

Rating Guide Appendix for the

CCME Reference Method for the Canada-Wide Standard for Petroleum Hydrocarbons (PHC) in Soil - Tier 1 Method

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

(CCME CWS PHC and PERCENT MOISTURE)

Laboratory Name

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.

TEST SPECIFIC CHECKLIST FOR CCME PETROLEUM HYDROCARBONS

Item	Clause	Requirement	1 2 3 Comments	1 2 3 Comments	1 2 3 Comments NA = not applicable
	17025: 2005 (17025: 2017)		(F1) C6 - C10 Hydrocarbons	(F2-F4) C10 - C50 Hydrocarbons	(F4G) Gravimetric Heavy Hydrocarbons and % Moisture
01.01		TEST METHOD CURRENCY			
	4.3 (7.2.1.2)	The current authorized test method and necessary supporting work instructions are available to the analyst.	<input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/>	<input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/>	<input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/>
02.01		TEST METHOD VERIFICATION/VALIDATION			
	5.4 (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 <input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/> implemented method comparable within 20% of CCME PHC method <input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/> accuracy assessed by acceptable recoveries from samples validated by the CCME method	benchmark CCME PHC method is 16-24 hour Soxhlet extraction, rotovap, silica gel, GC/FID <input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/> implemented method comparable within 20% of CCME PHC method <input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/> accuracy assessed by acceptable recoveries from samples validated by the CCME method	F4G benchmark CCME PHC method is 16-24 hour Soxhlet extraction, rotovap, silica gel, gravimetric. % moisture is gravimetric to constant weight. <input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/> implemented method comparable within 20% of CCME PHC method <input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/> accuracy assessed by acceptable recoveries from samples validated by the CCME method

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	17025: 2005 (17025: 2017)		(F1) C6 – C10 Hydrocarbons	(F2-F4) C10 – C50 Hydrocarbons	(F4G) Gravimetric Heavy Hydrocarbons and % Moisture
	5.4 (7.2.1.5)	MDL is done on 7 or more replicates at the 99% CI	<input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/> 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.	<input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/> 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.	<input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/> 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.
	5.4 (7.2.1.5)	precision at levels greater than 10 times MDL meets:	<input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/> C6 to C10 hydrocarbons 30%	<input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/> C10 to C50 hydrocarbons 20%	<input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/> F4G 30%
03.01		TEST METHOD CONTENT- TEST PROCEDURE			
	4.2.1 5.4.1 (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.	<input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/> method is based on CCME PHC Tier 1 Method including 100% poly(dimethylsiloxane) low bleed column using GC/FID <input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/> methanol extraction is done (minimum 2:1 methanol: wet solid) <input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/> samples are diluted to be less than highest calibration peak	<input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/> method is based on CCME PHC Tier 1 Method including 100% poly(dimethylsiloxane) low bleed column using GC/FID <input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/> 1:1 hexane:acetone is used as extraction solvent <input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/> samples are diluted to be less than highest calibration peak	<input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/> method is based on CCME PHC Tier 1 Method <input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/> 50:50 n-hexane:acetone is used as extraction solvent (minimum 20:1 solvent:dry soil ratio) <input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/> if F4G result without silica gel is less than 50% of CWS PHC criteria then report; if higher, then must do silica gel procedure

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			<p><input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/> integration is from the beginning of the nC6 peak to the apex of the nC10 peak</p> <p><input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/> methanol extraction is done within 48 hours of arrival at lab and analyzed within 40 days</p>	<p><input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/> chromatogram returns to baseline before C50; if it does not, then F4G required</p> <p><input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/> all samples are cleaned with 0.6 grams per gram dry sample of 100% activated 60-200 mesh silica gel</p> <p><input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/> toluene is added before evaporation, evaporation avoids nC10 loss</p> <p><input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/> integration is from apex to apex for C10-C16, C16-C34 and C34-C50</p> <p><input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/> extracted within 14 days and analyzed within 40 days</p>	<p><input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/> if polar/nonpolar separation is required, sample is redissolved in 50:50 Hexane:DCM</p> <p><input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/> 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</p> <p><input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/> F4G is not added to GC data</p> <p><input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/> F4G is dried at 100°C - 110°C to constant weight</p> <p><input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/> % moisture is dried at 100°C - 110°C overnight or to constant weight</p>

