

## 1 ANHYDROUS DIBASIC CALCIUM PHOSPHATE

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3  $\text{CaHPO}_4$  : 136.06 [7757-93-9]4 Anhydrous Dibasic Calcium Phosphate contains not less than 97.5% and not more than  
5 101.5% of  $\text{CaHPO}_4$ .6  
7 **Identification**8 (1) Dissolve 0.1 g in 10 mL of 2 mol/L hydrochloric acid TS by warming, add 2.5 mL of  
9 ammonia TS dropwise while shaking, and add 5 mL of ammonium oxalate TS: a white  
10 precipitate is produced.11 (2) Dissolve 0.1 g in 5 mL of dilute nitric acid, add 2 mL of ammonium molybdate TS  
12 after warming for 1 to 2 minutes at  $70^\circ\text{C}$ : a yellow precipitate is produced.13  
14 **Purity**15 (1) Acid-insoluble substances: Dissolve 5.0 g in 40mL of water and 10 mL of hydrochloric  
16 acid by boiling gently for 5 minutes. After cooling, collect the insoluble substances using  
17 ashless filter paper for assay. Wash with water until no more turbidity of the washing is  
18 produced when silver nitrate TS is added. Ignite to incinerate completely the residue and  
19 ashless filter paper for assay at  $600 \pm 50^\circ\text{C}$ : the weight is not more than 10mg (not more  
20 than 0.2%).21 (2) Chloride: Dissolve 0.20 g in 20mL of water and 13 mL of dilute nitric acid, by warming,  
22 if necessary, add water to make 100 mL, and filter, if necessary. Transfer 50 mL of this  
23 solution to a Nessler tube, and use as the test solution. Transfer 0.70 mL of 0.01 mol/L  
24 hydrochloric acid to another Nessler tube, add 6 mL of dilute nitric acid and water to  
25 make 50 mL, and use this solution as the control solution. Add 1mL of silver nitrate TS  
26 to the test solution and to the control solution, mix well, and allow to stand for 5 minutes  
27 protecting from direct sunlight. Compare the opalescence developed in both solutions  
28 against a black background by viewing downward or transversely. The opalescence  
29 developed in the test solution is not more than that of the control solution (not more than  
30 0.25%).31 (3) Sulfate: Dissolve 0.5 g in 5mL of water and 5mL of dilute hydrochloric acid, add water  
32 to make 100 mL, and filter, if necessary. Transfer 20 mL of this solution to a Nessler tube,  
33 add 1 mL of dilute hydrochloric acid and water to make 50 mL, and use this solution as  
34 the test solution. Transfer 1.0mL of 0.005 mol/L sulfuric acid VS to another Nessler tube,  
35 add 1mL of dilute hydrochloric acid and water to make 50 mL, and use this solution as  
36 the control solution. Add 2 mL of barium chloride TS to the test solution and to the

37 control solution, mix well, and allow to stand for 10 minutes. Compare the white  
38 turbidity produced in both solutions against a black background by viewing downward  
39 or transversely. The turbidity produced in the test solution is not thicker than that of the  
40 control solution (not more than 0.5%).

41 (4) Carbonate: Mix 1.0 g with 5 mL of carbon dioxide-free water, and add immediately  
42 2mL of hydrochloric acid: no effervescence occurs.

43 (5) Barium: Heat to boiling 0.5 g with 10mL of water, add 1mL of hydrochloric acid  
44 dropwise while stirring, and allow to cool and filter, if necessary. Add 2 mL of potassium  
45 sulfate TS to this solution, and allow to stand for 10 minutes: no turbidity appears.

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#### 47 **Loss on ignition**

48 6.5~8.5% (1 g, 800~825°C, constant weight).

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#### 50 **Assay**

51 Weigh accurately about 0.4 g, dissolve in 12 mL of dilute hydrochloric acid, by heating  
52 on a water bath, if necessary, and add water to make exactly 200 mL. Pipet 20 mL of this  
53 solution, add exactly 25 mL of 0.02 mol/L disodium ethylenediaminetetraacetate VS, 50  
54 mL of water and 5 mL of pH 10.7 ammonia-ammonium chloride buffer solution, and  
55 titrate the excess disodium ethylenediaminetetraacetate with 0.02 mol/L zinc sulfate VS  
56 (indicator: 0.025 g of eriochrom black T – sodium chloride indicator). Perform a blank  
57 determination in the same manner.

58 Each mL of 0.02 mol/L disodium ethylenediaminetetraacetate VS = 2.721 mg of  
59  $\text{CaHPO}_4$ .

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#### 61 **Reagents**

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#### 63 **Silver nitrate TS**

64 Dissolve 17.5 g of silver nitrate in water to make 1000 mL (0.1 mol/L). Preserve in light-  
65 resistant containers.

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67 **Barium chloride TS** Dissolve 12 g of barium chloride dihydrate in water to make 100  
68 mL (0.5 mol/L).