SVENSK STANDARD
SS-ISO 15651:2015

Fastställd/Approved: 2015-03-11
Publicerad/Published: 2015-03-13
Utgåva/Edition: 1
Språk/Language: engelska/English
ICS: 27.120.30

Kärnenergi – Bestämning av totalt väteinnehåll i pulver av PuO_2 och UO_2 och i sintrade kutsar av UO_2, $(UGd)O_2$ och $(U,Pu)O_2$ – Extraktion av inert gas och konduktivitetsmätning (ISO 15651:2015, IDT)

Nuclear energy – Determination of total hydrogen content in PuO_2 and UO_2 powders and UO_2, $(U,Gd)O_2$ and $(U,Pu)O_2$ sintered pellets – Inert gas extraction and conductivity detection method (ISO 15651:2015, IDT)

Standarder får världen att fungera

SIS (Swedish Standards Institute) är en fristående ideell förening med medlemmar från både privat och offentlig sektor. Vi är en del av det europeiska och globala nätverk som utarbetar internationella standarder. Standarder är dokumenterad kunskap utvecklad av framstående aktörer inom industri, näringsliv och samhälle och befrämjar handel över gränser, bidrar till att processer och produkter blir säkrare samt effektiviserar din verksamhet.

Delta och påverka
Som medlem i SIS har du möjlighet att påverka framtida standarder inom ditt område på nationell, europeisk och global nivå. Du får samtidigt tillgång till tidig information om utvecklingen inom din bransch.

Ta del av det färdiga arbetet
Vi erbjuder våra kunder allt som rör standarder och deras tillämpning. Hos oss kan du köpa alla publikationer du behöver – allt från enskilda standarder, tekniska rapporter och standard-paket till handböcker och onlinetjänster. Genom vår webbtjänst e-nav får du tillgång till ett lättnavigerat bibliotek där alla standarder som är aktuella för ditt företag finns tillgängliga. Standarder och handböcker är källor till kunskap. Vi säljer dem.

Utveckla din kompetens och lyckas bättre i ditt arbete
Hos SIS kan du gå öppna eller företagsinterna utbildningar kring innehåll och tillämpning av standarder. Genom vår närhet till den internationella utvecklingen och ISO får du rätt kunskap i rätt tid, direkt från källan. Med vår kunskap om standarders möjligheter hjälper vi våra kunder att skapa verklig nytta och lönsamhet i sina verksamheter.

Vill du veta mer om SIS eller hur standarder kan effektivisera din verksamhet är du välkommen in på www.sis.se eller ta kontakt med oss på tel 08-555 523 00.

Standards make the world go round

SIS (Swedish Standards Institute) is an independent non-profit organisation with members from both the private and public sectors. We are part of the European and global network that draws up international standards. Standards consist of documented knowledge developed by prominent actors within the industry, business world and society. They promote cross-border trade, they help to make processes and products safer and they streamline your organisation.

Take part and have influence
As a member of SIS you will have the possibility to participate in standardization activities on national, European and global level. The membership in SIS will give you the opportunity to influence future standards and gain access to early stage information about developments within your field.

Get to know the finished work
We offer our customers everything in connection with standards and their application. You can purchase all the publications you need from us - everything from individual standards, technical reports and standard packages through to manuals and online services. Our web service e-nav gives you access to an easy-to-navigate library where all standards that are relevant to your company are available. Standards and manuals are sources of knowledge. We sell them.

Increase understanding and improve perception
With SIS you can undergo either shared or in-house training in the content and application of standards. Thanks to our proximity to international development and ISO you receive the right knowledge at the right time, direct from the source. With our knowledge about the potential of standards, we assist our customers in creating tangible benefit and profitability in their organisations.

If you want to know more about SIS, or how standards can streamline your organisation, please visit www.sis.se or contact us on phone +46 (0)8-555 523 00

Den internationella standarden ISO 15651:2015 gäller som svensk standard. Detta dokument innehåller den officiella engelska versionen av ISO 15651:2015.

The International Standard ISO 15651:2015 has the status of a Swedish Standard. This document contains the official English version of ISO 15651:2015.

Upplysningar om sakinnehållet i standarden lämnas av SIS, Swedish Standards Institute, telefon 08-555 520 00. Standarder kan beställas hos SIS Förlag AB som även lämnar allmänna upplysningar om svensk och utländsk standard.

Information about the content of the standard is available from the Swedish Standards Institute (SIS), telephone +46 8 555 520 00. Standards may be ordered from SIS Förlag AB, who can also provide general information about Swedish and foreign standards.

Denna standard är framtagen av kommittén för Kärnenergi, SIS/TK 405.

Har du synpunkter på innehållet i den här standarden, vill du delta i ett kommande revideringsarbete eller vara med och ta fram andra standarder inom området? Gå in på www.sis.se - där hittar du mer information.

