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REPORT ON LITERATURE REVIEW OF RECENT DEVELOPMENT IN LOSS ON DRYING METHOD FOR MOISTURE DETERMINATION

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REPORT ON LITERATURE REVIEW OF LOSS ON DRYING METHOD FOR MOISTURE DETERMINATION

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1 INTRODUCTION

The presence and effects of moisture in materials can impact on almost any product or process in a vast range of sectors and research fields, such as pharmaceutical, food, forestry, textile, petrochemical, cosmetics, polymer or composite materials etc. Moisture content in materials affects their properties in various ways. Properties such as mechanical strength, corrosion, biological action (e.g. mould), electrical properties, bulk handling characteristics, and price when sold by weight are just some of the matters which are influenced by the extent and form of water present [1]. The term, "moisture" addressed in this report means both water and other volatile substances unless water is specified.

This report gives an overview of a literature survey of standards and the journal papers of moisture content measurements for the work carried out within the Deliverable 1.1.1 of EMRP Project of METefnet. The survey was conducted in July 2013 via British Standards (BS), European Standards (EN), Romanian standards (SR), International standards (ISO) as well as moisture measurement related journal papers published between 1991 and 2013. The key words used for searching for the standards and the paper are 'Moisture or Water content Measurement'. There were 45 British Standards, 40 European and Romanian Standards, 178 International Standards and 6834 journal paper meeting the search criteria. 158 representative papers were selected for further review. The detailed review of these selected papers showed that only 22 papers are really relevant to the survey. These standards and papers involve a broad range of applications in different industries and sectors, such as paper, wood, foodstuffs, agricultural products, polymer, plastics, building, construction, rubber, ceramics, personal care, pharmaceutical, cosmetics, chemical, textile and packaging. However this report will focus on sectors like paper, polymer, foodstuffs, wood, personal care and pharmaceutics.

2 DEFINITIONS

The moisture content of a material is normally expressed as a percentage of its wet mass ("wet basis") unless "dry basis" is specified.

In the conventional definition, "moisture content" does not consist exclusively of water, but may include other volatile substances which are not separately distinguished by a given measurement process. This convention has arisen because of the measurement and calibration methods which are considered to be "standard" for this area of measurement. When referring exclusively to the water present, the term "water content" may be clearer. However this term was not found to be adopted in any of the papers surveyed. This report adheres to the general usage of the term.

When expressing or interpreting values, care must be taken to distinguish between percentage moisture content and percentage of value. For instance at a moisture content of 15%, a change in moisture content of 3% corresponds to 20 percent of value [1].

Moisture content through loss-on-drying methods (LoD) can be defined as a percentage of its wet mass (wet basis) or dry mass (dry basis):

$$Moisture\ content\ (wet\ basis) = \frac{m_w}{m_m} \times 100\% = \frac{m_w}{m_w + m_d} \times 100\% \qquad (1)$$

$$Moisture\ content\ (dry\ basis) = \frac{m_w}{m_d} \times 100\% = \frac{m_m - m_d}{m_d} \times 100\% \qquad (2)$$

where: m_w = mass of moisture, m_m = mass of moist material and m_d = mass of dry material [2].

3 INFLUENCE OF MOISTURE ON MATERIALS

(1) Paper and board

Properties of paper are affected by moisture during manufacturing and storage and application. The moisture content and its distribution in paper is an important factor for quality control parameters, such as dimensions, flatness, conductivity, strength and so on. For example, wrongly conditioned paper becomes visibly puckered. The moisture content of paper surfaces during printing is also critical, affecting the print adhesion and quality of finish.

(2) Foodstuffs and agricultural products

The moisture content in foodstuffs is recognized as a critical factor in determining their storage, stability, processing, application performance, health and safety. Spoilage (mould and infestation, as well as less serious "staleing"), texture and handling properties, value by weight, cooking properties, and many other issues hinge on moisture content [1]. Furthermore, moisture content plays critical roles in the storage and performance of agricultural products like pesticides, herbicides, fertilizers and seeds.

