

## Titrimetric analysis of chloride

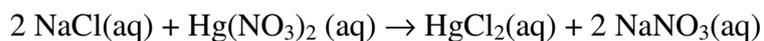
### 1 Scope

The mass concentration of sodium chloride in the sample solution has to be determined. The task consists of two parts:

1. Standardization of mercury(II) nitrate solution.
2. Determination of chloride concentration in the sample solution.

### 2 Principle

The sample is titrated with standardized  $\text{Hg}(\text{NO}_3)_2$  solution using diphenylcarbazone as indicator. The reaction can be described by the following equation:



### 3 Equipment

#### 3.1 Analytical balance

Precision balance

#### 3.2 Laboratory glassware

-volumetric flasks	500 mL	1 piece,
	250 mL,	1 piece,
- beaker	100 mL;	1 pieces;
	250 mL	2 piece,
	600 mL	2 piece
- conical flask, wide neck,	500 mL ,	3 piece
- transfer pipette	50 mL,	1 piece;
	25 mL	1 piece
- burette	50 mL,	1 piece,
- measuring cylinder	250 mL,	1 piece,
- Pasteur pipette,		1 piece,
- wash bottle,		1 piece
- funnel		1 piece.

#### General equipment

- ring stand with burette clamp,
- pipette filler,
- weighing spoon

### 3.3 Materials and their safety codes

Name	. CAS No.	R/S code
Nitric acid, concentrated $\text{HNO}_3$ , 68% $M_r$ 63,01	7697-37-2	R: 34 S: 23-26-36-45
Mercury(II) nitrate monohydrate $\text{Hg}(\text{NO}_3)_2 \cdot \text{H}_2\text{O}$ p.a. $M_r = 342,62$	7783-34-8	R:26/27/28-33-50-53 S: 13-28-45-60-61
Sodium chloride NaCl p.a. $M_r(\text{NaCl}) = 58,44$ $A_r(\text{Cl}) = 35,45$	7647-14-5	
Indicator: Bromophenolblue	115-39-9	S: 22-24/25
Indicator Diphenylcarbazone	538-62-5	

### 3.4 Reagents solutions

All reagent solutions, except mercury(II) nitrate are ready to use.

REAGENT	LOCATION
Solution of mercury(II) nitrate (see 4.1.1).	
Sodium chloride solution with exact concentration $c(\text{NaCl}) = 0,1000 \text{ mol L}^{-1}$ .	On the shelf above the bench
Nitric acid solution. $c(\text{HNO}_3) = 2 \text{ mol L}^{-1}$ .	On the shelf above the bench
Nitric acid concentrated $w = 68 \%$	
<i>Indicators</i>	
Bromphenolblue solution: $1 \text{ g L}^{-1}$ in ethanol $\phi(\text{ethanol}) = 95 \%$ .	On the shelf above the bench
Diphenylcarbazone solution: $5 \text{ g L}^{-1}$ in ethanol.	On the shelf above the bench
<i>Analysis sample</i>	
Sodium chloride solution	On the shelf above the bench

## 4 Procedure

### 4.1 Preparation and standardization of mercury(II) nitrate solution.

#### 4.1.1 Preparation:

Prepare 250 mL of mercury(II) nitrate solution with concentration  $0,05 \text{ mol L}^{-1}$ .

The solute is mercury(II) nitrate monohydrate.

Note: if the solute does not dissolve, add fewdrops of concentrated nitric acid and mix well.

#### 4.1.2 Standardization of mercury(II) nitrate solution.

Using 25,00 mL-pipette transfer 25,00 mL of standard solution of sodium chloride into a 500 mL-conical flask. Add 175 mL of distilled water by means of measuring cylinder. Then add 3 drops of indicator bromophenolblue and enough nitric acid ( $2 \text{ mol L}^{-1}$ ), use Pasteur pipette, to change the colour of the indicator from blue to yellow and 3 drops in excess. Then add 1 Pasteur pipette full of diphenilcarbazone solution.

Titrate this solution with prepared solution of mercury(II) nitrate, until the colour changes from yellow to intensive violet.

Record the data in an appropriate box on the answer sheet.

#### 4.1.3 *Blank sample.*

Using a measuring cylinder put 200 mL of distilled water in a 500 mL conical flask. Then add 3 drops of indicator bromophenolblue and enough nitric acid ( $2 \text{ mol L}^{-1}$ ), use Pasteur pipette, to change the colour of the indicator from blue to yellow and 3 drops in excess. Then add 1 Pasteur pipette full of diphenilcarbazone solution.

Titrate this solution with prepared solution of mercury(II) nitrate, until the colour changes from yellow to intensive violet.

Record the data in an appropriate box on the answer sheet.

#### 4.2 *Analysis of sample.*

Transfer 50,00 mL of sample solution of sodium chloride into a 500 mL volumetric flask and dilute it to the mark. Pipet 25,00 mL of this solution to a 500 mL conical flask and add 175 mL of distilled water .

Then add 3 drops of indicator bromophenolblue and enough nitric acid ( $2 \text{ mol L}^{-1}$ ), use Pasteur pipette, to change the colour of the indicator from blue to yellow and 3 drops in excess. Then add 1 Pasteur pipette full of diphenilcarbazone solution.

Titrate this solution with prepared solution of mercury(II) nitrate, until the colour changes from yellow to intensive violet.

Record the data in an appropriate box on the answer sheet.

## 5 Calculations

Calculate:

- Exact concentration of mercury(II) nitrate solution in  $\text{mol L}^{-1}$ ,
- Mass concentration of chloride ions in the sample solution in  $\text{g L}^{-1}$
- relative deviation in percent (RD %).

$$RD(\%) = \frac{\overline{\Delta x_i}}{\bar{x}} \quad \overline{\Delta x_i} = \frac{\Delta x_1 + \Delta x_2 + \dots}{N} \quad \Delta x_1 = x_1 - \bar{x} \quad \bar{x} = \frac{x_1 + x_2 + \dots}{N}$$

where  $x_1, x_2$  etc are individual measured values, N is the number of measurements.

Note: When calculating the result of the analysis the volume of mercury(II) nitrate solution for blank sample should be taken into account.