QUANTITATIVE DETERMINATION OF ANISE OIL IN AMMONIA-ANISE DROPS BY THE METHOD OF PEROXY ACIDOMETRY

Blazheyevskiy M. Ye., Agafonov O. M.
Scientific supervisor: prof. Blazheyevskiy M. Ye
National University of Pharmacy, Kharkiv, Ukraine
Blazheyevski@ukr.net

Introduction. Ammonia spirit-anise spirit drops (*lat.* Spiritus ammoniacatus anisatus, ATC code R05C A10) - combined medication with expectorant and anti-inflammatory action. 100 ml of drops contain anise oil 2.81 g, ammonia solution 15 ml the rest – auxiliary substances ethyl alcohol 90%. Anise oil stimulates the secretion of bronchial glands, ammonia contributes to the release of sputum and its easy release. In addition, anise oil helps digestion and has a laxative and antiseptic effect. Narrow-anise drops are used in the complex treatment of respiratory diseases: pharyngitis, tracheitis, bronchitis (acute and chronic), bronchopneumonia, bronchiectasis, pertussis in children. As a result of treatment, the digestion improves in the patients, the secretory and motor functions of the stomach and intestine are normalized, and flatulence disappears.

Epoxidation as alkenes using peroxy acids is one of the most fundamental reactions in chemistry, yet there are very few examples illustrate this important reaction in quantitative analysis. For the quantitative determination of the prevailing components of the essential oil, we used an epoxidation reaction with the use as oxidizing agent of the higher aliphatic peroxy acid – peroxycapric acid (C_{10}) in a medium of methylene chloride at the room temperature.

Aim. To develope of a simple method for quantitative determination of anise oil in ammonium anise drop by means of peroxy acid oxidation.

Materials and methods. Peroxydecanoic acid (peroxycapric acid) was obtained by D. Swern method. Method of iodometric titration. The standard solution is 0.0200 M sodium thiosulfate. Microburette for 10 ml. Aqueous solution of acetic acid (1:1). An aqueous solution of 5% KI. Methylene chloride, grade p.a. The object of the study was "Ammonia-anise drops" oral drops, solution in 25 ml bottles produced by "Ternopharm" (Ukraine, Ternopil). In 100 ml drops - anise oil 2.81 g, 10% ammonia solution 15 ml. 90% ethanol - 100 ml as excipient.

Results and discussion. It was found that using only a slight excess of peroxycaprinic acid maximized conversion of anethole to epoxide anethole. One mole peroxide is consumed per mole of anethole, that is, the reaction is quantitative (Fig.).

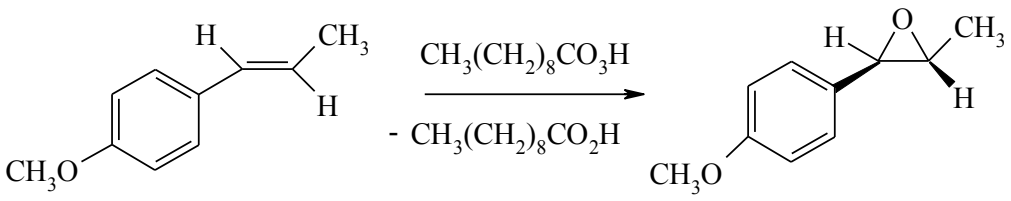

Fig. A scheme for the epoxidation reaction of *p*-Methoxy-*trans*-β-methylstyrene (*trans*-Anethole) by peroxydecanoic acid (peroxycapric acid)

Procedure of analysis. A mixture a preparation (5,0 mL) and sulfate acid diluted (10 mL) and saturated solution NaCl (10 mL) was stirred well (5 min) and leave to stand to layer separation. To the mixture was added 20 mL CH_2Cl_2 (2×10 mL) and resulting mixture was stirred for an additional 5 min and left to separation of layers. The organic layer was separated and washed with 10% aqueous Na_2CO_3 (1×25 mL) and dried (10 mg Na_2SO_4). A solution of peroxy capric acid (1.0 g/25) in CH_2Cl_2 (5.0 mL) was added drop wise. After the addition was complete, CH_2Cl_2 is added until the total volume of solution is 25.0 mL. The mixture is allowed to stand for 40-45 minutes until the reaction is complete at room temperature. An aliquot solution volume (1.00 mL) is titrated after preliminary addition of 1 ml of 5% KI and 2 mL of CH_3CO_2H (1:1) with a standard 0.0200 mol/L solution of $Na_2S_2O_3$ using microburette (*V*). The control experience is similarly carried out: To 20.0 ml of CH_2Cl_2 is added 5.0 ml of a solution of peroxycapric acid, thoroughly mixed and an aliquot of the resulting solution (1.00 mL) is titrated as before (V_0). The content of anise oil is determined from the difference in the volume of the titrant consumed in the control and working experiments, respectively (V_0 -*V*), using a titer found in a separate special experiment with an exact sample of the anise oil (0.1405 g / 5.00 mL). For n=7; P=0.95 RSD < 1.2 % (δ<RSD).

The verification of the correctness was carried out by comparing the results obtained with those of an independent (reference) GLC method using standard samples of individual basic components of anise oil and pharmacopoeial standard volumetry method of oil. The content of the main components relative to each other was established by the normalization method.

Conclusions. A simple method of analysis anise oil has been developed. The proposed method may be extended for the analysis of medicinal forms with anise oil.