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04.01		TEST METHOD CONTENT-SAMPLE HISTORY			
	5.7 7.4.1	Sample history requirements are adequate, documented and readily available.	<input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/> samples are stored at 4°C, minimal headspace or preservation <input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/> sample is not dried with Na ₂ SO ₄ <input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/> sample label is complete <input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/> chain of custody <input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/> BTEX is sampled from the same bottle, if required <input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/> separate 5g samples are taken	<input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/> samples are stored at 4°C, minimal headspace or preservation <input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/> sample is not dried with Na ₂ SO ₄ <input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/> sample label is complete <input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/> chain of custody <input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/> PAH and GHH are sampled from the same bottle, if required <input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/> separate 5g samples are taken	<input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/> samples are stored at 4°C, minimal headspace or preservation <input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/> sample is not dried with Na ₂ SO ₄ <input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/> sample label is complete <input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/> chain of custody <input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/> PAH, % moisture and C10-C50 are sampled from the same bottle, if required <input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/> separate 5g samples are taken for both GHH and % moisture

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	17025: 2005 (17025: 2017)		(F1) C6 - C10 Hydrocarbons	(F2-F4) C10 - C50 Hydrocarbons	(F4G) Gravimetric Heavy Hydrocarbons and % Moisture
05.01		TEST METHOD CONTENT-METHOD CALIBRATION			
	5.4.5 5.6.2.2 5.6.3 (6.4.6)	Method calibration is appropriate, documented and implemented.	<input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/> calibration and retention time marking with toluene, nC6 and nC10 in methanol <input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/> nC6 separates from solvent peak <input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/> nC6 and nC10 response factors are within 30% of toluene <input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/> appropriate petroleum product is used as 2 nd standard	<input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/> calibrate with C10, C16 and C34 in toluene with separate nC50 as retention time marker, not for quantitation <input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/> nC10 separates from solvent peak <input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/> 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. <input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/> nC50 response factor must be no less than 70% of average of C10, C16 and C34 response factors. <input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/> appropriate petroleum product is used as 2 nd standard	<input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/> balance is calibrated regularly

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			<input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/> chromatographic linearity demonstrated with products (such as gasoline) within 15% for the range and single compounds within 10%. <input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/> 3 point cal curve with toluene plus a blank <input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/> check daily and recalibrate if low standard deviates by more than 20% or midpoint standard deviates by more than 15% from the curve	<input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/> 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% <input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/> 3 point cal curve with nC10, nC16 and nC34 plus a blank <input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/> check daily and recalibrate if low standard deviates by more than 20% or midpoint standard deviates by more than 15% from the curve	

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06.01		TEST METHOD CONTENT – METHOD QUALITY CONTROL			
	5.9 7.1.1	Method quality control is appropriate, documented and implemented.	<input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/> QC samples run with each 20 samples include <ul style="list-style-type: none"> • method blank • method duplicate • performance sample (clean soils spiked with appropriate product or reference samples previously validated by CWS PHC) <input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/> suitable petroleum products are used as QC samples	<input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/> QC samples run with each 20 samples include <ul style="list-style-type: none"> • method blank • method duplicate • performance sample (clean soils spiked with appropriate product or reference samples previously validated by CWS PHC) <input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/> suitable petroleum products are used as QC samples	<input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/> QC samples run with each 20 samples include <ul style="list-style-type: none"> • method blank • method duplicate • performance sample (clean soils spiked with appropriate product or reference samples previously validated by CWS PHC) <input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/> suitable petroleum products are used as QC samples
			<input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/> identification of measurement uncertainty	<input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/> identification of measurement uncertainty	<input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/> identification of measurement uncertainty
			<input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/> trend analysis (e.g., control charts)	<input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/> trend analysis (e.g., control charts)	<input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/> trend analysis (e.g., control charts)
07.01		OTHER WORK INSTRUCTIONS/PROCEDURES			
	5.4.1 (7.2.1.2)	All necessary supporting work instructions are current and readily available, e.g., <ul style="list-style-type: none"> • glassware cleaning • supporting test methods • equipment instruction manuals • requisite reference texts • computer software related procedures 	<input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/>	<input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/>	<input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/>

