Palintest Ltd.

Determination of the Lead concentration in extracts from the Toxicity Characteristic Leaching Procedure (TCLP) using the SA-5000

The Palintest SA-5000 Scanning Analyzer, and equivelant models, provides accurate and precise means of determining lead in a number of sample types. The following application note describes two methods for determining the amount of lead present in the extraction fluid from a TCLP test using the Water Program or the Dust Program on the SA-5000.

Lead in TCLP extracts using the Water Program.

Test Range and units

The Water Program on the SA-5000 Scanning Analyzer gives the test result in terms of micrograms of lead per liter (ug/l). This is equivelant to parts per billion (ppb). The test range is 2 - 100 ug/l.

Equipment and Materials

Volumetric flask, 100ml.
Glass pipette, 1ml.
Sample bottle, 100ml.
Pipettor, 5ml.
PALINTEST SA-5000 SCANNING ANALYZER
Test tubes, 5ml. plastic
Palintest SE-1LW or SE-1 W Electrodes
Palintest SoluPrep SP-A tablets
Tablet Crushing/Stirring rods

The equipment and consumables required (except volumetric flask, glass pipette and sample bottle) are supplied in the Palintest SA-5000 Scanning Analyzer Standard Kit (PT 471) and Water Electrode Pack. (PT 478).

Sample Preparation

1. Accurately pipette 1ml. of extraction fluid into a volumetric flask (100ml.). Make up to 100ml. with deionised or distilled water. Mix thoroughly by inverting the volumetric flask several times. Pour the diluted extract into the sample bottle.

2. Take a 5ml. screw capped test tube from the Electrode Pack. Carefully pour a portion of the diluted extract into the tube filling to the 5ml. mark.

3. Add one SoluPrep SP-A tablet and crush with a new crushing/stirring rod until completely dissolved.

4. Replace the screw cap and invert the tube several times until well mixed.

5. Test the sample with the Scanning Analyzer- see Scanning Analyzer Test Procedure (SCAN.11, page 10) in the SA-5000 handbook. Use an SE-1 electrode supplied in the SE-1 LW or SE-1 W electrode pack and choose the Water Test from the Analysis Screen. Ensure that the correct calibration code shown on the electrode pack is keyed into the instrument. The Scanning Analyzer will provide the test result after a three minute period.

6. The result is displayed as ug lead/liter.

7. The concentration of lead in the TCLP extract is therefore

SA-5000 reading *100ug lead/liter

e.g. SA-5000 reading = 45ug/l
Lead in TCLP extract = 45 * 100=4500ug/l or 4.5 parts per million (ppm)

Lead in TCLP extracts can be determined over the range 0.2ppm to 10ppm

Lead in TCLP extracts using the Dust Program

Test range and units

The Dust Program on the SA-5000 Scanning Analyzer gives the test result in terms of micrograms of lead per sample (ug/sample). The test range is 25 - 1500 ug/sample.

Equipment and Materials

Volumetric flask, 50ml.
Glass pipette, 5ml. (Graduated in 0.25 steps)
Screw capped sample bottle, 100ml.

concentrated nitric acid

Pipettor, 5ml.
PALINTEST SA-5000 Scanning Analyzer
Test tubes, 5ml. plastic
Palintest SE-1 PDS Electrodes
Palintest SoluPrep SP-B tablets
Tablet Crushing/stirring rods

The equipment and consumables required (except nitric acid, measuring cylinder, glass pipette and sample bottle) are supplied in the Palintest SA-5000 Scanning Analyzer PDSW Outfit (PT 470) and Palintest Dust Sample Preparation and Electrode Pack (PT 475)

Nitric Acid is available from laboratory chemical suppliers, see instruction SCAN.13 of the SA-5000 Handbook for further information on the use and dilution of nitric acid.

Sample Preparation

1. Pour approximately 40ml. of extraction fluid into a volumetric flask (50ml.)

2. Carefully pipette 3.75ml. of conc. Nitric acid into the volumetric flask

3. Replace the stopper and invert the flask several times to mix the acid and extraction fluid.

4. Remove the stopper and make up to 50ml. with more extraction fluid.

5. Replace the stopper and mix again by inverting several times. Pour the acidified extract into the sample bottle.

6. Take a 5ml. screw capped test tube from the Electrode Pack. Carefully pour a portion of the acidified extract into the tube filling to the 5ml. mark.

7. Add one SoluPrep SP-B tablet and crush with a new crushing/stirring rod until completely dissolved.

8. Replace the screw cap and invert the tube several times until well mixed.

9. Test the sample with the Scanning Analyzer - see Scanning Analyzer Test Procedure (SCAN.11, page 10) in the SA-5000 handbook. Use an SE-1 Electrode supplied in the SE-1 PDS Electrode Pack and choose the Dust Test from the Analysis Screen. Ensure that the correct calibration code shown on the Electrode Pack is keyed into the instrument. The Scanning Analyzer will provide the test result after a 45 second scanning period.

10. The result is displayed as ug lead/sample.

11. To calculate the concentration of lead in the TCLP extracat:

Lead in TCLP extract (ppm) - (SA-5000 reading/50)*1.081
(Note: to convert ug lead/sample to ppm divide by 50. 1.081 is a factor to correct for dilution caused by acidification).

e.g. SA-5000 reading = 235 ug lead/sample.
Lead in TCLP extract = (235/50)*1.081 = 4.7*1.081 = 5.08ppm.

Lead in TCLP extracts can be determined over the range 0.5ppm to 30ppm.