
**Nanotechnologies — Characterization
of volatile components in single-
wall carbon nanotube samples
using evolved gas analysis/gas
chromatograph-mass spectrometry**

*Nanotechnologies — Caractérisation des composés volatils dans les
nanotubes de carbone à simple paroi (SWCNT) utilisant l'analyse
des gaz émis par chromatographie en phase gazeuse couplée à la
spectrométrie de masse*





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Contents

Page

Foreword	iv
1 Scope	1
2 Normative references	1
3 Terms and definitions	1
4 Principle	2
5 Apparatus	2
6 Sample preparation	4
7 Measurement procedures for EGA/MS and EGA/GCMS	4
7.1 General.....	4
7.2 Measurement procedure of EGA/MS.....	4
7.3 Measurement procedure of EGA/GCMS.....	4
8 Data analysis and interpretations of results	5
8.1 Qualitative analysis.....	5
8.2 Mass loss analysis.....	5
9 Accuracy and uncertainties	5
10 Test report	5
Annex A (informative) Case study	7
Bibliography	12

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 229, *Nanotechnologies*.

This second edition cancels and replaces the first edition (ISO/TS 11251:2010), which has been technically revised.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Nanotechnologies — Characterization of volatile components in single-wall carbon nanotube samples using evolved gas analysis/gas chromatograph-mass spectrometry

1 Scope

This document specifies a method for the characterization of evolved gas components in single-wall carbon nanotube (SWCNT) samples using evolved gas analysis/gas chromatograph mass spectrometry (EGA/GCMS).

NOTE Some difference could appear between qualitative and quantitative results of emitted gas and gas content in the sample due to the heating and the possible presence of catalysts.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO/TS 80004-3, *Nanotechnologies — Vocabulary — Part 3: Carbon nano-objects*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO/TS 80004-3 and the following apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

- ISO Online browsing platform: available at <https://www.iso.org/obp>
- IEC Electropedia: available at <http://www.electropedia.org/>

3.1 single-wall carbon nanotube SWCNT

carbon nanotube consisting of cylindrical graphene layer

3.2 evolved gas analysis EGA

technique in which the nature and/or amount of evolved gas product(s) released by a sample subjected to a controlled temperature program is(are) determined

Note 1 to entry: The method of analysis should always be clearly stated (Reference [1] in the Bibliography).

3.3 EGA/MS evolved gas analysis/mass spectrometry

technique using mass spectrometry to analyse gaseous components evolved from a sample as a function of temperature

Note 1 to entry: Although the gases evolved at any particular temperature are detected simultaneously, it might not be possible to uniquely identify the different components using MS alone.

3.4

EGA/GCMS

evolved gas analysis/gas chromatography coupled to mass spectrometry

technique combining a gas chromatograph and a mass spectrometer to identify the chemical composition of gases evolved from a sample as a function of temperature

Note 1 to entry: The evolved gases are passed through a gas chromatograph (GC) to separate each component so that it can be identified in the MS unit.

3.5

volatile compounds

compounds that are evolved from a sample at the temperature under consideration

4 Principle

EGA/MS and EGA/GCMS are used to characterize evolved gas impurities in samples of SWCNT. Evolved gas compounds are identified by measuring the mass spectra of the gaseous component evolved from the heated samples in a furnace or other suitable heating device, such as that used for programmed temperature pyrolysis or thermogravimetric analysis. EGA/MS is used to determine the temperature range over which the release of evolved gas components occurs. EGA/GCMS analysis is used to identify each component separately by the use of a GC capillary column. Quantitative information can additionally be obtained by the sample mass loss in thermogravimetric analysis (TGA) and the peak area in EGA/MS.

NOTE Some details of the technique are described in References [2] to [6] in the Bibliography. EGA/GCMS plays a complementary role to TGA, which is mainly devoted to quantifying the mass of the evolved gas components.

5 Apparatus

[Figure 1](#) shows a schematic diagram of EGA/MS which is composed of a furnace, a heating unit without a GC capillary column and a MS unit. In the EGA/MS, evolved gas from the furnace is led to the MS unit directly through a capillary tube without a separation process.

[Figure 2](#) shows a schematic diagram of an EGA/GCMS which is composed of a furnace, a GC with a capillary column and an MS unit. In the EGA/GCMS, all compounds evolved from the sample within the furnace are captured by the cooling trap and are then introduced to the GC capillary column unit by heating the trap. The compounds are separated by the column in the GC unit.