

## Palintest Ltd.

### Determination of Lead in Calcium Carbonate using the SA-5000

The Palintest SA-5000 Scanning Analyzer, and equivalent models, provides accurate and precise means of determining lead in a number of sample types. The following application note describes a method for determining the amount of lead present in a sample of Calcium Carbonate using the Dust program on the SA-5000.

#### **Test range and units**

The Dust program on the SA-5000 Scanning Analyzer gives the test results in terms of micrograms of lead per sample ( $\mu\text{g}/\text{sample}$ ). The result is used to calculate the concentration of lead present by weight in a known weight of Calcium Carbonate sample. The result is most often reported in terms of micrograms per gram ( $\mu\text{g}/\text{g}$ ) milligrams per kilogram ( $\text{mg}/\text{Kg}$ ) or parts per million (ppm). These units have the same numerical value and are equivalent to each other.

For samples having a very high level of lead, the Scanning Analyzer can be used over an extended test range. The use of this range involves dilution of the dissolved sample.

Dust regular range	Dust extended range
25 - 1500 $\mu\text{g}/\text{sample}$	125 - 7500 $\mu\text{g}/\text{sample}$

#### Equipment and Materials

Analytical Balance (not supplied)  
Sonicator tube rack  
Pipettor, 5 ml.  
Pipettor, 1ml. (Extended range only)  
Nitric Acid, 7.5%  
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) are supplied in the Palintest SA-5000 Scanning Analyzer PDSW Outfit (PT 470) and Palintest Dust Sample or Paint Sample Preparation and Electrode Pack (PT 475 or PT 474).

Nitric Acid is available from laboratory chemical supplies. See instructions SCAN.13 in the SA-5000 instruction handbook for further information on the use and dilution of nitric acid..

An analytical balance (not supplied) or other accurate type balance will be required for weighing samples for the determination of weight of lead per gram of sample.

### Sample Preparation

1. Using an accurate balance weigh 1.0 g (+/-0.1g.) of Calcium Carbonate sample into a clean dry 50ml sonicator tube. Record the weight of Calcium Carbonate.
2. Carefully add approximately 30 ml. of 7.5% Nitric Acid down the inside wall of the sonicator tube. Caution - the sample will effervesce quite vigorously. When the effervescence has ceased and all the sample has dissolved add more 7.5% Nitric Acid up to the 50 ml. mark. Cap the tube and invert several times to mix. Do not cap the tube until all effervescence has ceased.
3. Take a 5ml. screw capped test tube from the Electrode Pack. Carefully pour a portion of the sample into the tube filling to the 5 ml. mark.
4. Add one Soluprep SP-B tablet and crush with a new crushing/stirring rod until completely dissolved.
5. Replace the screw cap and invert the tube several times until well mixed.
6. 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-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 results after a 45 second scanning period.
7. The result is displayed as  $\mu\text{g. lead/sample}$ .

Example calculation:

SA-5000 reading - 95  $\mu\text{g. lead /sample}$

Weight of Calcium Carbonate - 1.05 g.

Concentration of lead in Calcium Carbonate -  $95/1.05 = 91 \mu\text{g. lead/g.}$

### 8. Extended Range Test

In cases where the lead concentration is higher than the upper limit of the Dust regular range then the sample can be analyzed using the extended range procedure. The same dissolved sample is used but a smaller sample is taken for analysis and a multiplication factor is applied to the instrument reading.

8.1 Take a clean 5 ml. screw-capped test tube. Using a pipettor add 1 ml. of the Calcium Carbonate solution from the sonicator tube.

8.2 Using a 1 ml. pipettor add 4 ml. of 7.5% Nitric Acid into the 5 ml. tube.  
(Alternatively, a variable volume precision pipettor can be used to directly dispense 4 ml. of acid into the tube). Cap the tube and shake gently to mix.

8.3 Proceed as in Step 6 above. Multiply the instrument reading by 5 to obtain the lead concentration in the original sample.

For example - instrument reading =  $750 \mu\text{g}/\text{sample}$

Extended Range result -  $750 \times 5 = 3750 \mu\text{g}/\text{sample}$

Weight of Calcium Carbonate - 1.05 g

Concentration of lead in Calcium Carbonate -  $3750/1.05 = 3571 \mu\text{g lead/g.}$