

The calculation of the substance quantitative content, %, was carried out according to the formula:

$$X, \text{ assay} = \frac{V \text{ titrant} \cdot K \cdot T \cdot 100}{m}$$

Conclusions. The synthesis was performed and the methodology for quality control of promising substance of 3-methylaminosuccinoylamido-N-(3',4'-dimethylphenyl)anthranilic acid, which has high anti-inflammatory, analgesic, diuretic and antifungal effects, was developed. The statistical processing of the obtained results was carried out.

ELECTROCHEMICAL METHOD FOR DETERMINATION OF CYCLAMATE USING 12-MOLYBDOPHOSPHORIC ACID

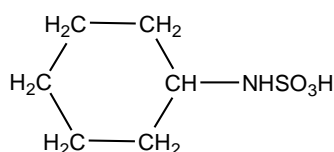
Kushko R.A., Pereverzeva A.S.

Scientific supervisor: associate prof. Olexiy Brizitskiy

National University of Pharmacy, Kharkiv, Ukraine

alexchebryz@gmail.com

Introduction. Cyclamate – low-calorie sweetener is sweeter than sucrose 30 times. Used in food and beverages and the manufacture of diabetic products. Unlike other sweeteners it can be used in the manufacture of food products, requiring additional heat treatment due to resistance to light, temperature and acidity. The structural formula of sweetener cyclamate (cyclohexylamine - N - sulfonic acid) is as follows:



There are many methods for quantitative determination of cyclamate: colorimetry using pikryl chloride, chromatographic, spectrophotometric with the formation of N,N-dichlorocyclohexylamine, indirect spectrophotometric determination of cyclamate by its degradation products capillary isotachopheresis and others. Chromatographic methods of analysis based on the separation of cyclamate mixture of sweeteners and food additives, followed by determination of individual components using a UV detector and spectrophotometric determination sweetener based on his previous destruction to N,N-dichlorocyclohexylamine. Then carry out specific reactions resulting product with various reagents – resulting in formation of colored complexes that determine photometrically. However, existing methods are complex performance multistage sample preparation and toxic reagents used in the analysis.

Aim. The development of alternative methods for the quantitative determination of cyclamate in substance and industrial production is relevant analytical problem. Newly analytical methods shall have a sufficiently metrological parameters and allow cyclamate determine the presence of auxiliary components without their prior separation.

Materials and methods. Plastifying PVC membrane for ICE synthesized by a known method. As the previously used electrode-active component (EAC) soluble in water ($2 \cdot 10^{-6}$ g/l) and soluble in

solvents, plasticizers membrane (10-12 g/l) deposit product structure interaction heteropoly anions Keggin's and cationic complex with barium particles.

For the synthesis of membrane ion-selective electrode are 0.4500 g of polyvinyl chloride and mix it with 4.5 ml of cyclohexanone (solvent PVC) at 35-40°C and with continuous stirring using a magnetic stirrer. Separately, 1.1 ml of membrane solvent-plasticizer (DBP) was added 0.01 g EAC and stirred to a homogeneous state. The resulting solution mixed with heated after complete dissolution mixing it transferred into a Petri dish with a diameter of 55 mm and kept in a fume hood until complete evaporation of the solvent (cyclohexanone). The result is a homogeneous elastic film containing components in mass %: EAC – 0.62; dibutyl phthalate (DBP) – 71.42; PVC – 27.96. By the same method plasticized membranes were synthesized using as solvent – dioctylphthalate plasticizer (DOP) and tricresyl phosphate (TCP). Before using ISE soaked in solutions of cationic complex with barium particles formed destruction cyclamate, with a concentration corresponding to mid-range content detectable substance. For electrode performance using an electrochemical range (chain): Ag|AgCl, KCl (sat.) | solution substance (10^{-4} M) | membrane ISE || sample solution | KCl (sat.), AgCl|Ag.

Due to the impossibility of obtaining the necessary associates through direct deposition anion sweetener typical analytical reagents, such as heteropolyanions Keggin's structure was investigated reaction of complete and partial decomposition cyclamate in an acidic environment. As we know from published data, the process is complete destruction of cyclamate by the following reaction (Fig. 1):

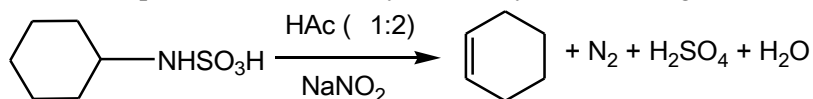


Fig. 1. The scheme complete destruction of chemical cyclamate

The reaction occurs in a strongly acid medium (pH 1-2) in the presence of 0.1 M solution of sodium nitrite. In the reaction of free nitrogen is released and produced cyclohexene and sulfate ion. However, as seen from the reaction equation, with the resulting products only barium sulfate can be used as an electrode-active component (EAC) for the synthesis of PVC film membranes ISE, which could indirectly determine cyclamate products for the destruction of the latter. The analysis of scientific data indicates that administration of barium sulfate phase in PVC membrane electrode film is difficult due to its low solubility in typical solvents by ionometry. Therefore, in this case, the use of solvent-plasticizer esters of phthalic acid are not, and specific organic reagents such as potassium tetrakis(4-chlorophenyl)borate, derivatives of bis-(1,2,3,6 – tetrahydrobenzo) – crown ethers. al., which reduces the availability of this method. In addition, techniques developed using a similar ISE have low sensitivity (10^{-3} M), which limits their use.

