❑❑ 3 point cal curve with nC10, nC16 and nC34 plus a blank	

Clause	Requirement	Y N N/A Comments	Y N N/A Comments	Y N N/A Comments
		❑❑❑ check daily and recalibrate if low standard deviates by more than 20% or midpoint standard deviates by more than 15% from the curve	❑❑❑ check daily and recalibrate if low standard deviates by more than 20% or midpoint standard deviates by more than 15% from the curve	
	TEST METHOD CONTENT – METHOD QUALITY CONTROL			
7.7.1	Method quality control is appropriate, documented and implemented.	❑❑❑ QC samples run with each 20 samples include • method blank • method duplicate • performance sample (clean soils spiked with appropriate product or reference samples previously validated by CWS PHC)	❑❑❑ QC samples run with each 20 samples include • method blank • method duplicate • performance sample (clean soils spiked with appropriate product or reference samples previously validated by CWS PHC)	❑❑❑ QC samples run with each 20 samples include • method blank • method duplicate • performance sample (clean soils spiked with appropriate product or reference samples previously validated by CWS PHC)

Clause	Requirement	Y N N/A Comments	Y N N/A Comments	Y N N/A Comments
		❑❑❑ suitable petroleum products are used as QC samples	❑❑❑ suitable petroleum products are used as QC samples	❑❑❑ suitable petroleum products are used as QC samples
		❑❑❑ identification of measurement uncertainty	❑❑❑ identification of measurement uncertainty	❑❑❑ identification of measurement uncertainty
		❑❑❑ trend analysis (e.g., control charts)	❑❑❑ trend analysis (e.g., control charts)	❑❑❑ trend analysis (e.g., control charts)
	OTHER WORK INSTRUCTIONS/PROCEDURES			
7.2.1.2	All necessary supporting work instructions are current and readily available, e.g., • glassware cleaning • supporting test methods • equipment instruction manuals • requisite reference texts • computer software related procedures	❑❑❑	❑❑❑	❑❑❑
	CONDUCT OF TESTING			

Clause	Requirement	Y N N/A Comments	Y N N/A Comments	Y N N/A Comments
7.2.1.1	The test procedure and all supporting work instructions are performed as documented	❑❑❑	❑❑❑	❑❑❑
	EQUIPMENT			
6.4.1	All instruments required for the test procedure are available, uniquely identified, functioning properly, and safeguarded from adjustments that would invalidate results.	❑❑❑	❑❑❑	❑❑❑
	SUPPORT EQUIPMENT			
6.4.1	All support equipment* required for the test procedure is available and functioning properly. *includes computers	❑❑❑	❑❑❑	❑❑❑
	OUT OF SERVICE EQUIPMENT			
6.4.9	Out of service equipment is isolated or clearly labeled or marked as being out of service, and is checked and validated before return to service.	❑❑❑	❑❑❑	❑❑❑
	EQUIPMENT REQUIRING CALIBRATION			

Clause	Requirement	Y N N/A Comments	Y N N/A Comments	Y N N/A Comments
6.4.8	All equipment requiring calibration is labeled to indicate calibration status, including the date last calibrated and expiry date, or date when recalibration is due.* * not required for equipment checked daily or as-used	❑❑❑	❑❑❑	❑❑❑
	SUPPLIES – AVAILABILITY			
6.4.1	All supplies required for the test procedure are available and meet requisite requirements and/or specifications.* *includes reagents, reference materials, silica gel	❑❑❑	❑❑❑	❑❑❑
	SUPPLIES – STORAGE			
6.3.1	All supplies are stored under appropriate conditions (e.g., 4^{o}C) and in a manner that satisfies requirements for safety, security, separation of incompatible materials and ease of retrieval.	❑❑❑	❑❑❑	❑❑❑
	SUPPLIES – LABELING			