(3) Polymer and plastics

Many polymers are hygroscopic; they absorb moisture and water when they are exposed to atmosphere. Some polymers like nylon contain hydrogen bonding and attract water or moisture to a much greater degree [3]. The moisture is known to affect a range of polymer properties, which in turn impact processability, dimensional stability, mechanical, acoustic, electrical, optical, and chemical properties, as well as performance of the products [4-5]. For example, water is a plasticizer for nylon, which means that it reduces the material's glass-transition temperature. Water also has the same hydrogen bonding as nylon, so when water enters a nylon material it has the same opportunity to become loosely attached to the nylon chain as does another nylon chain. It forces the spacing between the polymer chains to increase hence causes volumetric expansion.

(4) Building and construction materials

Moisture has a significant impact on building materials such as cement, wood, insulation materials and fibres. Moisture damage is a significant factor limiting a building's lifespan, for example, bituminous materials need to contain less than 0.1% moisture in their raw state. If

moisture content is incorrect, coatings can fail to bond, or can strip away some time after application. Furthermore moisture infusion through a building's outer structure can have a significant effect on indoor air quality and air-conditioning load.

(5) Wood and forestry

The moisture content of wood governs whether the wood or adjacent materials will suffer rot or similar deterioration. Moisture stability (rather than moisture content itself) is significant for wood in other ways. Most importantly, wood suffers dimensional changes if its moisture content changes. This leads to splitting and shrinkage of the wood, and peeling of coatings.

(6) Pharmaceutical and personal care

The moisture in pharmaceutical materials such as excipients, drug formulations and packaging films are critical factors in determining their storage, shelf-life, stability, processing and delivery performance. The moisture content in personal care materials is also regarded as an important factor, for instant, moisture content in skin cream has significant effect on the performance of moisturisation and hydration behavior of skin.

4 MOISTURE CONTENT MEASUREMENT TECHNIQUES

Techniques for moisture content measurement in solids can be classified as "Absolute" (direct) and "Inferential" (indirect) techniques. The direct techniques include distillation, Karl Fisher titration, thermogravimetric analysis (loss on drying - LoD) and more recently coulometric vapour evolution technique. Indirect techniques are electrical conductance, electrical capacitance, near infrared reflection (NIR), radio frequency and microwave absorption, neutron moderation and nuclear magnetic resonance (NMR). The direct measurement techniques currently provide the highest level of accuracy available in moisture content measurement. The indirect measurement techniques can be referenced or checked against direct techniques, by applying both measurements to the same batch or sample of material, and comparing the results [2].

The main approaches to moisture measurement and their applicabilities are summarised in Table 1 [6-7]. It can be seen that each technique has its own measurement advantages and disadvantages. For example, most indirect measurement techniques can be used to measure moisture content instantly and non-destructively hence can be applied to production in situ monitoring, but due to their indirect measurement nature, they need to be calibrated against direct measurement techniques like LoD, KF titration, distillation and vapour evolution coulometric methods. The disadvantages of some direct measurement methods are that they are time consuming and destructive to sample materials. For example, the drying procedure in LoD technique may require several hours, even several days to reach a constant dry mass. KF titration method employs titration reagents and involves chemical reaction, which is destructive to sample materials and may pose environmental concern in terms of chemical disposal.

Tables 2, 3 and 4 summarise the moisture content measurement techniques used for various applications. This information is gathered from the documented moisture content measurement

standards and related journal papers. It can be seen that compared to other moisture content measurement methods, LoD is still the most widely used technique, especially in measurement standards for foodstuffs and agricultural products. This is probably because the technique is absolute and relatively simple. For the indirect measurement, the widely used techniques are microwave or radio frequency, near infra-red and electric capacitance methods. LoD method is also used as a reference or calibration method against other indirect methods, especially in food sector.

