Determination of composition of Elixirum Thymi Compositum

The product is an anticough medicine for children. It contains among other minor additives ethanol, sugar and sodium bromide. The task is the quantitative determination of its ethanol, sacharose and sodium bromide content.

I. Determination of sacharose content

1. Task

The iodometric determination of sacharose content of Elixirum Thymi Compositum by the Schoorl-method.

2. Principle

Reducting sugars reduce the copper(II)-ions into copper(I)-ions, and the quantity of the remaining copper(II)-ions can be determined iodometrically.

3. Reagents

3.1. 0.1 mol/dm^3 HCl solution (ready for use)

3.2. Fehling-I-solution (69,28 g $CuSO_4 \cdot 5\ H_2O$ dissolved to 1 litre with distilled water)

3.3. Fehling-II-solution (346 g K-Na-tartarate and 100 g NaOH dissolved to 1 litre with distilled water) (ready for use)

3.4. 20 % HCl (ready for use)

3.5. 20 % sulphuric acide (ready for use)

3.6. 0.1 mol/dm^3 $Na_2S_2O_3$ solution (ready for use)

3.7. 0.1000/12 (= 0.008333) mol/dm^3 $KH(IO_3)_2$ solution (ready for use)

3.8. 1 % starch solution (ready for use)

3.9. KI

The chemicals, solutions are on the laboratory bench. The 0.1 molar $Na_2S_2O_3$ solution and the 0.1000/12 molar $KH(IO_3)_2$ solution are on the benches at the end of the laboratory. All the solutions necessary are ready for use. Some of the solutions are in refill burettes.

Remarks to units

The unit mol/dm^3 is equal to mole/litre 1 mol/ dm^3 is called in this text 1 molar solution and is denoted 1 M (not corresponding to the SI system and the IUPAC recommendations). Note that 0,1000/12 = 0.008333.

Safety, danger and hazards recommendations

Some chemicals are highly dangerous. Read the R/S codes of chemicals before working with them. Use eye-protecting glass or safety mask if necessary. One can use also pipette filler or Griffin balloon for pipette the dangerous solutions.

4. Procedure

4.1. Determination of the exact concentration of the 0.1 molar $Na_2S_2O_3$ solution

Pipette 10 cm^3 0.1000/12 molar $KH(IO_3)_2$ solution into 3 pcs. of 200 cm^3 conical (Erlenmeyer) flasks with ground glass plug, add approx. 0.3 g KI, dilute with 30 cm^3

distilled water, rinse well the grindings and 1 cm^3 20 % HCl solution to it. Close the flask firmly with the plug, shake well and titrate after 2 minutes waiting by the thiosulphate solution of unknown exact concentration. The advance of the titration is indicated by the disappearance of the colour of iodine. When the solution is already pale yellow add 8-10 drops of starch solution and continue the titration until the blue colour disappears. The solution in the closed flask should not be return into blue colour over 1 minute.

(It is advisable to perform this step during the waste time of inverting the sugar.)

4.2. Inverting of sacharose

Pipette 1.2 cm^3 (approx. 1.3 g) Elixirum Thymi Compositum into a 100 cm^3 conical (Erlenmeyer) flask with ground glass plug by an 5 cm^3 syringe. Measure its mass by analytical balance. Add 40 cm^3 distilled water, 10 cm^3 0.1 molar HCl-solution and some boiling chips into to it. Put a small funnel in the neck. Bring it to boil, and continue boiling the solution with small flame during half an hour. Cool down with tap water, filter the solution with filter-paper and prepare 100 cm^3 stock solution from it.

4.3. Determination of sugar content.

Measure 10 cm^3 Fehling-I-solution and 10 cm^3 Fehling-II-solution into a 200 cm^3 conical (Erlenmeyer) flask with ground glass plug than 10 cm^3 solution to be analysed (stock solution) dilute it with 20 cm^3 distilled water. Add some boiling chips, put small funnel into the neck. Bring the solution to boil by bigger flame, than boil it for 6 minutes by small flame. After having cooled down the solution to room temperature by tap water add approx. 3 g KI and 20 cm^3 20 % H_2SO_4 to the mixture. Rinse the grinding well, close the flask and stand it for 10 minutes. Titrate the arisen iodine by the $Na_2S_2O_3$ solution till pale yellow. Add 2 cm^3 starch solution to it and titrate further until the disappearance of the blue colour. One can convince of the end of titration by adding some further drops of starch solution.

Determine the copper content of the Fehling-I-solution similarly (blank test). Use for this step only 4 cm^3 both from Fehling-I and Fehling-II solutions.

If necessary the 100 cm^3 conical (Erlenmeyer) flask can also be used in step 4.3. step of the sugar analysis.

