

草酸含量的测定

Method of Oxalic acid content analysis

4、1 原理 Principle:

草酸与氯化钙反应生成草酸钙沉淀，而草酸钙在酸性条件下与高锰酸钾反应生成二氧化碳，高锰酸钾被还原为二氧化锰沉淀，以消耗高锰酸钾溶液的用量计算试样中草酸的百分含量。

Oxalic acid and calcium chloride happen chemical reaction produce calcium oxalate precipitation, Calcium oxalate precipitation with potassium permanganate happen chemical reaction produce carbon dioxide under the condition of acid, and reduction of potassium permanganate for manganese dioxide precipitation. according to the amount consume of the standard solution of potassium permanganate to calculated the percentage of Oxalic acid in the sample.

5、4、2 试剂和溶液

应符合 GB/T631、HG/T2327、GB/T670、GB/T625 的要求。

Reagent and solution

Need conforming to GB/T631、HG/T2327、GB/T670、GB/T625 regulations.

5、4、3 草酸含量的测定步骤

称取 5 克试液, 称准到 0.0002 克, 置于 100 毫升烧杯中, 加 50 毫升水, 滴加 (1+1) 氨水, 调整 PH 值至 5-6, 加热至 65°C 加 15ml 10%CaCl₂ 继续煮沸, 在室温静置 120 分钟后过滤, 洗涤至不含氯离子为止。(用硝酸银检验) 将沉淀连同滤纸一起移入 250 毫升的三角瓶中, 加 50 毫升 (8+72) 硫酸溶液, 加热至 65°C, 用 0.1mol/l 高锰酸钾标准溶液滴定, 滴定至溶液呈粉红色保持 30 秒。

Weighed 5g samples(allowable 0.0002g error), put it in 100ml beaker ,add 20ml water, add (1+1) of ammonia, adjust the PH as 5-6, then heated to 65 °C and add 15ml 10%CaCl₂ continues to heated boil. Let stand at room temperature for 120minutes, then filtering until do not contain chlorine.(using silver nitrate test) put precipitate together with the filter paper into 250ml of triangle in the bottle, add 50ml(8+72) sulfuric acid solution, heated to 65 °C , use 0.1mol/l potassium permanganate standard solution titration until show pink solution to stay for 30 seconds.

4、4 草酸含量% (X) 按下式计算

Oxalic acid content%(x) calculation formula:

$$X = V \times C \times 4.502 \times 100\% / M$$

式中: V 为试样所消耗高锰酸钾标准溶液的体积

V is the volume consume of potassium permanganate standard solution

C 为高锰酸钾标准溶液的浓度

C is the concentration of potassium permanganate standard solution

M 为试样的质量

M is the weight of sample

4.502 为草酸的克当量

4.502 is the weight of oxalic acid

Allowable error:

取两次平行测定结果的算术平均值为测定结果，平行测定结果的绝对差值不大于 0.20%。

Take two parallel determination results of arithmetic mean as the determination results,the absolute difference between the parallel determination results is not more than 0.20%.