Fill out a New SOP when:
   1) The extraction solution is prepared.

Fill out New Appendix when:
   2) Previously prepared extraction solution is on a day different than the prepared date.

1.0 SCOPE

1.1 The acid ammonium oxalate extraction (McKeague and Day, 1965) targets poorly crystalline iron and aluminum, while leaving the more crystalline forms of iron and aluminum intact.

2.0 DEFINITIONS

2.1 Laboratory Control Sample: The laboratory control sample is an intralaboratory developed sample whose true value is approximated by the average of repeated measures.

2.2 Duplicate Samples: A duplicate test involves splitting a sample to sub-samples and processing each through the same sample preparation procedure in order to determine the precision of the method.

2.3 Preparation Blank: The Preparation Blank is a sample that contains only the reagents used in the extraction procedure. The preparation blanks is processed through the same preparation procedures as the samples and therefore gives an indication of any contamination picked up during the sample preparation process.

2.4 ICP-AES: Inductively Coupled Plasma-Atomic Emission Spectrometry.

3.0 EQUIPMENT AND SUPPLIES

3.1 Automatic extractant dispenser, 25 mL capability.

3.2 pH Meter accurate to 0.05 units

3.3 Laboratory Balance: Any laboratory balance accurate to within ± 0.0001 grams may be used (all weight measurements are to be within ± 0.001 grams).

3.4 Extraction vessels, 50ml centrifuge tubes
3.5 ≥18 MΩ deionized water (DI)
3.6 Benchtop shaker
3.7 Glass scintillation vials
3.8 15ml Falcon tubes
3.9 High speed centrifuge
3.10 Ammonium oxalate (NH$_4$)$_2$C$_2$O$_4$ · H$_2$O
3.11 Oxalic acid H$_2$C$_2$O$_4$ · 2H$_2$O
3.12 Trace metal grade nitric acid

4.0 PROCEDURE

4.1 Oven dry samples at 60°C.
4.2 Grind samples with either mortar and pestle or puck mill if <250µm fraction is being used. No preparation is necessary for >250µm size fractions.
4.3 Calibrate pH meter and record result in Appendix.
4.4 0.2M acid ammonium oxalate solution (Ph 3.0).
   4.4.1 Solution A: 0.2M Oxalate solution (NH$_4$)$_2$C$_2$O$_4$ · H$_2$O (28.3g/L)
   4.4.2 Solution B: 0.2M Oxalic acid solution (H$_2$C$_2$O$_4$ · 2H$_2$O (25.2 g/L)
   4.4.3 Mix 700ml of A and 535 ml of B, adjust pH to 3.0 with A or B
4.5 Weigh 0.25 (±0.001g) into 50ml centrifuge tubes and separate into batches of 14 according to analysis sheet labels.
4.6 Check extraction solution pH at time of extraction and record in Appendix.
4.7 Check bottle top dispenser calibration with DI water and record results in Appendix.
4.8 Add 25ml of extraction fluid in batches of 14 samples.
   4.8.1 Write start time of extraction on each batch of 14.
   4.8.2 Stagger batches by 15 (or more) minutes to allow for centrifugation to stop extraction at exactly four hours.
   4.8.3 Cover tubes to allow extraction to take place in darkness and shake for four hours.
4.9 After four hours, remove extractions from shaker and immediately centrifuge for 15 minutes at 9,000 rpm.

4.10 Being careful not to transfer soil, pour off extracts into labeled scintillation vials.

4.11 Dilute extracts x5 with 3% HNO$_3$ into labeled falcon tubes.

5.0 QUALITY CONTROL

5.1 Laboratory Control Sample (LCS): The laboratory control sample must fall within ± 20% of the known value or within the 95% prediction interval of the certified value. The laboratory control sample must be run with each batch (14) of extractions.

5.2 Sample Duplicates: The relative percent difference (RPD) must be no more than ±20%. At least one sample duplicate must be run with every batch (14) of extractions.

\[
RPD = 100 \times \frac{(S - D)}{Avg. (S,D)}
\]

5.3 Preparation Blank: If any analyte concentration is above the method detection limit in the preparation blank, the lowest concentration of the analyte reported in associated samples must be ≥ 10 times the preparation blank concentration. A preparation blank must be run with every batch (14) of extractions.

6.0 REPORTING

7.0 CORRECTIVE ACTION

8.0 REFERENCES


9.0 APPENDIX

Extraction Solution pH at time of extraction

Initials/Date

Pipette Calibration
# Standard Operating Procedure

## Ammonium Oxalate Extraction

Soil Environmental Chemistry Program, The Ohio State University

**Version 4**

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## 10.0 INTERPRETATION