Table 1 Methods of moisture measurement

Method	**Range of moisture content ("wet basis")**	**Uses, restrictions and sampling requirements**
Electrical conductance Current (DC or AC) is passed between electrodes. Pure water and most solids do not conduct - actually measures presence of aqueous solutions.	0 - 25%	Affected by other conductive substances in the material (e.g. salts, wood preservatives). Surface effects may dominate the results. Accuracy affected by inhomogeneities in the material.
Electrical capacitance between plates or electrodes. (Dielectric permittivity)	0 - 30% (beyond which most materials are conductors)	Conductivity and density of the material affect the measurements. Geometry-sensitive. Particular surface characteristics may affect readings. (Some instruments use insulated electrodes and high frequencies (>10 GHz) to deal with this). Dielectric spectroscopy a developing extension of this field.
Near infrared reflection (or rarely transmission) at "absorbed" and "reference" wavelengths.	0 - 95%	A surface measurement (or with very limited penetration of 2-3 mm). Not generally colour sensitive, but reflective surfaces and very dark materials may be hard to measure. Mainly designed for granular or particulate material.
Microwave absorption	0 - 100% (laboratory) 3 - 45% (on-line)	Sensitive to changes in density and temperature of material.
Radio frequency	0 - 100%	Sensitive measurement. Affected by density and position.
Neutron moderation	0 - 100%	Detects all hydrogen, not just water.
Nuclear magnetic resonance	0 - 100%	Affected by hydrogen present in the sample other than in water (e.g. in oils). "Pulsed" NMR can be water-specific. Ground-up samples or solid plugs of material.
"Wet chemistry" methods Karl Fischer titration	0 - 100%	Ground material mixed with solvents or reagents for laboratory chemical analysis. Karl Fischer method regarded as "absolute" but there are other chemical methods (e.g. carbide reaction) which do not have the same standing.
Thermogravimetric	0 - 100%	Measures total volatile content, not only water, so

analysis by weighing before and after moisture is driven off using conventional oven or microwave heating.		may read falsely high. Oven drying may drive off an unspecified amount of bound water as well as free water. Nonetheless regarded as an "absolute" reference measurement. Normally need ground-up samples.
Distillation	0-100%	Water (and perhaps other volatiles) are driven off and measured by volume, and compared to weight of original substance. Used for materials such as bitumen.

Table 2 European, British and Romanian standards (EN, BS and SR)

	Loss on Dry (LoD)	Gain on Wet (GoW)	Distillation	KF titration	Coulometric vapour evolution (P_2O_5)	LoD used as reference and calibration method
Building and construction(16)	12	1	3			
Polymer and plastics (10)	3	2	1	3	1	
Wood and forestry (1)	1					
Foodstuffs and agricultural products (3)	3					6
Pharmaceutical and personal care (2)	1	1				
Cosmetics (2)	2					
Paper and board	2					
Others (13)	11		1	2		
Total: 42	33	3	5	5	1	6

Table 3 International standard (ISO)

	Loss on Dry (LoD)	Gain on Wet (GoW)	Distillation	KF titration	Electrical conductance	NIR	Others	LoD used as reference and calibration method
Building and construction(9)	3	2			1	2	2	
Polymer and plastics (4)	2	1		1		1	1	
Wood and forestry (6)	3				1		1	
Foodstuffs and agriculture (30)	18		1	2	2	2	5	6

Personal care and pharmaceutical (1)	1							
Cosmetics (1)	1							
Paper and board (3)	2		1				2	
Others (32)	27			1		1	3	
Total: 86	57	3	2	5	4	6	14	6

Table 4 Journal papers

	Loss on Dry (LoD)	KF titration	Electrical conductance	Electric capacitance	Near infrared (NIR)	Nuclear magnetic resonance (NMR)	Microwave /radio frequency	Neutron technique	X-ray computed tomography (CT)	Mass spectrometry	Time domain reflectometry (TDR)	Coulometric vapour evolution (P_2O_5)	Dynamic vapour sorption (DVS)	Thermogravimetric anaysis (TGA)	Others	LoD used as reference and calibration method
Building and construction (14)	1		1	1		1	3		1		2				5	1
Polymer and plastics (8)	1	1		1			1					1	1		3	1
Wood and forestry (20)	2		1		2	4	6		3		1		1	1	6	3
Foodstuffs and agriculture (70)	8	3	4	13	10	1	13	1		1	3				22	2
Personal care and pharmaceutical (10)		1		1	5		1					1	1		1	1
Cosmetics (3)						1									2	
Paper and board (5)					1		2								2	1
Others (16)	3	3	1	2	3		2			2	1		2		5	3
Total: 146	15	8	7	18	21	7	28	1	4	3	7	2	5	1	46	12

5 OVEN DRYING METHOD (LOSS ON DRYING)

The oven drying method is simple and straightforward, and involves three steps: a sample is weighed, and then dried in an oven, and weighed afterwards again. Protocols vary for the sample preparation, drying temperature and duration, and the criteria of the "end point" detection. This method is considered to be "fundamental" and to need no calibration or validation, and thus is widely used for determination of moisture content in solids. However it is useful to investigate if there is any recent development in this technique. A literature survey was conducted for this purpose and the results of the survey on LoD standards is summarised in the Tables 5 to 10 according to the category of the materials. The sample size, drying temperature and drying time are plotted in the Figures 1 to 6 for comparison. It should be pointed out that the survey was conducted in July 2013 via EN, BS, SR and ISO standards

as well as journal papers published between 1991 and 2013. The journal papers were searched using the search engine, “Web of Science”. Therefore this survey may not cover a complete list of standards and papers related to moisture content measurement. Some of the searched papers are not relevant to the task of the survey hence are not included in this survey.

