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# Assay by Volumetric Titration

Practical 7 - Analytical practical

## Aim

To determine the content of an official compound using a suitable standardised volumetric method.

## Principle

A titration relates analyte amount to the amount of standard reagent required by a defined reaction. Reaction stoichiometry, endpoint selection, blank consumption and reagent standardisation determine the calculation. The test preparation, blank, calibration or titrant standardisation and independent controls are part of the measurement. State the actual concentration units, test conditions and adopted method before interpreting the result.

## Materials and Requirements

- Selected official compound and adopted monograph
- Appropriate standardised titrant and primary-standard record
- Endpoint indicator or potentiometric system
- Qualified balance and volumetric equipment
- Controlled method and raw-data worksheet

## Procedure

1. Identify the material, intended attribute, method version and acceptance criteria. Record preparation and instrument or reagent identities.
2. Prepare the prescribed blank and reference or standardised reagent. Verify the measurement with an independent check where applicable.
3. Prepare a representative weighed test portion in the specified solvent. Ensure the reaction conditions and dissolution are appropriate.
4. Titrate using the defined endpoint, recording initial and final burette readings and a reagent blank.
5. Repeat on independent portions and calculate the content using the reaction stoichiometry and the actual titrant concentration.
6. Record individual readings for independently prepared test portions. Keep all raw observations, including unsuccessful measurements and their investigation.
7. Calculate the result on the stated mass, volume or area basis, including dilution, blank and recovery corrections as justified.
8. Compare the evidence with the applicable criteria and write a conclusion limited to the tested preparation and conditions.

## Observations and Record

Enter actual measurements or the supplied teaching data. Record the method, material identity, conditions and units.

| Preparation / attribute | Method condition | Raw measurement | Calculated result |
|---|---|---|---|
| Titrant standardisation | Record | Record | Calculate |
| Blank | Record | Record | Calculate |
| Test portion 1 | Record | Record | Calculate |

| Preparation / attribute | Method condition | Raw measurement | Calculated result |
|---|---|---|---|
| Test portion 2 | Record | Record | Calculate |

## Calculations

• Analyte amount (mol) = blank-corrected titrant volume (L) x actual titrant molarity x stoichiometric ratio
• Assay (%) = measured analyte mass / weighed preparation mass x 100

### Worked Example

Illustrative 1:1 reaction: corrected titrant volume 10.00 mL, molarity 0.1000 mol/L and analyte molar mass 180.16 g/mol.

Measured analyte = 0.01000 x 0.1000 x 180.16 = 0.18016 g.

For a 0.1820 g test portion, assay = 98.99% approximately. This example does not assign the reaction to a particular official drug.

## Result and Interpretation

• State the endpoint and stoichiometric basis, including whether water or other correction factors are required by the monograph.
• Report replicate variability and the evidence that the method can measure the intended attribute in this matrix.

## Precautions

• Use the actual standardised reagent concentration.
• Avoid endpoint overshoot and parallax.
• Select a blank with the same reagent and solvent conditions.
• Use the adopted laboratory procedure, suitable controls and the designated waste arrangements.

## Viva Questions and Answers

### Why standardise a titrant?

Its actual concentration can differ from the nominal preparation.

### Why is stoichiometry essential?

The reagent-to-analyte mole relationship determines the mass calculation.

### Why correct for the blank?

Other components may consume titrant.

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