Test limit of heavy metal(calculated as Pb)

In the weak acid (pH3-4) conditions, heavy metal ions in the sample will react with hydrogen sulphide role, generating lead sulfide with black color.By comparing the colors we could judge whether the content of heavy metals exceed the requirement.

1. reagent

1.1 nitric acid.

1.2 sulphuric acid

1.3 hydrochloric acid of 6 mol/l: take 50 ml hydrochloric acid and dilute the to 100 m l.

1.4 hydrochloric acid of 1 mol/l: take 8.3 ml hydrochloric acid and dilute the to 100 m l.

1.5 ammonia water of 6mol/l: take 40 m l. ammonia and dilute to 100 m L.

1.6 ammonia water of 6mol/l: take 40 m l. ammonia and dilute to 100 m L.

1.7 acetic acid salt buffer of pH3.5:dissolve 25.0 g acetic acid soluble in 25 ml water then add 45 ml 6 mol/L hydrochloric acid, adjust the PH to 3.5 with rare hydrochloric acid or dilute aqua then dilute to 100mL.

1.8 phenolphthalein

1.9 Hydrogen sulphide saturated water: put hydrogen sulfide gas into the water without carbon dioxide until the water is saturated with hydrogen (this solution should be prepared just a few minutes before it is needed)

2.0 Lead standard solution: take 0.1598 g high purity lead nitrate and dissolve it with 10ml 1% nitric acid. Remove all the solution into a 100ml volumetric flask and dilute the solution to the volume with water. There's 1mg lead in 1ml solution. Dilute 1ml solution into 100ml with water before using so 10 μg/ml lead solution is got.

2.1 1% nitric acid: take 1 mL nitric acid and dilute it to 100 ml with water.

2. Instruments

Note all the glass instruments need to be dipped into 10%~20% nitric acid for over 24h and then washed several times with water.

50-ml colorimetric cylinder

3 Procedures

3.1 Sample treatment

wet digestion: take 5.0 g samples into 250 ml triangle flask, add 10 ml-15 mLnitrite acid to make the sample wet. Put the wet sample for a moment or overnight, then slowly heating until the reaction is not quite fierce, then cool for a while and add 5 mL sulfuric acid by making the sulfuric acid fluid down slowly alongside the flask wall. Then slowly heat the flask until the color of the solution turns to brown. Keep dropping nitrite acid until all the organic compositions decompose completely (pay attention to avoid explosion during this operation). Go on to heat until a large amount of white sulfur dioxide is generated. The final solution should be colorless or slightly yellow. After cooling add 20ml water and boil the solution until white smoke is generated. Repeat adding 20ml water and boiling it again to eliminate the remained nitrite acid. Remove the solution into volumetric flask and wash the triangle flask with water. Transfer the washed water into volumetric flask and dilute to the volume with water. Mix the solution well. Repeat the above procedure only using nitrite acid and sulfuric acid to do blank experiment against reagent.

3.2 Test

3.2.1 Tube A: Transfer 1.5ml 10μg/ml standard lead solution into 50-ml colorimetric cylinder. Dilute to 25ml with water and mix, add 1 drop phenolphthalein then the solution will become red.Adjust the PH to neutral with 6 mol/L rare hydrochloric acid or 1mol/L aqua ammonia until the red just disappear. Add 5ml,PH 3.5 acetic acid salt buffer and mix.Mark the cylinder as TubeA.

3.2.2 Tube B: take another 50-ml colorimetric cylinder and add 13ml sample solution. Dilute to 25ml with water and mix, add 1 drop phenolphthalein then the solution will become red.Adjust the PH to neutral with 6 mol/L rare hydrochloric acid or 1mol/L aqua ammonia until the red just disappear. Add 5ml, PH 3.5 acetic acid salt buffer and mix. Mark the cylinder as Tube B

3.2.3 Tube C: take another 50-ml colorimetric cylinder and add 13ml sample solution and 1.5ml 10μg/ml standard lead solution . Dilute to 25ml with water and mix, add 1 drop phenolphthalein then the solution will become red.Adjust the PH to neutral with 6 mol/L rare hydrochloric acid or 1mol/L aqua ammonia until the red just disappear. Add 5ml,PH 3.5 acetic acid salt buffer and mix. Mark the cylinder as Tube C.

3.3.4 Respectively add 10ml freshly-prepared Hydrogen sulphide saturated water into tube A, B and C and dilute to 50ml with water. Shake the cylinder to make the solution mix well then place them into a dark place and keep for 5 min. Put them out and observe the color against white background. The color of solution in Tube B should not be darker than Tube B, and color of solution Tube C should be equal or darker than Tube A.