Contents

Page

Foreword ..... **iv**

1 Scope **1**

2 Normative references **1**

3 Principle **1**

4 Interference **1**

5 Reagents and materials **1**

6 Apparatus **2**

7 Sampling **2**
7.1 Sampling procedure 2
7.1.1 Powders 2
7.1.2 Pellet 3
7.2 Preparation 3
7.2.1 Powder 3
7.2.2 Pellet 3

8 Procedure **3**
8.1 Blank test 3
8.2 Calibration 3
8.2.1 Calibration of the analyser 3
8.2.2 Check of the calibration 3
8.3 Determination 4

9 Calculation **4**

10 Precision **4**

11 Test report **5**

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).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT), see the following URL: Foreword — Supplementary information.

The committee responsible for this document is ISO/TC 85, *Nuclear Energy*, Subcommittee SC 5, *Fuel Technology*.

Nuclear energy — Determination of total hydrogen content in PuO_2 and UO_2 powders and UO_2, $(U,Gd)O_2$ and $(U,Pu)O_2$ sintered pellets — Inert gas extraction and conductivity detection method

1 Scope

This International Standard describes a procedure for measuring the total hydrogen content of UO_2 and PuO_2 powders (up to 2 000 µg/g oxide) and of UO_2 and $(U,Gd)O_2$ and $(U,Pu)O_2$ pellets (up to 10 µg/g oxide). The total hydrogen content results from adsorbed water, water of crystallization, hydrocarbon, and other hydrogenated compounds which can exist as impurities in the fuel.

2 Normative references

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

ISO/IEC Guide 98-3:2008, *Uncertainty of measurement — Part 3: Guide to the expression of uncertainty in measurement (GUM: 1995)*

3 Principle

The total hydrogen content is determined using a hydrogen analyser. The hydrogen analyser is based on the carrier gas method using argon or nitrogen as carrier gas. The samples to be analysed are heated up to a temperature of more than 1 770 °C in a graphite crucible. At that temperature, all volatile components are removed. The hydrogen containing compounds are cracked and released as hydrogen, oxygen, nitrogen, and carbon monoxide. The released gas is purified in the carrier gas stream, for example by oxidation and adsorption columns. The hydrogen is separated by chromatographic means and analysed in a thermal conductivity cell.

4 Interference

The temperature of >1 770 °C should be reached in a short time, within approximately 5 s; if not, the decomposition of the released water to hydrogen and oxygen might not be complete.

At temperatures of more than 2 200 °C, carbon dioxide is released because of a reduction of UO_2 by graphite according to the reaction below; carbon dioxide will interfere with the thermal conductivity measurement.

$$UO_2 + C \rightarrow UO_{2-x} + x/2\ CO_2$$

5 Reagents and materials

Use reagents of recognized analytical grade. The reagents and materials below serve as examples to be used according to manufacturer recommendation.

5.1 Carrier gas. Use argon with a purity of a volume fraction ≥99,995 % or nitrogen with a purity of a volume fraction ≥99,998 %.

5.2 Calibration gas. If calibration is performed with gas, use Argon or nitrogen with certified hydrogen content or carrier gas mixed with a known amount of hydrogen with a purity of a volume fraction ≥99,999 %.

5.3 Reference material.[1)] If calibration or calibration check is performed with a standard material, use a reference material with certified hydrogen content (e.g. titanium or zirconium).

5.4 Copper(II) oxide, CuO purifies the carrier gas (Ar/N_2), converting H_2 to H_2O.

5.5 Absorption media for H_2O. After converting H_2 to H_2O (§5.4), anhydrone [$Mg(ClO_4)_2$] is used to trap H_2O.

5.6 Oxidation reagent for CO, Schutze reagent (iodine pentoxide over silica gel) preceded by Hopcalite [manganese oxide/copper(II) oxide] oxidizes CO to CO_2 present in the carrier gas or extracted during measurement.

5.7 Absorption media for CO_2, sodium hydroxide over clay or equivalent will then absorb the CO_2.

5.8 Flux reagents for reference material. Tin, copper, or nickel granules are used as flux to accelerate the melting of reference material.

6 Apparatus

6.1 Hydrogen analyser. It will consist in a furnace capable to reach temperature of at least 2 200 °C, a thermal conductivity cell and gas purifying systems.

6.2 High purity graphite crucible, suitable for the appropriate sample types.

The impurity content should not exceed 2 µg/g.

6.3 Hydrogen free tin, copper, or nickel capsules.

6.4 Balance, with precision of 1 mg.

7 Sampling

7.1 Sampling procedure

7.1.1 Powders

Sampling is done with a tube shaped powder sampler having an inner diameter of more than 2,5 times of the maximum powder particle size. The sample shall be exposed to ambient conditions for not longer than 5 min because alterations of the powder sample due to moisture adsorption or desorption or oxidation have to be avoided. The sample has to be stored in tight containers. The gas volume in the container should be as low as possible. In case the analysis is not performed immediately after the sampling, the sample mass has to be controlled before and after storage period prior to the analysis. These precautions can be relaxed if the analysis process is made under controlled gas environment (glove box environment).

---

1) Reference materials are available for example from “National Institute of Standard and Technology (NIST)” or “LECO Corporation”. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of these products.