According to the survey, it appears that there is no significant change or development in LoD method which still involves three main steps, i.e. weighing of wet mass, drying of wet mass in an oven and weighing of dry mass. Only minor development in the drying step was found. For example, instead of using an traditional oven at ambient temperature, a vacuum or a reduced pressure drying process is utilised to dry solid fertilizers and liming materials (BS EN 12049:1997), cereals and pulses (BS 4317-2:1987 ISO 711:1985) and concentrate food (SR 8613-1:2009). In another case, a microwave oven is used to replace the traditional oven to dry the fine aggregate sample (BS 812-109:1990). In order to avoid humidity influence in the oven, a stream of dried air is passed through milk powders packed in a column in an oven at 87.0 ± 1.0°C [8]. It is also found that a desiccator only was used for drying inorganic thermal insulating materials with an upper temperature limit less than 120 °C (BS 2972:1989).

Neither the LoD standards nor the journal papers in the scope of the survey address traceability issues, e.g. how the oven temperature and the balance are calibrated. Only requirements of the oven temperature tolerance and the balance accuracy are mentioned.

Table 5 Moisture content measurement standard for paper and board

Standard number	Title	Procedure
BS ISO 11093-3:1994	Implementation of ISO 11093-3:1994 Paper and board —Testing of cores — Part 3: Determination of moisture content using the oven drying method	A minimum 50 g sample is weighed to an accuracy of 0.05 %. Dried at 105 ± 2 °C in an oven for an appropriate time and weighed within 30 s after removal from oven until the difference between two successive weighings is no more than 0.1 %
SR EN 920:2002	Paper and board intended to come into contact with foodstuffs-determination of dry matter content in aqueous extract	Two 250 ml samples and two control samples are weighed to the nearest 0.1 mg. Dried at 105 ± 2 °C in an oven for 30 or 60 min in an oven until the difference between two successive weighings does not differ by more than 0.5 mg.
SR EN ISO 287:2009	Paper and board. Determination of moisture content of a lot. Oven drying method	A sample is weighed to an accuracy of 0.05 %. Dried at 105 ± 2 °C in an oven for 30 or 60 min in an oven until the difference between two successive weighings does not differ by more than 0.1 %

Table 6 Moisture content measurement standard for Foodstuffs and agricultural products

Standard number	Title	Procedure
BS EN 12048 : 1997	Solid fertilizers and liming materials - Determination of	10 g sample is weighed to the nearest 0.001 g. Dried at 105 ± 2 °C in an oven for 5 hr, then

