

A130 CALA Checklist for TCLP Revision 1.2 – January 30th, 2018

Laboratory Name: _____

Appendix Name: _____

Appendix Number: _____

Assessors: _____

Date: _____



CALA

Laboratory Accreditation

**Note: This method-specific checklist is to be used in conjunction with
A03 - Rating Guide Appendix**

Item	Specification	Y	N	N/A
<i>Equipment</i>	A03 Item: 09.01 A03-2017 Clause: 6.4			
Agitator	End-over-end at 30±2 rpm (must) (EPA 1311 4.1)
Zero-Headspace Extraction (ZHE)	<ul style="list-style-type: none"> used only for volatiles (must) 500-600ml internal volume (must) accommodates 90-110 mm filter (must) withstands 50 psi filtration pressure (must) suitable devices known to EPA are in Table 3 of EPA 1311 (EPA 1311 4.2.1)
Bottle Extraction	<ul style="list-style-type: none"> for nonvolatile extraction (must) no plastic bottles, except PTFE, if testing for organics (must) (EPA 1311 4.2.2)
Filtration device for bottle extractions	<ul style="list-style-type: none"> minimum 300 ml internal volume (must) accommodate minimum 47 mm filter (must) vacuum filtration can only be for wastes with low solids (<10%) and highly granular, liquid-containing wastes (must). (Note: any device capable of separating the liquid from the solid phase of the waste is suitable, providing that it is chemically compatible with the waste and constituents to be analyzed) inert materials that will not leach or absorb used for extraction vessels and filtration devices (must); (e.g., glass, PTFE or 316 stainless steel for organics and inorganics) HDPE, PP or PVC cannot be used for organics (must) suitable filter holders known to EPA are in Table 4 of EPA 1311 (EPA 1311 4.3)
Filters	<ul style="list-style-type: none"> borosilicate glass fiber, no binders (must) 0.6-0.8 µm pore size, or equivalent (must) no prefilters (must) for metals: acid-washed with 1N nitric acid then 3 rinses of DI distilled water (must) (EPA 1311 4.4)
ZHE collection	TEDLAR bags, stainless steel or PTFE gas-tight syringes for initial liquid phase and final extract (must) (EPA 1311 4.6)
ZHE extract transfer	Transfer device for extraction fluid to ZHE does not change nature of extraction fluid (must) (EPA 1311 4.7)

Item	Specification	Y	N	N/A
Equipment (Continued)				
Balance	Weighing to $\pm 0.1g$ (must) ; (requires minimum 2 place balance) (EPA 1311 4.8)
Reagents	A03 Item: 10.01 A03- 2017 Clause: 6.4			
	<ul style="list-style-type: none"> reagent/ACS grade minimum (must) reagent water: interferants below target MDLs (must) Extraction Fluid #1: pH = 4.93 ± 0.05 (must) Extraction Fluid #2: pH = 2.88 ± 0.05 (must) extraction fluids monitored frequently for impurities pH of extraction fluids checked before use if impurities found or pH of extraction fluid out of spec, fluid discarded (must) (EPA 1311 5)
Sample Collection and Preservation	A03 Item:04 A03-2017 Clause: 7.3.1			
	<ul style="list-style-type: none"> collected using appropriate sampling plan (must) preservatives not added before extraction (must) samples not refrigerated if this causes irreversible physical change; if precipitate forms, entire sample (including precipitate) is extracted samples for volatiles taken and stored to prevent losses (must); (e.g., Teflon-lined septum capped vials, stored at 4°C) extracts for metals acidified with nitric acid to pH <2 unless precipitation occurs (must) extracts for organics have no headspace (must) (EPA 1311 6)
Preliminary Evaluations	A03 Item: 03.01 A03-2017 Clause:7.2.1.1			
Percent solids	<ul style="list-style-type: none"> determined for samples that are not 100% solid (must) minimum 100g sample (must) filtered at 1-10 psi (must) if filtration not complete, increased to 50 psi in 10 psi increments at 2 minute intervals until filtration complete (must) if >1% waste remains in transfer flask, this weight is subtracted from subsample weight (must) for matrices that won't filter (oily, paints), only one filter used (must) percent solids (wet) calculated (must) (EPA 1311 7.1.1) <ul style="list-style-type: none"> if percent solids (wet) $\geq 0.5\%$ and some filtrate is entrained on filter, percent solids (dry) determined (must):

