
**Powder metallurgy — Hot isostatic
pressing — Argon detection using
gas chromatography and mass
spectrometry techniques**

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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 119 *Powder metallurgy*, Subcommittee SC 3, *Sampling and testing methods for sintered metal materials (excluding hardmetals)*.

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.

Introduction

It is essential to detect argon in powder metallurgy hot isostatic pressing (PM HIP) material in order to ensure the desired performance of the PM HIP component. Argon from the production of powder can remain in the powder grains. Argon from the powder filling processes can remain in the voids between powder grains and become trapped during consolidation. Can imperfections can result in ingress of argon from the HIP chamber and potentially introduce, or increase the level of, argon.

Argon in the resulting metal powder produced component can be detected using the techniques included in this document

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Powder metallurgy — Hot isostatic pressing — Argon detection using gas chromatography and mass spectrometry techniques

IMPORTANT — It is the responsibility of the purchaser of the PM HIP service to specify in the purchase order if an argon detection test is needed. If so, the agreed argon limit shall be specified.

1 Scope

This document specifies a gas chromatography and a mass spectrometry method of detecting the presence of argon in metal powder produced components, consolidated by hot isostatic pressing.

This document specifies the calibration and functionality test for the equipment covered. It also specifies methods for sampling, sample preparation and sample test procedure of PM HIP components to detect argon presence.

Components produced by additive manufacturing are not covered in this document.

2 Normative references

There are no normative references in this document.

3 Terms and definitions

For the purposes of this document, the following terms and definitions 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 <https://www.electropedia.org/>

3.1

blank test

test performed without sample in the same manner as, and parallel with, a test using an analytical sample

[SOURCE: ISO 11323:2010, 8.13]

3.2

calibration

operation that, under specified conditions, in a first step, establishes a relation between the quantity values with measurement uncertainties provided by measurement standards and corresponding indications with associated measurement uncertainties and, in a second step, uses this information to establish a relation for obtaining a measurement result from an indication

Note 1 to entry: A calibration may be expressed by a statement, calibration function, calibration diagram, calibration curve, or calibration table. In some cases, it may consist of an additive or multiplicative correction of the indication with associated measurement uncertainty.

Note 2 to entry: Calibration should not be confused with adjustment of a measuring system, often mistakenly called “self-calibration”, nor with verification of calibration

Note 3 to entry: Often, the first step alone in the above definition is perceived as being calibration.

[SOURCE: ISO/IEC Guide 99:2007, 2.39]

3.3

can capsule canister

container used to encapsulate the powder during the pressure consolidation process

Note 1 to entry: It is partly or fully removed from the final part.

3.4

detection limit

smallest actual amount of an analyte that can be detected by a measuring method

[SOURCE: ISO 20553:2006, 3.14, modified — "measurand" has been replaced by "analyte".]

3.5

functionality test

assessment of the performance of a measuring system, based on specific parameters

[SOURCE: ISO/TS 14907-1:2015, 3.10, modified — measurand has been replaced by analyte]

3.6

gas chromatograph

device that physically separates components of a mixture in the gaseous phase and measures them individually with a detector which signal is processed

[SOURCE: ISO 14532:2014, 2.4.3]

3.7

mass spectrometer

instrument which separates ionized particles of different *mass/charge ratios* (3.8) and measures the respective ion currents

[SOURCE: ISO 3529-3:2014, 2.5.1]

3.8

mass/charge ratio

mass of a charged particle in atomic mass units divided by its elementary charge

3.9

powder metallurgy hot isostatic pressing

PM HIP

process for simultaneously heating and forming a compact in which the powder is contained in a sealed formable enclosure usually made from metal and the so-contained powder is subjected to equal pressure from all directions at a temperature high enough to permit plastic deformation and consolidation of the powder particles to take place

[SOURCE: ASTM A988/A988M – 15A]

3.10

reference sample

material or substance which property values are sufficiently homogeneous and well established to be used for the *functionality test* (3.5) of an apparatus, the assessment of a measurement method, or for assigning values to materials

3.11

solvent cleaner

liquid cleaning substance that is either ethanol, CH₃CH₂OH, acetone, (CH₃)₂CO or iso-propanol, CH(CH₃)₂OH

