

New Technique of Preparation of Standard Gaseous Mixtures Needed for Validation of Analytical Procedures Used in Environmental Studies

M. Słomińska¹, P. Konieczka and J. Namieśnik

¹ Department of Analytical Chemistry, Chemical Faculty, Gdansk University of Technology (GUT) 11/12 Narutowicza Street, 80-233 Gdansk, Poland

Abstract. Standard gas mixtures are indispensable reference materials for ensuring the reliability of analytical work. Paper presents one novel approach in the field of production of gaseous reference materials based on the application of thermal decomposition process of proper surface compound for obtaining the desired gaseous analyte. The main fields of application of reference materials are presented. The field of application of thermal decomposition process among static and dynamic techniques available for preparation of standard gas mixtures is described.

Keywords: Thermal decomposition, standard gas mixtures, gaseous reference materials, surface compounds.

1. Introduction

Protection of environment, proper management of its resources and progress of environmental risks depend upon the quality of analytical measurements.

In order to carry out tasks in this field and to ensure the reliability of analytical measurement results, two basic tools are required:

- control equipment and devices according for the analysis of properly prepared samples,
- appropriate analytical procedures for determination of wide range of xenobiotics in different environmental samples;
- reference materials with different metrological characteristic necessary to ensure the proper functioning of quality assurance and quality control system.

According to the definition reference material is a material sufficiently homogenous and stable with reference to specified properties which have been established as fit for its intended use in measurement or in examination of nominal properties [1].

Reference materials play a very important role in the quality control and quality assurance system. They are commonly used for validation of analytical procedures, interlaboratory comparisons, in estimating of the uncertainty of a measurements and documenting traceability [2].

Generally reference materials are classified due to:

- properties (chemical composition, biological and clinical properties, engineering properties),
- chemical nature (single major constituent, matrix types),
- traceability (0 primary class, I class, II class, etc.),
- uncertainty of determination of analyte concentration,
- field of application.

Taking into account the matrix's type, reference materials are classified as solid, liquid and gaseous reference materials (standard gas mixtures).

2. Production of Reference Materials

Production of reference materials is very expansive, labour-intensive and time-consuming process. Fig. 1 presents the general steps of procedure of preparation reference.

