

DEVELOPMENT OF A REFERENCE SAMPLE FOR RAPID ANALYSIS OF AN ELEMENTAL COMPOSITION OF MEDICINAL PLANT RAW MATERIALS

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ABSTRACT

Objective: Development and validation of a technique for preparation of a reference sample for elemental microanalysis using the XRF technique in terms of repeatability, reproducibility, and optimization of the technique for rapid determination of the elemental composition of medicinal plants based on X-ray fluorescence analysis.

Methods: Samples: fresh shoots of *Kalanchoe daigremontiana*, ready reference sample "Birch Leaf" LB-1 (A. P. Vinogradov Institute of Geochemistry, Siberian Branch of the Russian Academy of Sciences, Irkutsk, Russia); and IAEA reference sample SRM 2976 (IAEA, MEL, Monaco). The dispersed fraction was analyzed using a Master Sizer 2000 instrument (Malvern Panalytical, Worcestershire, UK). Elemental analysis using an energy dispersive X-ray fluorescence spectrometer EDX-7000 Shimadzu (Shimadzu Corporation, Kyoto, Japan), GZ-AAS using an Agilent instrument, model 240Z AA instrument (Agilent Technologies, Inc., Santa Clara, USA) with electrothermal atomization and Zeeman background correction, and ICP-MS using an Agilent 7500 CE instrument (Agilent Technologies, Inc., Santa Clara, USA).

Results: By the LALLS method, they were separated by the maximum distribution, which was 63 microns, and a minor fraction of 39 microns. This indicates sufficient homogeneity in the sample. Further, homogeneity was proved by the XRF method by measuring six independent samples obtained by the quartering method. Also, the elemental composition of the reference samples was determined: completely dried, homogenized before sifting, and homogenized after sifting. Further, the obtained reference sample of *K. daigremontiana* was compared with reference samples: IAEA SRM 2976 and "birch leaf" methods: ICP-MS, GZ-AAS, XRF.

Conclusion: The reference sample will allow for rapid analysis of medicinal plant raw materials. Standardization of medicinal plants by the content of microelements will allow observing species differences as well as adjusting the concentrations of microelements for therapeutic purposes using medicinal plants.

Keywords: Reference sample, Trace element analysis, XRF, X-ray fluorescence analysis, Medicinal plants

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INTRODUCTION

Medicinal plants are an extremely advantageous source of both microelements (trace elements) and biologically active substances, precursors for the synthesis of modified ligands, as well as biologically active nanoparticles, the production of which often results in significant costs. Trace elements play a vital role in a variety of biological effects of medicinal plants, and, therefore, they determine not only the therapeutic effect of a particular medicinal plant but also its toxic properties. The biological value of some elements (zinc, iron, copper, chromium, and cobalt) is well known, which allows for establishing their irreplaceability. The toxic properties of these elements appear only at high concentrations, while metals such as cadmium, lead, and mercury do not participate in biochemical reactions and are extremely toxic. The use of medicinal plants in the treatment of many diseases is progressing everywhere due to the breadth of the therapeutic range and the low incidence of side effects. Also, medicinal plants are used for the manufacture of various delivery systems for medicinal substances, for example, nanoparticles based on various forms of metal [1-3].

Medicinal plant raw materials of inadequate quality can pose a significant danger to human health. In this regard, it is extremely important to ensure quality control of medicinal plants to protect patients from toxic microelements. Plants are susceptible to pollution due to unfavorable environmental conditions. Most often, contamination occurs during cultivation and processing [4, 5].