4 Equipment for argon detection

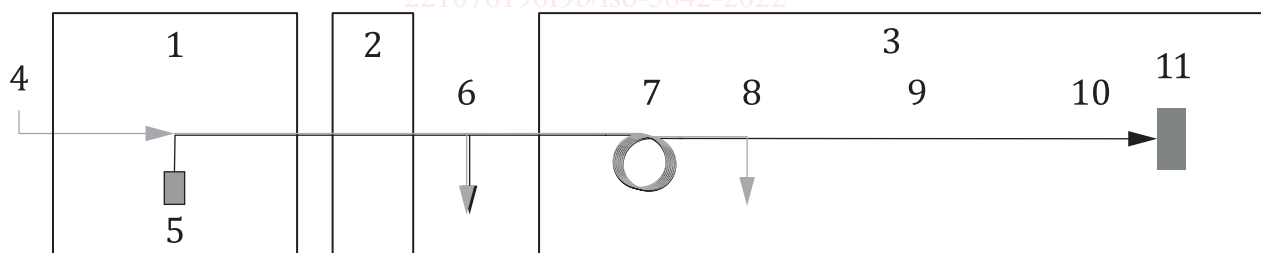
4.1 Gas chromatography

4.1.1 Principle

Gas chromatography (GC) is a separation technique where the mobile phase is gaseous. When GC is used as analytical technique, a known amount of sample is evaporated and dissolved into the mobile phase, also called the carrier gas. The sample compounds are carried by this mobile phase through a chromatographic column and further through detectors. Due to different specific interactions between the sample compounds and stationary phase (stationary phase is usually a chemical that can selectively attract components in a sample mixture), the sample compounds are retained individually and “travel” through the column with different velocities. This leads to a separation of individual components in time. The so-called retention time is the amount of time that elapsed from injection of the sample to the recording of the peak maximum of the component band (peak). When using non-specific detectors, the retention time is the only indication in chromatography for the correct identification of an individual sample component.

4.1.2 Apparatus and reagents

The carrier gas for argon detection shall be helium with purity $\geq 99,9999$ % by volume. Measurement starts with fusion of a test portion in a graphite crucible under helium gas at a temperature of about 2 200 °C. The instrument samples a known volume of gas which subsequently enters the column(s) where the gases are separated. The apparatus shall, by an integrated function, correctly identify the signal which belongs to the separated argon. As an example, in [Figure 1](#), a time relayed valve ejects all other gases to leave-out argon as the only analyte producing a signal. However, the chromatograph column may not be able to separate argon from all gases that may originate from the test sample. In that case, it can be necessary to integrate into the apparatus a separation and/or chemical conversion step prior to the gas stream entering the chromatograph column. The argon is ionized by an ionization source in a chamber followed by detection and quantification by the detector. An illustration of an apparatus is shown in [Figure 1](#).



Key

- | | | | |
|---|-----------------------------|----|--------------------------|
| 1 | furnace | 7 | gas chromatograph column |
| 2 | particle filter | 8 | relayed valve |
| 3 | gas chromatograph | 9 | ion source |
| 4 | helium carrier gas | 10 | ionized argon |
| 5 | test sample | 11 | detector |
| 6 | sampling valve (gas sample) | | |

Figure 1 — Example illustration of a gas chromatography apparatus

4.1.3 Equipment detection limit

The detection limit of the equipment shall at most correspond to a content of 0,02 µg/g argon in a test sample prepared according to [Clause 6](#).

NOTE 1 The detection limit does not correspond to the lowest level that can be quantified with statistical certainty.

NOTE 2 0,02 µg/g is sometimes referred to as 0,02 mg/kg, 0,02 ppm by mass or 20 ppb by mass.

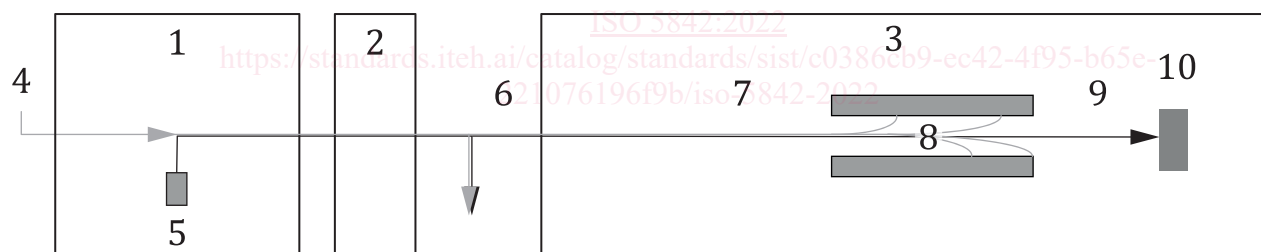
4.2 Mass spectrometry

4.2.1 Principle

Mass spectrometry (MS) is an instrumental method to identify and quantify chemical compounds or elements. It involves a sample that is being dissolved in a carrier gas. The carrier gas carries the compounds through the instrument to the detector. After ionization, the instrument detects extracted argon based on the mass/charge ratio.

4.2.2 Apparatus and reagents

The carrier gas for argon detection shall be helium with purity ≥ 99,9999 % by volume. Measurement starts with fusion of a test portion in a graphite crucible under helium gas at a temperature of about 2 200 °C. The gases are ionized by an ionization source in a chamber. Subsequent detection and quantification of the extracted argon (mass/charge ratio of 40) is made by a mass spectrometer. However, a chemical compound or element that originates from the test sample may give rise to an overlapping signal with the extracted argon. In that case, it can be necessary to integrate into the apparatus a separation and/or chemical conversion step prior to the ionization. An illustration of an apparatus is shown in [Figure 2](#).



Key

1	furnace	6	aliquot (gas sample)
2	particle filter	7	ion source
3	mass spectrometer	8	mass selector
4	helium carrier gas	9	ionized argon
5	test sample	10	detector

Figure 2 — Example illustration of a mass spectrometry apparatus

4.2.3 Equipment detection limit

The detection limit of the equipment shall at most correspond to a content of 0,02 µg/g argon in a test sample prepared according to [Clause 6](#).

NOTE 1 The detection limit does not correspond to the lowest level that can be quantified with statistical certainty.

NOTE 2 0,02 µg/g is sometimes referred to as 0,02 mg/kg, 0,02 ppm by mass or 20 ppb by mass.