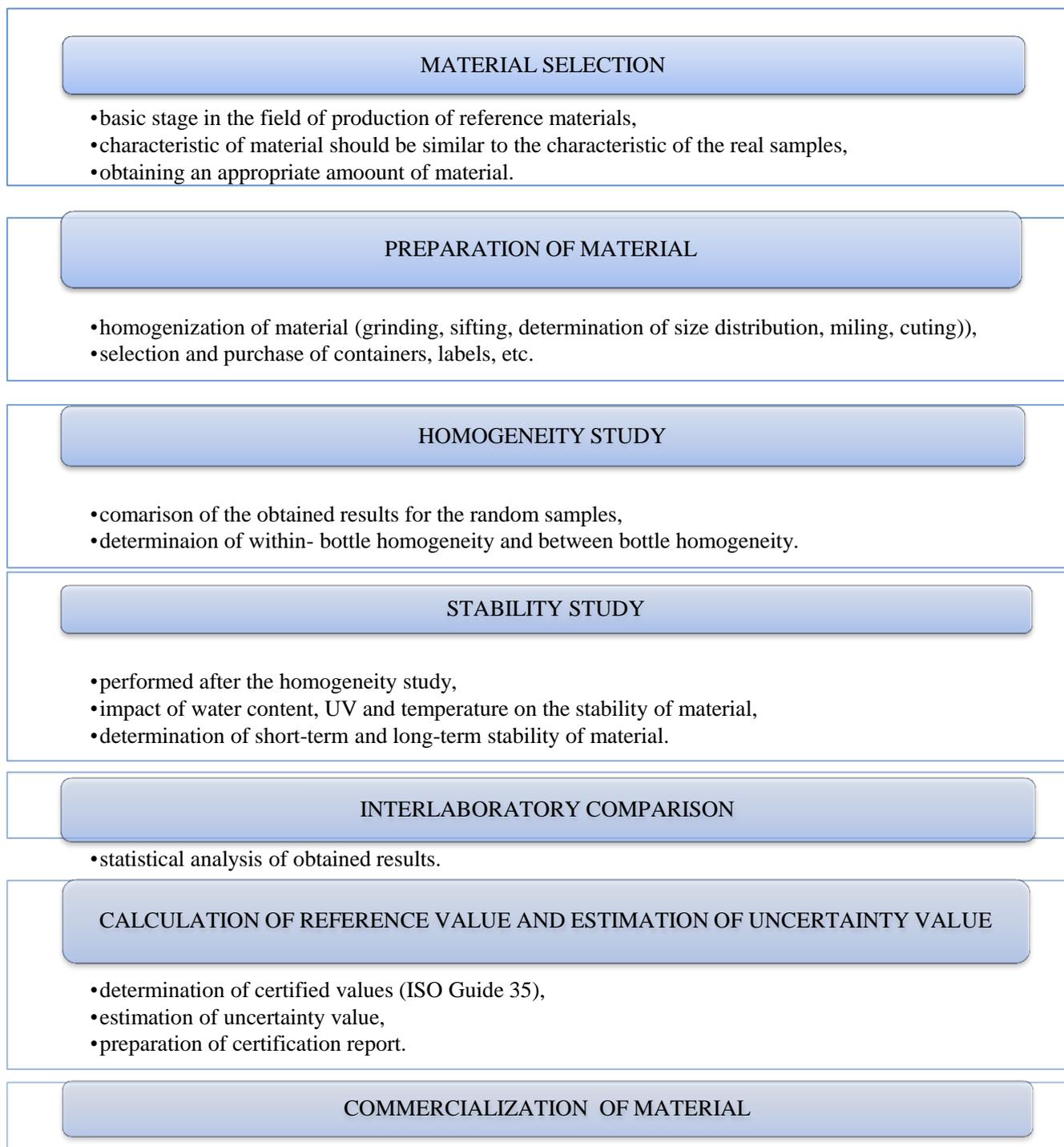


Fig. 1: General procedure for preparing reference materials [3].

Seen from the standpoint of the technology of manufacturing liquid and solid reference materials, the preparation of standard gas mixtures is very much more difficult. The degree of this difficulty varies inversely with the change in analyte concentration: the lower the concentration of analytes in a standard gas mixtures, the more difficult it is to produce such a mixture.

3. Thermal Decomposition Process as an Alternative for Production of Standard Gas Mixtures

The use of standards with matrix compositions and analyte concentrations resembling those of environmental samples enhances the reliability of analytical results [2]. The increasingly frequent analysis of representative samples of various gaseous media is stimulating demand for new techniques of preparing standard gas mixtures, which in certain cases act as matrix-free reference materials [4].

The field of standard gas mixtures has for a number of years now witnessed rapid advances in methodological approaches for producing matrix-free reference materials. Among the available static and dynamic techniques for preparation of standard gas mixtures, the thermal decomposition of a surface compound has recently gained in popularity [5]-[14].

At the Department of Analytical Chemistry of The Gdansk University of Technology research on the application of thermal decomposition techniques for obtaining standard gas mixtures has been in progress for a good 15 years. The “milestones” in the development of thermal decomposition of surface compounds for the preparation of standard gas mixtures are presented in the Fig. 2.

