



Oznaczanie wapnia w płynach biologicznych
Determination of Calcium in Biological Fluids

MS *Spektrum*

Calcium is precipitated out of the sample with an ammonium oxalate/oxalic acid mixture, which also acts as a pH buffer. The mixture is then centrifuged and the supernatant fluid decanted off to be used for Sodium and Potassium determination if required. The precipitate is dissolved in perchloric acid and then aspirated into the calibrated BWB Flame Photometer.

Equipment Required:

1. BWB Flame Photometer.
2. Balance weighing to +/- 0.0005 g.

Reagents Preparation:

Ammonium Oxalate/Oxalic acid:

1. Dissolve 12.6 g $(\text{COOH})_2 \cdot 2\text{H}_2\text{O}$ to 1 litre distilled water in a volumetric flask. This is 0.1M oxalic acid.
2. Dissolve 12.2 g $(\text{COONH}_4)_2 \cdot \text{H}_2\text{O}$ to 1 litre of distilled water in a volumetric flask. This is 0.1M ammonium oxalate.
3. To 5 ml of 0.1M oxalic acid in a 100 ml volumetric flask add 0.1M ammonium oxalate up to the mark.

0.05 Perchloric acid:

Half fill a 1 litre volumetric flask with distilled water, add 5 ml of 60% perchloric acid and dilute to the mark.

Standard Preparation:

1. Dissolve 1.834 g $\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$ (Reagent) in 0.05M perchloric acid and dilute to 500 ml. This is the Calcium 1000 mg/l stock standard. Dilute the stock standard 1:10 with 0.05M perchloric acid. This is the Calcium 100 mg/l standard.
2. A range of standards is now prepared by successive dilution of the 100 mg/l standard with 0.05M perchloric acid.

Method:

1. Pipette 2 ml of serum into a rounded centrifuge tube and add 3 ml of Ammonium oxalate/oxalic acid mixture.
2. Mix and let stand for 30 minutes.
3. Centrifuge well and decant the supernatant fluid and drain the sample.
4. Add 4 ml of 0,05M perchloric acid to the pellet, vortex or shake vigorously to dissolve the precipitate fully. The sample is now ready for analysis.
5. Treat 2 ml of the Calcium 100 mg/l standard in the same way as the sample, i.e. Stages 1 – 4, to obtain a Blank Solution.
6. Aspirate the Blank Solution and Calcium standards into the BWB flame photometer and enter the values of standards.
7. Aspirate the sample solution into the flame photometer and read the Calcium concentration.



MS Spektrum

ul. Lubomira 4 lok. 4

04-002 Warszawa

Tel.: +48 22 810-01-28

Faks: +48 22 870-24-08

Biuro serwisowe: +48 22 402-43-04

E-mail: biuro@msspektrum.pl

www.msspektrum.pl