SR EN 12048 : 2001	moisture content - Gravimetric method by drying at (105 ± 2) °C	cooled to ambient temperature in a desiccator and weighed to the nearest 1 mg until the difference between two successive weighings is less than 0.1 %
BS EN 12049 : 1997	Solid fertilizers and liming materials - Determination of moisture content - Gravimetric method by drying under reduced pressure	10 g sample is weighed to the nearest 0.001 g. Dried at 25 ± 3 °C and (66 ± 1.3) x 10^3 Pa [(500 ± 10) mmHg] in a vacuum desiccator for 24 hr, then weighed to the nearest 1 mg until the difference between two successive weighings is less than 0.1 %
BS EN 13040:2007	Soil improvers and growing media —Sample preparation for chemical and physical tests, determination of dry matter content, moisture content and laboratory compacted bulk density	50 g sample is weighed to an accuracy of 0.01 g. Dried at 103 ± 2 °C in an oven, then cooled in a desiccator and weighed to an accuracy of 0.01 g until the difference between two successive weighings is less than 0.1 g
BS 4317-2:1987 ISO 711:1985 SR ISO 711:1999	Methods of test for Cereals and pulses — Part 2: Determination of moisture content of cereals and cereal products (reference method)	About 3.5 g sample is weighed to the nearest 0.2 mg after pre-conditioning if necessary. Dried at a reduced pressure of 1.3 to 2.6 kPa and 45 to 50 °C in an oven for about 100 hr, then cooled to ambient condition with presence of diphosphorus pentaoxide in a drying tube and weighed to the nearest 0.2 mg until the difference between two successive weighings at an interval of 48 hr is less than 0.6 mg The difference between two independent measurements should be no more than 0.1 %.
BS 5752-14:1995 ISO 11294:1994	Methods of test for Coffee and coffee products —Part 14: Roasted ground coffee: Determination of moisture content [loss in mass at 103 °C (routine method)]	5 g sample is weighed to the nearest 0.1 mg. Dried at 103 °C in an oven for 2 ± 0.1 hr, then cooled to room temperature in a desiccator and weighed to the nearest 0.1 mg until the difference of moisture content between two successive measurements with a short interval is less than 0.1 %. Result is compared with that of KF titration
BS 4401-3:1997 ISO 1442:1997	Methods of test for Meat and meat products — Part 3: Determination of moisture content (reference method)	5 to 8 g sample is weighed to the nearest 0.001 g. Dried at 103 °C ± 2 °C °C in an oven for 2 hr, then cooled to room temperature in a desiccator and weighed to the nearest 0.001 g until the difference between two successive weighings separated by 1 hr is no more than 0.1 %.
BS EN ISO 1666:1998	Starch — Determination of moisture content — Oven-drying method	5 ± 0.25 g sample is weighed to the nearest 0.001 g. Dried at 130 to 133 °C °C in an oven for 1.5 hr, then cooled to room temperature in a desiccator for 30 to 45 min and weighed to the nearest 0.001 g within 2 min after removal from the desiccator. The difference between two independent measurements separated by a short interval should be no more than 0.5 %.
BS 5766-8:1999 ISO 6496:1999	Methods for analysis of animal feeding stuffs — Part 8: Determination of moisture and other volatile matter content	10 g sample is weighed to the nearest 1 mg. Dried at 103 °C in an oven for 30 ± 1 min, then cooled to room temperature in a desiccator and weighed to the nearest 1 mg until the difference between two weighings is no more than 0.1 %. Or dried in vacuum chamber at 80 °C and 13 kPa for 4 hr ± 0.1 hr, then cooled to room temperature in a desiccator and weighed to the nearest 1 mg until the difference between two weighings is no more than 0.2 %.

BS EN ISO 665:2000 BS 4289-3:2000	Oilseeds - Determination of moisture and volatile matter content	5 ± 0.5 g (grated products) or 5 to 10 g (whole seed) sample is weighed to the nearest 0.001 g. Dried at 103 ± 2 °C in an oven for 3 hr (12 to 16 hr in the case of cottonseed with adherent linters), then cooled to room temperature in a desiccator and weighed to the nearest 0.001 g until the difference between two successive weighings separated by 1 hr is no more than 0.005 g.
BS EN ISO 3727-1:2002 *Incorporating Corrigendum No. 1* SR EN ISO 3727-1: 2009	Butter — Determination of moisture, non-fat solids and fat contents — Part 1: Determination of moisture content (Reference method)	5 g sample is weighed to the nearest 1 mg. Dried at 102 °C °C in an oven for 2 hr, then cooled to room temperature in a desiccator and weighed to the nearest 1 mg until the difference between two consecutive weighings (the second drying for additional 0.5 hr) does not exceed 1 mg or until the mass increases. The difference between two independent measurements separated by a short interval should be no more than 0.1 % for 5 % of identical ground samples.
BS EN ISO 5537:2004 SR EN ISO 5537: 2005	Dried milk — Determination of moisture content (Reference method)	5 ± 0.3 g sample is weighed to the nearest 1 mg. Dried at 87 ± 1 °C in an ventilation oven (air flow: 33 ml/min, moisture content < 0.01 mgH_2O per litre at atmospheric pressure) for 5 hr, then cooled in a desiccator for 60 ± 5 min and weighed to the nearest 1 mg. The difference between two independent test results separated by a short interval should be no more than 0.15 % for 5 % of identical ground samples.
BS ISO 5550:2006 SR ISO 5550: 2008	Caseins and caseinates — Determination of moisture content (Reference method)	5 g (Caseins) or 2 g (Caseinates) sample is weighed to the nearest 1 mg. Dried at 102 ± 2 °C in an oven for 3 hr, then cooled to room temperature in a desiccator and weighed to the nearest 1 mg until the mass decreases by 1 mg or less, or increases between two successive weighings (the second drying for 1 hr).
BS ISO 24557:2009 SR ISO 24557: 2010	Pulses —Determination of moisture content — Air-oven method	50 g sample is weighed to nearest 0.001 g. Dried at 130 ± 1 °C in an oven for 15 min or 120 min (ground sample), then cooled to room temperature without use of a desiccator for 2 hr and weighed to the nearest 0.001 g. 2 to 3 g ground sample is weighed to the nearest 0.001 g. Dried at 130 ± 1 °C in an oven for 120 min, then cooled to room temperature in a desiccator normally for 45 to 60 min and weighed to the nearest 0.001 g. The difference between two independent test results separated by a short interval should be no more than 0.3 % for 5 % of identical ground samples.
BS EN ISO 712:2009 SR EN ISO 712: 2010	Cereals and cereal products — Determination of moisture content — Reference method (ISO 712:2009)	5 ± 1 g sample is weighed to the nearest 0.001 g. Dried at 130 to 133 °C in an oven for 120 ± 5 min (90 min for flour), then cooled in a desiccator (generally between 30 and 45 min) and weighed to the nearest 0.001 g. The difference between two independent test results separated by a short interval should be no more than 0.12 % for 5 % of identical ground samples.
SR 8613-1:2009	Concentrate food. Determination of moisture content	3 g sample is weighed to the nearest 0.001 g. Dried at 70 °C in a vacuum chamber with a