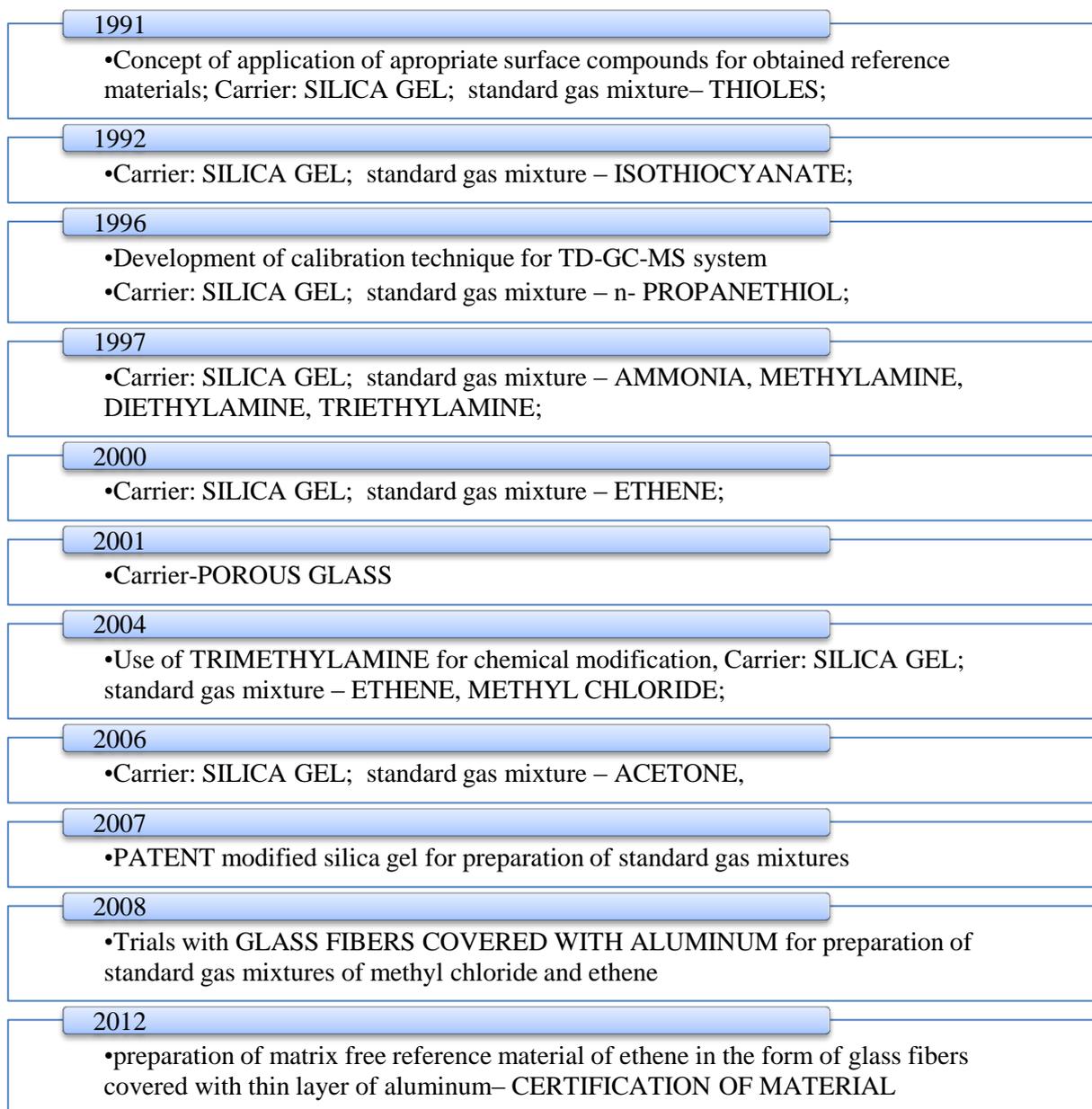


Fig. 2: Milestones in the preparation of standard gas mixtures using thermal decomposition of surface compounds.

The starting point for the studies are conducted from many years research on the production of new reference materials of gaseous substances, which are focused on:

- chemical modification of solid materials which are characterized by properly developed the active surface in order to obtain the desired surface compounds;
- searching for optimal conditions for the thermal decomposition of surface compounds in order to obtain well-defined quantities of the desired analytes;
- searching for quick and convenient calibration techniques.

The use of the thermal decomposition of surface compounds technique permits to eliminate the errors connected with determination of the concentration of the gaseous standard mixture. The chemical modification applied (determines the degree of surface coverage of the carrier) and the temperature of thermal decomposition of surface compound allow to define explicitly the concentration of analytes in the mixture, on the basis of the length or mass of the carrier in use [7]. Characteristics of the technique, its possibilities and limitations are presented in the Figure 3.

