

Saccharin in nickel bath

Function: Differential Pulse Voltammetry (DPV/a)

| | |
|-------------------------|-------|
| Start Potential (mV) | -900 |
| End Potential (mV) | -1300 |
| Current range | 102.4 |
| Scan Speed (mV/s) | 30 |
| Number of cycles | 3 |
| Delay before sweep (s) | 5 |
| Purge and stir time (s) | 300 |
| Stirring speed (rpm) | 300 |
| Drop Size (a.u.) | 60 |

Saccharin concentrated standard solution (1 g/l) – Working standard solution

Dissolve 100 mg of pure saccharin in 100 ml of 95° ethanol in a volumetric flask.

Supporting electrolyte

0.1 M HCl

Dilute 0.8 ml of 37% HCl in 100 ml of distilled water.

Procedure

Sample treatment

Pour 10 ml of sample in a 100 ml separatory funnel. Add 1 ml of 37% HCl and 5 ml of methanol. Extract for 4 times with 10 ml of ethyl ether. Collect all the ether extracts and dry in a rotating evaporator. Dry 10 minutes at 110°C in a oven. Add 10 ml of 0.1 M HCl to the residue.

Voltammetric analysis

Pour 10 ml of 0.1 M HCl in the cell, add 50-300 µl of the above sample solution.

Warnings

It is necessary to subtract the blank curve before the calculation of the peak height (point to point blank subtraction function).

Analytical Report

Analysis: Nickel bath

Sample Concentration = 0.65 g/l

Method: 5 additions

Blank: point to point subtraction

Volumes Table

| | |
|-----------------|-------------|
| Solvent Volume | 0 (ml) |
| Supporting Sol. | 10 (ml) |
| Sample Volume | 0.3 (ml) |
| Standard Conc. | 1000 (mg/l) |

Height Table

| # | Peak Pot. | Height |
|---|-----------|---------------------|
| 0 | -1066.5 | 16.70 μA |
| 1 | -1066.5 | 21.04 μA |
| 2 | -1067.4 | 25.04 μA |
| 3 | -1068.8 | 29.09 μA |
| 4 | -1069.7 | 32.94 μA |
| 5 | -1072 | 37.37 μA |

Regression Data

| # | Add Conc. | Height x dilution |
|---|-----------|---------------------|
| 0 | 0 mg/l | 573.5 μA |
| 1 | 167 " | 726.2 μA |
| 2 | 333 " | 868.1 μA |
| 3 | 500 " | 1.013 mA |
| 4 | 667 " | 1.153 mA |
| 5 | 833 " | 1.314 mA |

$$y = ax + b$$

$$a = 879.3 \text{ nA} \cdot \text{l/mg}$$

$$b = 575.0 \mu\text{A}$$

$$r^2 = .9996$$