Since the production of medicinal plants is large-scale and control measurements are continuous, the technique necessary for quality control must meet the following criteria: a) rapidity of the measurement itself; b) ease of sample preparation; c) low qualification requirements for operators of hardware and software systems; d)

sample preparation, as well as the measurement process itself, should not lead to a change in the sample matrix, all the more so to its destruction. The most popular methods in elemental microanalysis can be divided into three groups: non-destructive (neutron activation analysis, XRF) methods with plasma atomization (ICP-MS, ICP-OES), atomic absorption spectroscopy with electrothermal atomization, and Zeeman background correction (GZ-AAS), which is the most sensitive method. Unlike destructive methods, XRF can be used for simultaneous multielement flow determination and allows in situ analysis of samples. Reducing the number of steps in the analytical sequence will result in a decrease in the level of possible contamination and a decrease in the consumption of expensive reagents. Despite the obvious advantages, the main drawback of this technique is the presence of the complex organic matrix effect, which is the influence of a dielectrically inhomogeneous organic microenvironment on the intensity of X-ray fluorescence. To minimize this effect, a certain sample preparation process was used in this study, designed to universalize the organic matrix, as well as a comparison with other techniques of elemental analysis with subsequent correction of values taking into account variations in the results obtained by XRF. Consequently, XRF is dependent on the classical GZ-AAS and ICP-MS techniques, on the basis of which reference samples with a similar matrix are developed to perform calibration. The complexity and multistage nature of the sample preparation process and high requirements for personnel qualifications in techniques such as GZ-AAS and ICP-MS significantly increase the cost of measurements, especially continuous measurements [6].

One way to solve this problem is to use software that uses constants of fundamental parameters in combination with influence coefficients. However, standardless techniques are limited in their application due to their low accuracy [7].

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Table of Contents Manual For Elemental Analysis In Plant Materials

1. Understanding the eBook Manual For Elemental Analysis In Plant Materials
 - The Rise of Digital Reading Manual For Elemental Analysis In Plant Materials
 - Advantages of eBooks Over Traditional Books
2. Identifying Manual For Elemental Analysis In Plant Materials
 - Exploring Different Genres
 - Considering Fiction vs. Non-Fiction
 - Determining Your Reading Goals
3. Choosing the Right eBook Platform
 - Popular eBook Platforms
 - Features to Look for in an Manual For Elemental Analysis In Plant Materials
 - User-Friendly Interface
4. Exploring eBook Recommendations from Manual For Elemental Analysis In Plant Materials
 - Personalized Recommendations
 - Manual For Elemental Analysis In Plant Materials User Reviews and Ratings
 - Manual For Elemental Analysis In Plant Materials and Bestseller Lists

5. Accessing Manual For Elemental Analysis In Plant Materials Free and Paid eBooks
 - Manual For Elemental Analysis In Plant Materials Public Domain eBooks
 - Manual For Elemental Analysis In Plant Materials eBook Subscription Services
 - Manual For Elemental Analysis In Plant Materials Budget-Friendly Options
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 - Manual For Elemental Analysis In Plant Materials Enhanced eBook Features
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 - Adjustable Fonts and Text Sizes of Manual For Elemental Analysis In Plant Materials
 - Highlighting and Note-Taking Manual For Elemental Analysis In Plant Materials
 - Interactive Elements Manual For Elemental Analysis In Plant Materials
8. Staying Engaged with Manual For Elemental Analysis In Plant Materials
 - Joining Online Reading Communities
 - Participating in Virtual Book Clubs
 - Following Authors and Publishers Manual For Elemental Analysis In Plant Materials
9. Balancing eBooks and Physical Books Manual For Elemental Analysis In Plant Materials
 - Benefits of a Digital Library
 - Creating a Diverse Reading Collection Manual For Elemental Analysis In Plant Materials
10. Overcoming Reading Challenges
 - Dealing with Digital Eye Strain
 - Minimizing Distractions
 - Managing Screen Time
11. Cultivating a Reading Routine Manual For Elemental Analysis In Plant Materials
 - Setting Reading Goals Manual For Elemental Analysis In Plant Materials
 - Carving Out Dedicated Reading Time
12. Sourcing Reliable Information of Manual For Elemental Analysis In Plant Materials
 - Fact-Checking eBook Content of Manual For Elemental Analysis In Plant Materials
 - Distinguishing Credible Sources
13. Promoting Lifelong Learning

- Utilizing eBooks for Skill Development
- Exploring Educational eBooks

14. Embracing eBook Trends

- Integration of Multimedia Elements
- Interactive and Gamified eBooks

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