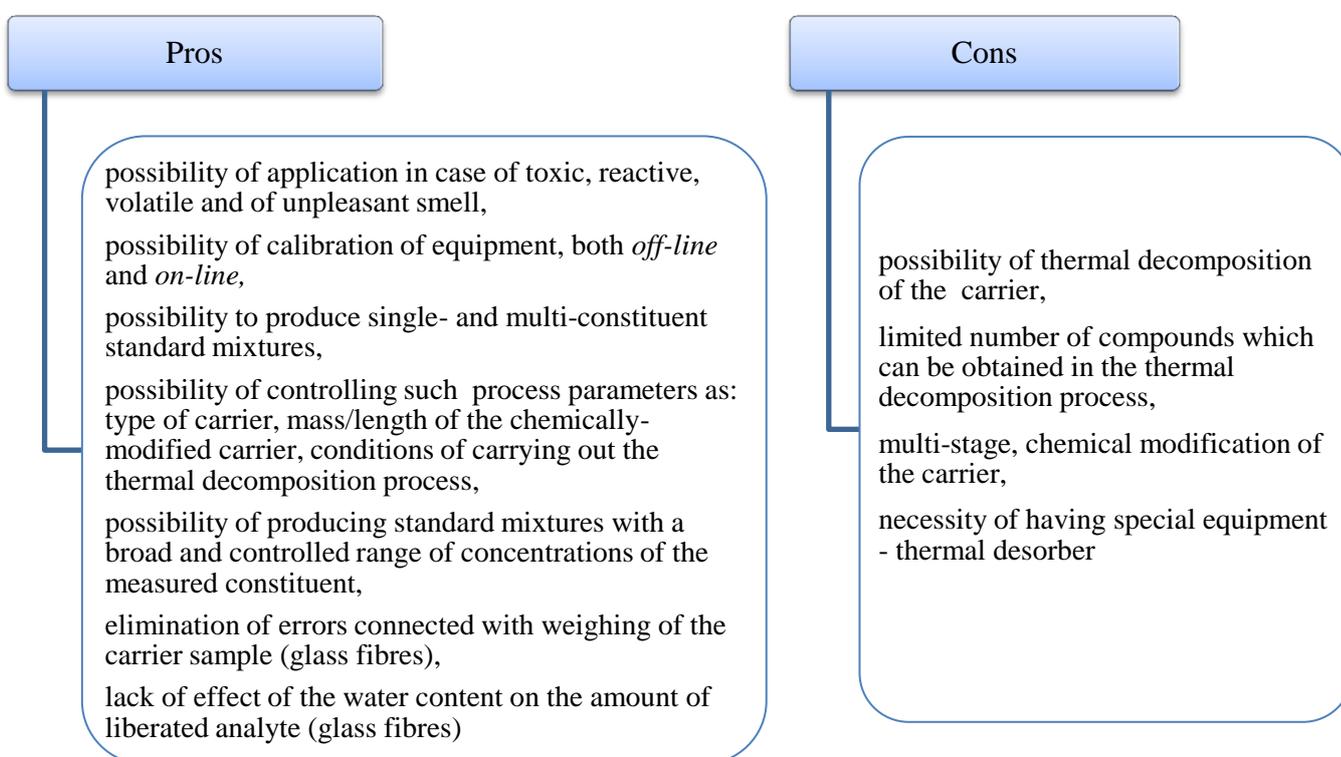


Fig. 3: Advantages and disadvantages of using thermal decomposition process in the preparation of standard gas mixtures.

4. Summary

Reference materials are an excellent tool for the quality control and assurance of analytical results. Standard gas mixtures are unique among reference materials. The rapid progress of SGM production is connected with the instability of gaseous analytes which make them difficult to obtain [6]. Thermal decomposition of surface compounds gives possibilities to [11]-[14]:

- simplify the calibration stage of apparatus,
- shorten the time of analysis,
- eliminate sources of error,
- obtain higher level of precision and accuracy of the analysis.

Moreover, the production of non-matrix reference materials in the form of glass fibers or silica gel covered with an appropriate surface compound provides a useful tool for routine screening in daily laboratory practice.

5. Acknowledgments

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6. References

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