Partial chemical destruction cyclamate spend on softer terms, compared with the full destruction. So, to create an acidic environment using acetic acid concentration of 1:10, in contrast to the complete destruction of the chemical, which used acetic acid concentration of 1:2. This acidity of the medium after the destruction of pH 4-5. Under such conditions the degradation product is cyclohexensulfamine cyclamate, which is further treated with barium salt, resulting in a cationic complex with barium share that the interaction with an excess of 12-molybdophosphoric heteropoly acid forms a soluble compound with lithium-associative nature of the interaction macroions. Methods of UV and IR spectroscopy was confirmed speculation that a weakly acidic medium partial degradation product cyclamate in the presence of doubly charged metal ions in solution exists in the form of stable cationic complex particle that is able to form soluble ionic associates with heteropoly anion $\text{PMo}_{12}\text{O}_{40}^{3-}$. As the ion metal was used Ba^{2+} , however, given the ability to form chelate complexes with product partial destruction cyclamate, promising also be used cations Mg^{2+} and Ca^{2+} .

Results and discussion. The resulting sparingly soluble compounds meet the requirements imposed on the electrode-active agents (of sustainability, low water solubility, high solubility in organic solvents) and thus can be used as an EAC for the development of ion-selective electrodes, working to product degradation cyclamate. It was established that the dependence of electromotive force vs the logarithm of the concentration of cationic complex in the range $1 \cdot 10^{-5} - 1 \cdot 10^{-2}$ mol / l and linear expressed by the equation:

$$E = a + b \lg C$$

The possibility of quantitative determination of the cyclamate in the food additive E-952 and other industrial products using the developed ISE is shown. The method meets all the requirements of modern analysis – simple, safe and inexpensive, sufficient precise, sensitive and selective. Sensor response time doesn't exceed 50 s and membrane life (35-55 days) allow to spend analysis without replacement. By means of sensor it is possible to determine Cyclamate in solutions containing 10^{-5} – 10^{-2} mol/l. RSD is less than 2.3%. $(\bar{X} - \mu) 100\%/\mu < \text{RSD}$.

Conclusions. Procedure for quantitative determination of the Cyclamate (E-952) by a direct ionometric method with ion-selective electrode (ISE) that is reversible to the product of Cyclamate – cationic complex of cyclohexene sulfamic acid with barium ions has been developed. Polyvinylchloride membrane ionic associate of cyclohexene sulfamic acid with barium ions and 12-molybdophosphoric acid proposed. It has been experimentally studied the influence of various factors on the characteristics of electrode. It was the influence of various factors on the characteristics of the developed electrode:

- pH test solution;
- nature of the solvent-plasticizer for the membrane;
- EAC content in the membrane.

ANALYSIS OF UKRAINIAN SUNFLOWER OIL QUALITY PARAMETERS IN ACCORDANCE WITH THE REQUIREMENTS OF THE EUROPEAN PHARMACOPOEIA

Onyshchenko O. O., Savchenko L. P., Petrushova L. O.

Scientific supervisor: Assoc. prof. Alexeeva T. V.

National University of Pharmacy, Kharkiv, Ukraine

onishenko199783@gmail.com

Introduction. According to WHO, musculoskeletal disorders as the cause of disability and mortality are ranked 4th in the world after cardiovascular, cancer and diabetes. In the nearest future experts predict an epidemic of osteoporosis, indicating the aging of the planet's population. According to statistics data, every fifth inhabitant of the globe suffers from back pain, while the proportion of osteochondrosis is up to 90 %. Chronic diseases of the musculoskeletal system are also one of the most common problems in Ukraine, and about 3.5 million people are encountered a problem of locomotor apparatus functional impairment. Its serious complications require continuous multi-year therapy. Treatment of osteochondrosis should be individual, taking into account the phase, pathogenetic features of the disease and the psychological component of the pain syndrome. The main method of osteochondrosis diseases treatment is the use of nonsteroidal anti-inflammatory drugs in various pharmaceutical forms, which also have a pronounced analgesic and anti-inflammatory effect. It is known that the patient's needs can not always be met with the help of industrial medicines. In this case, medicines prepared in the pharmacy, with necessary dosing of active pharmaceutical ingredients or in the adapted to each particular situation form, come to the aid. One of the main components of such medicines is sunflower oil. The problem for the compounding pharmacy today is the lack of the article in the State Pharmacopoeia of Ukraine (SPhU) that regulates its quality.

Aim. The purpose of the study was to determine the quality parameters of sunflower oil which can be used for preparing of extemporal dosage forms for external use. It was done for estimation of its quality correspondence to the European Pharmacopoeia (EurPh) article «Sunflower oil, refined» requirements and assessing the possibility of this article introducing into the SPhU.

Materials and methods. The quality parameters of three sunflower oils samples «Schedryiy dar» (Щедрий Дар), «Tsarska» (Царська), «Schebpak» (Щебпак) were analyzed during the research. Analysis was done by the EurPh article «Sunflower oil, refined» requirements. HPTLC-Plate Nano-Sil C₁₈-100/UV₂₅₄ (10*10 cm), 20×20 cm twin trough glass chamber, CAMAG Linomat 5 sample applicator and CAMAG TLC Visualizer 2 were used for the fatty oils identification.

Results and discussion. All quality parameters except composition of fatty acids were determined during the research. Appearance and solubility of the oils: clear, light yellow liquid,