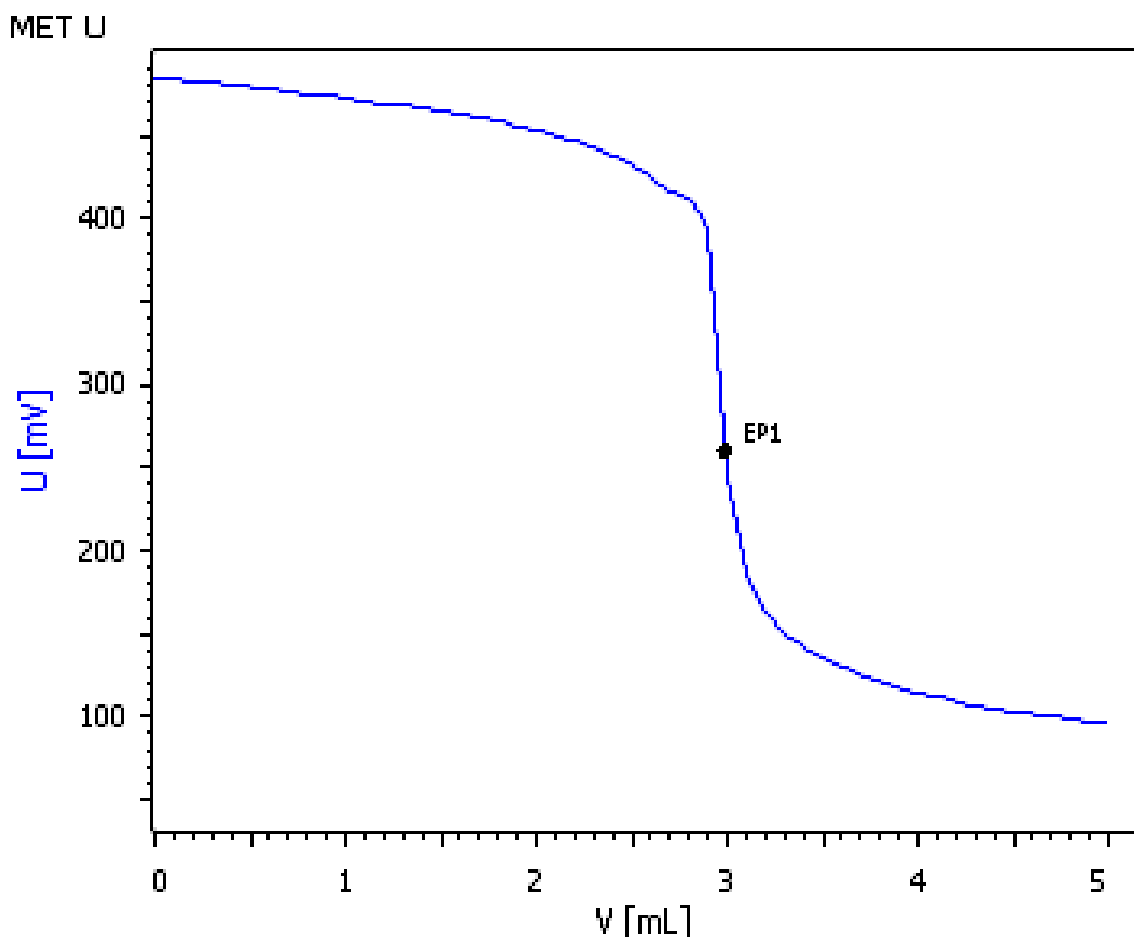


## Determination of iodometric assay according to USP<425>



Penicillin and its related antibiotics are used to prevent and treat a number of bacterial infections, such as respiratory tract infections, urinary tract infections, meningitis, etc. It may also be used to prevent group B streptococcal infection in newborns. The  $\beta$ -lactam ring of penicillin binds to the enzyme DD-transpeptidase, which prevents the formation of cross links during cell wall formation of new bacterial cell, i.e., the division of bacterial cells.

USP<425> describes a back-titration method to determine the assay of pharmacopeial penicillin antibiotic drugs and their dosage forms by iodometric titration. The method is illustrated by determining the aminopenicillin content in an ampicillin capsule.

# Method description

## Sample

Ampicillin capsules

## Sample preparation

The content of 5 ampicillin capsules (250 mg labelled) are carefully transferred into a clean 1000 mL volumetric flask. It is then dissolved and diluted up to the mark with carbon dioxide-free water (The final concentration of ampicillin will be 1.25 mg/mL).

## Configuration

905 Titrand	2.905.0010
801 Magnetic stirrer	2.801.0040
800 Dosino	2.800.0010
Combined Pt-ring electrode	6.0451.100
Electrode cable /1m/F	6.2104.020
20 mL Dosing unit	6.3032.220
Tiamo 2.5 full CD: 1 license	6.6056.252

## Solutions

Titrand	c (Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> ) = 0.01 N Around 2.6 g of sodium thiosulfate and 200 mg of sodium carbonate are weighed in a 1000 mL volumetric flask. It is dissolved and diluted up to the mark with carbon dioxide free water.
Iodine solution	c (I <sub>2</sub> ) = 0.01 N Around 1.4 g of iodine is weighed accurately and transferred into a clean 1000 mL volumetric flask. 100 mL of 3.6% potassium iodide and 3 drops of conc. HCl solution are added. The content is diluted up to the mark with carbon dioxide free water.
Hydrochloric acid solution	c (HCl) = 1.2 N 102 mL of conc. HCl is pipetted out into a clean 1000 mL volumetric flask. It is then diluted up to the mark with carbon dioxide-free water.

Sodium hydroxide c (NaOH) = 1 N  
Around 40 g of sodium hydroxide is weighed accurately and transferred into a clean 1000 mL volumetric flask. The content is then dissolved and diluted up to the mark with carbon dioxide-free water.

## Analysis

2 mL of prepared sample is taken in a 100 mL iodine flask. 2 mL of sodium hydroxide solution (1 N) is added. The content is kept in closed condition for 15 minutes. 2 mL of hydrochloric acid solution (1.2 N) and 10 mL of iodine solution (0.01 N) are added. The content is again kept in closed condition for 15 minutes. The content of iodine flask is carefully transferred into a 150 mL glass beaker with carbon dioxide-free water rinsing. It is then titrated against sodium thiosulfate (0.01 N) until the first equivalence point.

Ampicillin standard and blank analysis is also performed in the same way.

## Parameters

Titration mode	MET U
Stirring rate	8
Pause	30 s
Start condition	
Start volume	0 mL
Titration parameter	
Signal drift	50 mV/min
Min waiting time	0 s
Max waiting time	26 s
Volume increment	0.1 mL
Stop condition	
Stop volume	5 mL
Potentiometric evaluation	
EP criterion	30 mV
EP Recognition	Greatest

## Results

Ampicillin assay/(%), (n = 5)	s(rel)/%
102.68	0.07

\*USP criteria – Assay 90 to 120%.

# Method description

## Comments

Analysis is carried out in closed condition as given in general chapter.

The content after inactivation has to be completely transferred into 150 mL beaker by rinsing 2 to 3 times minimum.

Besides Ampicillin capsules, this method is also suitable for the following samples:

- Amoxicillin,
- Ampicillin sodium,
- Cloxacillin sodium,
- Cyclacillin,
- Dicloxacillin sodium,
- Methicillin sodium,
- Nafcillin sodium,
- Oxacillin sodium,
- Penicillin G potassium,
- Penicillin G sodium,
- Penicillin V potassium,
- Phenethicillin potassium.