5. Calculations

5.1. Determination of the exact concentration of the $Na_2S_2O_3$ solution

$$C = \frac{V_{KH(IO_3)_2} \cdot C_{KH(IO_3)_2} \cdot 12}{V_{Na_2S_2O_3}}$$

where

C is the exact concentration of $Na_2S_2O_3$ solution (in mol/dm^3)

$V_{KH(IO3)2}$ is the volume of 0.1000/12 M $KH(IO_3)_2$ solution (in cm^3)

$C_{KH(IO3)2}$ is the concentration of 0.1000/12 M $KH(IO_3)_2$ solution (in mol/dm^3)

$V_{Na2S2O3}$ is the consumption of $Na_2S_2O_3$ solution (in cm^3)

5.2. Determination of saccharose content

$$N = \frac{(2.5V_2 - V_1) \cdot C}{0.1}$$

where

N is the volume of 0.1 M of $Na_2S_2O_3$ solution equivalent with the sacharose content of the analysed 10.00 cm^3 solution (in cm^3).

V_1 is the mean value of consumption of $Na_2S_2O_3$ solution for Elixirum Thymi Compositum containing samples (in cm^3)

V_2 is the mean value of consumption of $Na_2S_2O_3$ solution for bank test (in cm^3)

The sacharose content of the analysed 10.00 cm^3 solution (in mg) can be obtained from the empirical Schoorl table. One can use linear interpolation if necessary.

N in cm^3	Sacharose content in mg	N in cm^3	Sacharose content in mg
1	3.1	11	35.0
2	6.2	12	38.3
3	9.3	13	41.6
4	12.4	14	44.9
5	15.6	15	48.2
6	18.8	16	51.4
7	22.0	17	54.6
8	25.2	18	57.8
9	28.4	19	61.0
10	31.7	20	64.3

5.3. Sacharose content of the Elixirum Thymi Compositum in mass fraction

Calculate the sacharose content of the stock solution, then calculate the sacharose content of the Elixirum Thymi Compositum in mass fraction

$$w \text{ (in ‰)} = \frac{\text{g saccharose}}{1000 \text{ g Elixirum Thymi Compositum}}$$

II. Determination of the ethanol content

1. Task

The chromatometric determination of ethanol content of Elixirum Thymi Compositum.

2. Principle

The $K_2Cr_2O_7$ oxidises in strongly acetic medium ethanol into acetic acid. The surplus of the oxidant can be determined by $Fe(NH_4)_2(SO_4)_2$ solution (Mohr salt solution)

3. Reagents

3.1. 0.4000/6 (= 0.06667) mol/dm^3 $K_2Cr_2O_7$ solution, highly acidic with sulphuric acid (ready for use)

3.2. 0.2 mol/dm^3 $Fe(NH_4)_2(SO_4)_2$ solution, highly acidic with sulphuric acid (ready for use)

3.3. concentrated sulphuric acid

The chemicals, solutions are on the laboratory bench. All the solutions necessary are ready for use. The concentrated sulphuric acid is in the hood at the end of the laboratory.

Remarks to units

The unit mol/dm^3 is equal to mole/litre 1 mol/ dm^3 is called in this text 1 molar solution and is denoted 1 M (not corresponding to the SI system and the IUPAC recommendations). Note that 0.4000/6 = 0.06667.

Safety, danger and hazards recommendations

Some chemicals (especially the concentrated sulphuric acid) are highly dangerous. Read the R/S codes of chemicals before working with them. Use eye-protecting glass or safety mask if necessary. One can use also pipette filler or Griffin balloon for pipette the dangerous solutions.

4. Procedure

Pipette 8-9 drops of Elixirum Thymi Compositum into a 100 cm^3 conical (Erlenmeyer) flask with ground glass plug with medical syringe. Measure its mass by analytical balance. Add immediately 15 cm^3 distilled water to it to avoid the evaporation of the ethanol. Assemble the small analytical distilling apparatus, pour the sample quantitatively into the distilling flask by means of small funnel. The total volume of the solution in the flask should be approx. 50 cm^3. Add boiling chips and insert the condenser. Pour into an 200 cm^3 conical (Erlenmeyer) flask with ground glass plug 40 cm^3 distilled water, use this flask as receiver. The end of the waste pipe of the condenser should be below the water level in the receiver. Boil the solution uniformly with small flame. Take care that the apparatus does not draw back. Distil approx. the half of the solution. At the end of the distillation remove carefully the receiver under continuos heating.

