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## Determination of the Anorexigenic Drug Sibutramine in Biologically Active Dietary Supplements

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### 1. Introduction

Overweight is currently a common problem in modern society. People from all over the world are in search of the easiest and fastest, as well as the most effective ways to lose weight [1]. To solve this problem, people turn to various fitness routines, but most people resort to taking anorexigenic biologically active dietary supplements, as well as medications. The most effective drug on the pharmaceutical market is sibutramine. It is included in the list of potent and toxic substances and is prescription-only, which makes it difficult to buy sibutramine in a pharmacy; therefore, people often prefer herbal supplements. Sibutramine has been banned in the United States, as well as the European Union, since 2010 following the decision of the Food and Drug Administration (FDA). However, some unscrupulous manufacturers add sibutramine to anorexigenic dietary supplements to increase their effectiveness [2]. Thus there is a need to develop analytical methods for quality control of food additives, to detect cases of fraud on the pharmaceutical market in a timely manner.

### 2. Materials and methods

The test objects used in the study were: a standard sample of sibutramine hydrochloride and biologically active dietary supplements.

Reagents: acetonitrile (PanReac AppliChem, Germany); methanol (J.T. Baker, Poland); ammonium formate (Honeywell, Germany); Milli-Q water.

Equipment: Agilent 1100 high-performance liquid chromatograph (Agilent Technologies Inc., USA) equipped with a diode-matrix detector; Thermo Scientific Ultimate 3000 high performance

liquid chromatograph with a TSQ Quantum Access Max Mass Spectrometer.

The conditions of chromatographic separation were determined by the selection method during the experiment. Sibutramine was determined by HPLC with a diode array spectrophotometric detector using a MACHERRY-NAGEL NUCLEOSIL 5  $\mu$ m C18 150x4.6 mm chromatographic column. The drug was extracted with methanol. A 30:70 mixture of an aqueous solution of 50 mmol ammonium formate (eluent A) and acetonitrile (eluent B) was used as a mobile phase in isocratic elution mode at a rate of 1.0 ml/min. Detection was carried out at an analytical wavelength of 225 nm.

The determination of sibutramine by HPLC-MS was carried out using the chromatographic column described previously, but via gradient elution of a mixture of an aqueous solution of 10 mmol ammonium formate (eluent A) and acetonitrile (eluent B) in gradient mode of elution at a rate of 0.5 ml/min.

### 3. Results

Based on the existing methods for determining sibutramine in biologically active dietary supplements [3,4], we have developed a method for detecting sibutramine in food additives using HPLC-DMD, followed by confirmation by HPLC-MS.

In the indicated range, the value of the relative error of determination did not exceed 1.5% (0.90% for the HPLC-MS method, 1.10% for the HPLC-DAD method). The confidence interval ( $\pm$ ) is  $100.19\% \pm 1.10\%$  according to HPLC-DAD and  $99.89 \pm 0.75\%$  according to HPLC-MS. This method meets the requirements of linearity, since the resulting correlation coefficient ( $r$ ) for the HPLC-MS method was 0.9999, and 0.9988 for HPLC-DAD.

#### 4. Conclusions

The developed analytical methods make it possible to determine illegally added sibutramine in biologically active dietary supplements of anorexiogenic effect. These methods can be used in quality control of biologically active additives to ensure the safety of public health.

#### References

1. *World health statistics 2018: monitoring health for the SDGs, sustainable development goals. World health organization, 2018: 18–20.*
2. *Rocha T, Amaral J. S, Beatriz M. P.P. Oliveira. Adulteration of Dietary Supplements by the Illegal Addition of Synthetic Drugs: A Review // Comprehensive Reviews in Food Science and Food Safety. 2016. - № 15. - P. 43 – 62.*
3. *Huang Z., Xiao S., Kuo D., Chen B., Yao S. Simultaneous determination of sibutramine and N-Di-desmethyl-sibutramine in dietary supplements for weight control by HPLC-ESI-MS. J. Chromatogr. Sci. 2008. 46: 707-711.*
4. *Kilicarslan G., Imamoglu E., Kucuk A., Ozdemir A. UV spectrophotometric, derivative spectrophotometric and RP-HPLC-DAD determination of sibutramine. Rev. Anal. Chem. 2011. 29: 169-196.*