Item	Specification	Y	N	N/A
	<ul style="list-style-type: none"> ○ residue dried at 100±20°C until 2 successive readings are within ±1% (must) • if percent solids (dry) ≥0.5%, new waste subsample taken and particle size reduction determination performed (must) • pre-weigh filters and receiving containers (EPA 1311 7.1.2)
Particle Size reduction determination	<ul style="list-style-type: none"> • reduction determination completed if percent solids (wet or dry) >0.5% (must) • performed unless solid surface area per gram ≥ 3.1 cm², or ≤1 cm in narrowest dimension (passes through 9.5 mm sieve) (must) • reduction by crushing, cutting or grinding (must) (EPA 1311 7.1.3)
Extraction Fluid for volatiles	<ul style="list-style-type: none"> • Extraction Fluid #1 for volatiles (must) (EPA 1311 7.1.4)
Extraction Fluid selection for non-volatiles	<ul style="list-style-type: none"> • 5.0g, particle size <1 mm, mixed with 96.5 ml reagent water, stirred 5 minutes with a watch glass cover (must) • pH < 5.0: Extraction Fluid #1 used (must) • pH >5.0: 3.5 ml HCl , slurry, heat to 50°C with a watch glass cover, hold for 10 minutes; cooled, pH reread; if pH <5.0 Extraction Fluid #1 used, otherwise Extraction Fluid #2 used (must) (EPA 1311 7.1.4)
Subsequent aliquot needed	<ul style="list-style-type: none"> • not needed if original was 100% solid and 100 g remain for non-volatiles or 25g for volatiles • new aliquot for volatiles taken if original was filtered (must) (EPA 1311 7.1.5)

Item	Specification	Y	N	N/A
Procedure	A03 Item:03.01 A03-2017 Clause:7.2.1.1			
For non-volatiles	<ul style="list-style-type: none"> • 100g minimum (must) • liquid/solid filtration done, if sample is multi-phasic (must) • filters acid-washed if metals to be analysed (must) • filtered at 1-10 psi (must) • if filtration not complete, increased to 50 psi in 10 psi increments at 2 minute intervals until filtration complete (must) • if >1% waste remains in transfer flask, this weight is subtracted from sample weight (must) • for matrices that won't filter (oily, paints), only one filter used (must) • reduction (crushing, cutting or grinding) completed if percent solids (dry) >0.5% and particle size reduction previously performed (must) • filter included in extractor bottle with solids (must) • Extraction fluid volume: (20x %solids(wet)x weight of waste filtered)/100 (must) • Waste+extraction fluid rotated 30±2 rpm for 18±2 hrs (must) • Temperature: 23±2°C during extraction (must) • After extraction time, sample filtered through new filter (acid-washed filters if metals analysis required) (must); more than one filter can be used <p>(EPA 1311 7.2)</p>
TCLP Preparation – non-volatile analytes	<ul style="list-style-type: none"> • If waste sample contained no initial liquid phase, the filtered liquid after extraction is TCLP extract (must) • If single phase will result, combined filtered liquid from extraction and initial liquid from first filtration is TCLP extract (must) • If multiphases will result, filtered liquid from extraction and initial liquid from first filtration are not combined but analysed separately as TCLP extracts and mathematically combined (must) <p>(EPA 1311 7.2.13)</p> <ul style="list-style-type: none"> • pH of extract recorded • extracts aliquotted and preserved immediately (must) • metal aliquots acidified with nitric acid to pH<2 (must); <ul style="list-style-type: none"> ○ if precipitate forms, remaining extract for metals not to be preserved and analysed asap (must) ○ metal extracts digested unless this causes analyte losses (must) • other aliquots refrigerated storage (4°C) (must) • if multi-phase extracts analysed separately, results combined: $C = \frac{v1c1 + v2c2}{v1+v2}$ (must)

Item	Specification	Y	N	N/A
	<ul style="list-style-type: none"> volumes measured $\pm 0.5\%$ (must) (EPA 1311 7.2.14)
TCLP Preparation - Volatile analytes	<ul style="list-style-type: none"> use ZHE only (must) ZHE device capable of holding sufficient extraction fluid for 20:1 ratio extraction fluid:solid phase (w/w) (must), so maximum 25 g solid and ZHE with approximately 500 ml internal capacity (must) ZHE charged once only and not opened until final extract collected (must) Exposure of waste, liquid phase or extract to atmosphere prevented (must) Manipulation of waste, liquid phase or extract done at 4°C (must) If TEDLAR bags used, liquid from ZHE transferred to bag; aliquots taken from bag (must) (EPA 1311 7.3.1-2) for 100% solid wastes: 25g maximum subsample used (must) (EPA 1311 7.3.3) for <0.5% dry solids wastes, liquid after filtration used as TCLP extract (must) for $\geq 0.5\%$ dry solids, sample size (g) based on percent solids: <ul style="list-style-type: none"> Wastes with <5% solids (wet), use 500g Wastes with >5% solids (wet), use 25x100/%solids (wet) If particle size reduction previously done on samples: <ul style="list-style-type: none"> Crush, cut, or grind solids until solid surface area per gram $\geq 3.1 \text{ cm}^2$, or $\leq 1 \text{ cm}$ in narrowest dimension (passes through 9.5 mm sieve); sieving is not recommended at this stage as volatiles may be lost (must) No heat created by crushing, cutting or grinding (must) Atmospheric exposure minimized (must) No centrifuging wastes prior to filtration (EPA 1311 7.3.6) if >1% waste remains in transfer container, this weight is deducted from sample weight for filtration (must) (EPA 1311 7.3.7) headspace removed from ZHE slowly if 100% solid waste, pressure increased to maximum 50 psi to force out most of headspace (EPA 1311 7.3.8) extract at 1-10 psi, increasing to 50 psi in 10 psi increments at 2 minute intervals until filtration complete (must) (EPA 1311 7.3.9)