		pressure of 3333.05 Pa for 4 hr, or at 103 ± 2 °C for 4 hr or 130 ± 2 °C in an thermolegulated electric chamber for 40 min, respectively, then cooled to room temperature in a desiccator normally for 25 to 30 min and weighed to the nearest 0.001 g. The difference between two independent test results of the same sample should be no more than 2 % (as absolute value).
SR 110-3:1995	Sugar. Methods of analysis. Determination of moisture content	20 g sample is weighed to the nearest 1 mg. Dried at 103 ± 2 °C for 3 hr until constant mass is reached, then cooled in a desiccator (generally between 30 and 35 min) and weighed to the nearest 0.001 g. The difference between two independent test results of the same sample should be no more than 5% of the average value.
SR 6124-1999	Agricultural seeds. Determination of moisture content	Two samples with possible dimensions: under 8 cm diameter and 4 or 5 g weight, or at least 8 cm diameter and 10 g weight, are weighed to the nearest 0.001 g. Dried at 103 ± 2 °C for 17 ± 1 hr then cooled in a desiccator between 30 and 45 min and weighed to the nearest 0.001 g. The difference between two independent test results of the same sample should be no more than 0.2 %. Dried at 130 ... 133°C for 4 hr (maize), 2 h (other cereals) or 1 hr (other species) then cooled in a desiccator between 30 and 45 min and weighed to the nearest 1 mg. The difference between two independent test results of the same sample should be no more than 0.2 %.
SR EN ISO 6540:2010	Maize. Determination of moisture content (on milled grains and on whole grains)	30 g sample is milled and weighed to nearest 0.001 g. Dried at (130 ... 133) °C in an oven for 3 hours, then cooled to room temperature using of a desiccator and weighed to the nearest 0.001 g. For ventilation efficacy, the difference between two independent test results separated by a short interval should be no more than 0.15 %. If grains have dimensions less than 1.7 mm, the sample is not milled, just homogenized and if they are more than 1.7 mm, grains are milled without using preconditioning (if humidity is between 9 and 15 %) or using preconditioning (if humidity is less than 9 or more than 15 %), until humidity is in the interval (9 ... 15) %. Both cases need special conditions. 8 g ground sample is weighed to the nearest 0.001 g. Dried at 130 to 133 °C for 4 hr after heating, then cooled to room temperature in a desiccator normally for 30 to 45 min and weighed to the nearest 0.001 g. The difference between two independent test results separated by a short interval should be no more than 0.15 %. The standard includes also a procedure for whole grains, that it is not used for calibration or testing of moisture-meters, or for expertise reports. Absolute method: drying is done using phosphorus pentoxide, and (1.3 ... 2.6) kPa with vacuum pump, for 100 h at (45 ... 50) °C. Cooling

		is done at room temperature, then weighed to the nearest 0.2 mg. