A130 CALA CHECKLIST FOR TCLP

Revision #1.3

January 3, 2020

Laboratory Name: _____

Appendix Name: _____

Appendix Number: _____

Assessors: _____

Date: _____

CALA

Trust, measured accurately

NOTE: THIS METHOD-SPECIFIC CHECKLIST IS TO BE USED IN CONJUNCTION WITH A03 – RATING GUIDE APPENDIX

Item	Specification	Y	N	N/A
Equipment	**A03-2017 Clause: 6.4**			
Agitator	End-over-end at 30±2 rpm **(must)** (EPA 1311 4.1)	.....	.....	.....
Zero-Headspace Extraction (ZHE)	• 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	• for nonvolatile extraction **(must)** • no plastic bottles, except PTFE, if testing for organics **(must)** (EPA 1311 4.2.2)	....	....	....
Filtration device for bottle extractions	• 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)	....	....	....

Item	Specification	Y	N	N/A
Filters	• 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)	.....	.....	.....
Balance	Weighing to ±0.1g **(must)**; (requires minimum 2 place balance) (EPA 1311 4.8)	.....	.....	.....
Reagents	**A03- 2017 Clause: 6.4**			
	• 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)	.....	.....	.....

Item	Specification	Y	N	N/A
Sample Collection and Preservation	**A03-2017 Clause: 7.3.1**			
	• 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)	.....	.....	.....

Item	Specification	Y	N	N/A
Preliminary Evaluations	**A03-2017 Clause:7.2.1.1**			
Percent solids	• 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) • if percent solids (wet) ≥0.5% and some filtrate is entrained on filter, percent solids (dry) determined **(must)**: • 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	• 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	• Extraction Fluid #1 for volatiles **(must)** (EPA 1311 7.1.4)	.....	.....	.....

Item	Specification	Y	N	N/A
Extraction Fluid selection for non-volatiles	• 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)	.....	.....	.....
Susbsequent aliquot needed	• 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 Procedure	Specification A03-2017 Clause: 7.2.1.1	Y	N	N/A
For non-volatiles	• 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 (EPA 1311 7.2)	…… …… …… …… …… … …… …… …… …… …… …… …… …… …… …… …… …… …… …… …… …… ……	…… ….	…… ……

Item	Specification	Y	N	N/A
TCLP Preparation – non-volatile analytes	• 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)** (EPA 1311 7.2.13) • pH of extract recorded • extracts aliquotted and preserved immediately **(must)** • metal aliquots acidified with nitric acid to pH<2 **(must)**; • 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)** • volumes measured ±0.5% **(must)** (EPA 1311 7.2.14)	.....	.....	.....

TCLP Preparation – Volatile analytes	• 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 ≥0.5% dry solids, sample size (g) based on percent solids: ▪ Wastes with <5% solids (wet), use 500g ▪ Wastes with >5% solids (wet), use 25x100/%solids (wet) (EPA 1311 7.3.4) • If particle size reduction previously done on samples: • Crush, cut, or grind solids until solid surface area per gram ≥ 3.1 cm2, or ≤1 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)** (EPA 1311 7.3.6) • No centrifuging wastes prior to filtration (EPA 1311 7.3.7)	.....	.....	.....

Item	Specification	Y	N	N/A
	• if >1% waste remains in transfer container, this weight is deducted from sample weight for filtration **(must)** • 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) • If original waste <0.5% dry solids, filtrate is TCLP extract, otherwise separate liquid and solid phases **(must)** (EPA 1311 7.3.10)			

Item	Specification	Y	N	N/A
Weight of Extraction Fluid #1 to add to ZHE	• For liquid phase: Weight = (20 x %wet solids x wt of waste filtered)/100 **(must)** (EPA 1311 7.3.11) • 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) • If pressure not remaining in ZHE after extraction, extraction repeated with new aliquot **(must)** • Filtered through ZHE **(must)** (EPA 1311 7.3.13) • 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) • 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)	.....	.....	.....
Quality Assurance	**A03-2017 Clause: 7.7**			
Blanks	• minimum 1 blank/20 extractions with same extraction fluid **(must)** (EPA 1311 8.1)	.....	.....	.....
Matrix Spikes	• 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)	.....	.....	.....

<table>
<tr><th>Item</th><th>Specification</th><th>Y</th><th>N</th><th>N/A</th></tr>
<tr><td>Internal calibration for metals</td><td>• 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)</td><td>.....
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<tr><th>Hold Times</th><th>A03-2017 Clause: 7.4</th><th></th><th></th><th></th></tr>
<tr><td>Hold times</td><td>SAMPLE MAXIMUM HOLDING TIMES [DAYS]
<table><tr><th></th><th>From: Field collection

To: TCLP extraction</th><th>From: TCLP extraction

To: Preparative extraction</th><th>From: Preparative extraction

To: Determinative analysis</th><th>Total elapsed time</th></tr><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></table>(must)
• if hold time exceeded, result is considered minimum (must)
(EPA 1311 8.5)</td><td>.....
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