Item	Specification	Y	N	N/A
	<ul style="list-style-type: none"> If original waste <0.5% dry solids, filtrate is TCLP extract, otherwise separate liquid and solid phases (must) (EPA 1311 7.3.10)
Weight of Extraction Fluid #1 to add to ZHE	<ul style="list-style-type: none"> For liquid phase: Weight = (20 x %wet solids x wt of waste filtered)/100 (must) (EPA 1311 7.3.11) <ul style="list-style-type: none"> For solid phase: ZHE filled with no headspace (must) Pressurized to 5-10 psi if necessary to bleed out the headspace (must) Waste+extraction fluid rotated 30±2 rpm for 18±2 hrs (must) Temperature: 23±2°C during extraction (must) (EPA 1311 7.3.12) <ul style="list-style-type: none"> If pressure not remaining in ZHE after extraction, extraction repeated with new aliquot (must) Filtered through ZHE (must) (EPA 1311 7.3.13) <ul style="list-style-type: none"> Liquid and solid phases separated; initial liquid (if any) and to this liquid (in TEDLAR bag unless multiphase would occur or insufficient bag volume (must) (EPA 1311 7.3.14) <ul style="list-style-type: none"> refrigerated storage (4°C) (must) if multi-phase extracts analysed separately, results combined: $C = \frac{v1c1 + v2c2}{v1+v2}$ (must) (EPA 1311 7.3.14)

Item	Specification	Y	N	N/A																																			
Quality Assurance	A03-Item: 06.01 A03-2017 Clause: 7.7																																						
Blanks	<ul style="list-style-type: none"> minimum 1 blank/20 extractions with same extraction fluid (must) (EPA 1311 8.1)																																			
Matrix Spikes	<ul style="list-style-type: none"> matrix spike per waste type unless result >regulation (must) minimum 1 matrix spike per analytical batch (must) matrix spike added after filtration of extract before preservation (must) matrix spike not less than 5xMDL (must) (EPA 1311 8.2)																																			
Internal calibration for metals	<ul style="list-style-type: none"> standard additions used if recovery <50% and concentration below regulatory limit, and concentration is within 20% of regulatory limit (must) 4 identical aliquots adding known amounts to 3 aliquots (must) 1st addition: 50% expected concentration of sample 2nd addition: 100% expected sample concentration 3rd addition: 150% expected sample concentration all aliquots made to same volume (EPA 1311 8.4)																																			
Hold Times	A03 Item:04.01 A03-2017 Clause: 7.4																																						
Hold times	<table border="1" style="width: 100%; border-collapse: collapse;"> <thead> <tr> <th colspan="5">SAMPLE MAXIMUM HOLDING TIMES [DAYS]</th> </tr> <tr> <th></th> <th>From: Field collection</th> <th>From: TCLP extraction</th> <th>From: Preparative extraction</th> <th></th> </tr> <tr> <th></th> <th>To: TCLP extraction</th> <th>To: Preparative extraction</th> <th>To: Determinative analysis</th> <th>Total elapsed time</th> </tr> </thead> <tbody> <tr> <td>Volatiles</td> <td>14</td> <td>NA</td> <td>14</td> <td>28</td> </tr> <tr> <td>Semi-volatiles</td> <td>14</td> <td>7</td> <td>40</td> <td>61</td> </tr> <tr> <td>Mercury</td> <td>28</td> <td>NA</td> <td>28</td> <td>56</td> </tr> <tr> <td>Metals, except mercury</td> <td>180</td> <td>NA</td> <td>180</td> <td>360</td> </tr> </tbody> </table> <p>(must)</p> <ul style="list-style-type: none"> if hold time exceeded, result is considered minimum (must) (EPA 1311 8.5)	SAMPLE MAXIMUM HOLDING TIMES [DAYS]						From: Field collection	From: TCLP extraction	From: Preparative extraction			To: TCLP extraction	To: Preparative extraction	To: Determinative analysis	Total elapsed time	Volatiles	14	NA	14	28	Semi-volatiles	14	7	40	61	Mercury	28	NA	28	56	Metals, except mercury	180	NA	180	360
SAMPLE MAXIMUM HOLDING TIMES [DAYS]																																							
	From: Field collection	From: TCLP extraction	From: Preparative extraction																																				
	To: TCLP extraction	To: Preparative extraction	To: Determinative analysis	Total elapsed time																																			
Volatiles	14	NA	14	28																																			
Semi-volatiles	14	7	40	61																																			
Mercury	28	NA	28	56																																			
Metals, except mercury	180	NA	180	360																																			