Add very carefully (!) 15 cm^3 concentrated sulphuric acid to the distilled mixture by means of graduated cylinder. The solution warms up, cool it down to tap water temperature. Add 10 cm^3 0.4000/6 molar $K_2Cr_2O_7$ solution to it, stop the flask by its wet plug.

After 1 hour add 2 drops Ferroin indicator and titrate the solution with 0.2 molar Mohr-salt solution. At the equivalence point the colour changes from green (caused by Cr^{3+}-ions) into brown.

Carry out the same measurement also without ethanol (blank test). Use 60 cm^3 distilled water, 15 cm^3 concentrated sulphuric acid, 5 cm^3 0.4000/6 molar $K_2Cr_2O_7$ solution and 2 drops of Ferroin indicator. This solution can be titrated by 0.2 molar Mohr-salt solution without staying.

5. Calculations

5.1. Determination of ethanol content

$$m = \frac{0.2\,(2V_1 - V_2)\,M}{4}$$

where

m is mass of ethanol in the sample in mg

V_1 is the mean value of the consumption of the Mohr-salt solution at the blanck test (in cm^3)

V_2 is the consumption of the Mohr-salt solution at the sample (in cm^3)

M is the molar mass of ethanol (46,07 g/mol)

5.2. Ethanol content of the Elixirum Thymi Compositum in mass fraction

$$w\ (\text{in } ‰) = \frac{\text{g ethanol}}{1000\ \text{g Elixirum Thymi Compositum}}$$

III. Determination of the sodium bromide content

1. Task

The argentometric determination of the sodium bromide content of Elixirum Thymi Compositum.

2. Principle

The argentometric titration can be carried out by potentiometric method with bromide selective electrode.

3. Reagents

3.1. Na-acetate buffer solution (0,28 % Na-acetate and 1% acetic acid)
3.2. 0.1000 mol/dm^3 $AgNO_3$ solution
3.3. 1 mol/dm^3 KNO_3 solution

The chemicals, solutions are on the laboratory bench. All the solutions necessary are ready for use. Some of the solutions are in refill burettes.

Remarks to units

The unit mol/dm^3 is equal to mol/litre 1 mol/ dm^3 is called in this text 1 molar solution and is denoted 1 M (not corresponding to the SI system and the IUPAC recommendations).

Safety, danger and hazards recommendations

Some chemicals are highly dangerous. Read the R/S codes of chemicals before working with them. Use eye-protecting glass or safety mask if necessary. One can use also pipette filler or Griffin balloon for pipette the dangerous solutions.

4. Procedure

Measure 3.0-3.5 cm^3 of Elixirum Thymi Compositum into 150 cm^3 beaker by syringe, close tightly by Parafilm elastic sheet. Measure its mass by analytical balance. Add 20 cm^3 Na-acetate/acetic acid buffer to it, dilute with distilled water up to a suitable volume, so that the bromide selective electrode be immersed in the solution. Place the stirrer rod in the beaker, place the it on magnetic stirrer. Fasten onto the saturated calomel reference electrode the separator tube (with glass fritt), fill into it with 1 molar KNO_3 solution approx. to half of height. Fasten the reference electrode into the beaker. Start stirring with suitable speed.

Put in operation the already warmed up pH-mV-meter.

Fill the burette with the $AgNO_3$ solution, and start the titration. In course of the first (rough) titration add the titrating solution in 1 cm^3 portions, read the electromotive force in mV. After having finished the titration switch the MEASURE knob of the instrument into

0 position, lift both electrodes out, wipe the precipitate by tissue paper and rinse thoroughly. Determine the range of the equivalence (the big potential change).

Repeat the titration. Add the titrating $AgNO_3$ solution in the range of the equivalence in 0.1 cm^3 portions. Between titrations control the solution level in the separating tube.

5. Calculations

5.1. Graphic evaluation of the titration measurement

Plot the potential (in mV) against the titrating volume (in cm^3) for each measurement. Determine the equivalence point (point of inflexion of the curve).

5.2. Determination of bromide content

Calculate the sodium bromide content of the measured sample

$$m = V \cdot C \cdot M$$

where

m is the mass of sodium bromide in mg

V is the consumption of the $AgNO_3$ solution (in cm^3)

C is the concentration of the $AgNO_3$ solution (in mol/dm^3)

M is the molar mass of sodium bromide (102.9 g/mol)

5.3. Sodium bromide content of the Elixirum Thymi Compositum in mass fraction

$$w \text{ (in ‰)} = \frac{\text{g NaBr}}{1000 \text{ g Elixirum Thymi Compositum}}$$

IV. Summary of the analysis of Elixirum Thymi Compositum

Give the summary of the results in form of a table

Component	w (in ‰) mean value
Sacharose	
Ethanol	
sodium bromide	