Clause	Requirement	Y N N/A Comments	Y N N/A Comments	Y N N/A Comments
6.4.8	All reagents and media are labeled with material, concentration or purity, date prepared and/or expiry date.	❑❑❑	❑❑❑	❑❑❑
	SUPPLIES – LABWARE			
6.4.1	All labware is adequately cleaned and, where required, labware quality control includes analytical testing, e.g., • glassware rinsed with hexane and air-dried • glassware blank run with every set of samples.	❑❑❑	❑❑❑	❑❑❑
	RECORD KEEPING			
7.5.1	Records related to the performance of the test method are retained; e.g., • analyst worksheet or notebook	❑❑❑	❑❑❑	❑❑❑

Clause	Requirement	Y N N/A Comments	Y N N/A Comments	Y N N/A Comments
	• record of non-conformances and action taken • reagent preparation log • equipment maintenance log • record of gravimetric traceability • record of volumetric traceability • record of temperature traceability			
	REPORTING – See separate checklist on next page			

CCME PETROLEUM HYDROCARBON REPORTING TEMPLATE

Y N N/A	Header information to identify the laboratory and the sample
❑❑❑	Name and address of laboratory
❑❑❑	Name and address of client
❑❑❑	Report number
❑❑❑	Identification of test sample
❑❑❑	Description of test sample
❑❑❑	Identification of test method
❑❑❑	Dates of sampling and reporting

Y N N/A	Hydrocarbon results expressed on a dry weight basis
❑❑❑	F1 C6 to C10 hydrocarbons in mg/kg, $F1_{-BTEX}$ after BTEX is subtracted
❑❑❑	F2 C10 to C16 hydrocarbons in mg/kg, $F2_{-napth}$ after naphthalene is subtracted
❑❑❑	F3 C16 to C34 hydrocarbons in mg/kg, $F3_{-PAH}$ after PAHS are subtracted
❑❑❑	F4 C34 to C50 hydrocarbons in mg/kg
❑❑❑	F4G by gravimetric heavy hydrocarbons in mg/kg, if analyzed: (Note that both of the two results for F4 and F4G are reported for F4 and a statement added to the report to effect that the greater of the two numbers are to be used in application to the CWS PHC
❑❑❑	$F4G_{-sg}$, if analyzed, is the result of gravimetric heavy hydrocarbons after silica gel treatment in mg/kg
❑❑❑	% moisture
❑❑❑	Total Organic Carbon, if requested
❑❑❑	Method detection limits
❑❑❑	Validator signature
❑❑❑	A note stating that gravimetric heavy hydrocarbons cannot be added to the C6 to C50 hydrocarbons
❑❑❑	Linearity is within 15%
❑❑❑	A note stating that BTEX and selected PAHs have been subtracted from the appropriate fractions

Y N N/A	Comments that are clearly separated from the results of analysis:

❑❑❑	A statement that the method complies with the Reference Method for the CWS PHC and is validated for use in the laboratory	
❑❑❑	All deviations from the method required are to be noted and reported for any particular sample	
❑❑❑	Qualifications on results	
❑❑❑	Subcontractors used	
❑❑❑	Did the chromatogram descend to baseline by the retention time of nC50?	
❑❑❑	Were the quality criteria met?	
❑❑❑	nC6 and nC10 response factors within 30% of response factor for toluene:	
	❑❑❑	nC10, nC16 and nC34 response factors within 10% of their average
	❑❑❑	C50 response factors within 70% of nC10 + nC16 + nC34 average
	❑❑❑	Linearity is within 15%
❑❑❑	Statement that the data for QC samples is available on request or the data for QC samples:	
	❑❑❑	Blank
	❑❑❑	Duplicate
	❑❑❑	Reference Sample
	❑❑❑	Spiked sample
❑❑❑	Extraction and analysis limits for holding time were met (Y/N)	
❑❑❑	Professional judgement, if requested, of what the material is, based on information that is stated (product profiles, retention times, professional experience, etc.)	