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08.01		CONDUCT OF TESTING			
	5.4.1 4.2.1 (7.2.1.1)	The test procedure and all supporting work instructions are performed as documented	<input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/>	<input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/>	<input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/>
09.01		EQUIPMENT			
	5.5.1 5.5.2 5.5.4 5.5.12 (6.4.1)	All instruments required for the test procedure are available, uniquely identified, functioning properly, and safeguarded from adjustments that would invalidate results.	<input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/>	<input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/>	<input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/>
09.02		SUPPORT EQUIPMENT			
	5.5.1 (6.4.1)	All support equipment* required for the test procedure is available and functioning properly. *includes computers	<input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/>	<input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/>	<input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/>
09.03		OUT OF SERVICE EQUIPMENT			
	5.5.7 5.5.9 (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.	<input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/>	<input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/>	<input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/>

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09.04		EQUIPMENT REQUIRING CALIBRATION			
	5.5.8 (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	<input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/>	<input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/>	<input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/>
10.01		SUPPLIES - AVAILABILITY			
	4.6.2 5.5.1 (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	<input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/>	<input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/>	<input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/>
10.02		SUPPLIES - STORAGE			
	5.3 (6.3.1)	All supplies are stored under appropriate conditions (e.g., 4°C) and in a manner that satisfies requirements for safety, security, separation of incompatible materials and ease of retrieval.	<input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/>	<input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/>	<input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/>

Item	Clause	Requirement	1 2 3 Comments	1 2 3 Comments	1 2 3 Comments NA = not applicable
10.03		SUPPLIES - LABELING			
	4.13.2 (6.4.8)	All reagents and media are labeled with material, concentration or purity, date prepared and/or expiry date.	<input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/>	<input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/>	<input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/>
10.05		SUPPLIES - LABWARE			
	5.5.1 (6.4.1)	All labware is adequately cleaned and, where required, labware quality control includes analytical testing, e.g., <ul style="list-style-type: none"> • glassware rinsed with hexane and air-dried • glassware blank run with every set of samples. 	<input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/>	<input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/>	<input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/>
11.01		RECORD KEEPING			
	4.13.2 4.9 4.13.2 5.5.5 5.6 (7.5.1)	Records related to the performance of the test method are retained; e.g., <ul style="list-style-type: none"> • analyst worksheet or notebook (1) • record of non-conformances and action taken (2) • reagent preparation log (3) • equipment maintenance log (4) • record of gravimetric traceability (5) • record of volumetric traceability (6) • record of temperature traceability (7) 	<input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/>	<input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/>	<input type="checkbox"/> <input type="checkbox"/> <input type="checkbox"/>

Item	Clause	Requirement	1 2 3 Comments	1 2 3 Comments	1 2 3 Comments NA = not applicable
12.01		REPORTING			
	5.10 (7.8)	See checklist on page 12			

- (1) includes, as appropriate, calibration data, test date (including QC data), experimental variables (e.g. temperature, etc.); analyst ID; sample ID; equipment ID; test method ID; date and time of test.
- (2) includes, as appropriate, nonconformances related to: test method variances; sample history; method performance; interferences; and data validation.
- (3) includes, as appropriate, supplier, grade, batch no; dates of preparation or verification; measurement of weights, volumes, time intervals, temperatures and related calculations; relevant processes (e.g., pH adjustment, sterilization); verification results; discard date.
- (4) includes, as appropriate, identity of the equipment and it's software; manufacturer, model, serial no, checks that equipment complies with laboratory specifications; date commissioned; repair and maintenance history; calibration history; location; any damage, malfunction or modification to the equipment.
- (5) includes, as appropriate, traceability of balance and/or weights to a national standard; daily or as-used checks (See A61-Traceability Policy).
- (6) includes, as appropriate, traceability of auto pipettes, dilutors, etc. that play a defining role in analytical accuracy, and daily or as-used checks (see A-61-Traceability Policy).
- (7) includes, as appropriate, calibration of working thermometers against a calibrated thermometer for those working thermometers that measure temperatures that play a defining role in analytical accuracy (see A61- Traceability Policy).

CCME PETROLEUM HYDROCARBON REPORTING TEMPLATE

1 2 3

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

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-naphth 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

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?

Rev. 1.11

Were the quality criteria met?

1 2 3

- 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.)