

论文

氯霉素含量测定法的研究 (二)快速重氮化法

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摘要:

关键词:

ESTIMATION OF CHLORAMPHENICOL BY DIAZOTIZATION METHOD

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Abstract:

Application of rapid diazotization technique to the estimation of chloramphenicol after reduction is investigated and discussed. Reduction of chloramphenicol to the corresponding amino compound was complete within 10 minutes. The rate with which nitrous acid reacts with reduced chloramphenicol was found to be slow, but it was greatly-accelerated in the presence of potassium bromide. Since nitrous acid is volatile, during titration, the temperature should be kept under 27°C. A constant volume should be maintained at the end point, excessive dilution would give high result Procedure recommended is as following: Weigh out accurately about 0.3g of sample and place it in a 250ml conical flask. 2g of zinc dust and 20ml each of water and diluted hydrochloric acid (1:1) are added. It is then heated for ten minutes over water bath at a temperature not lower than 85°C. After completion of reaction, it is cooled externally with cold water, shaking vigorously to remove gas bubbles adhered on zinc dust. Filter the content into a 300ml beaker, wash with water and combine washings to the filtrate. After the addition of 10ml of hydrochloric acid and 2g of potassium bromide, the solution is then titrated at a temperature below 27°C against 0.05M sodium nitrite solution with rapid titration technique. When the solution still gives a light blue colouration with starch-iodide indicator after 5 minutes standing, the end point is reached and the reading is taken. Each ml of 0.05M sodium nitrite solution is equivalent to 0.01616g of chloramphenicol. The results were found to be reproducible with a mean deviation of ±0.15%. The determination can be completed within 30 minutes.

Keywords:

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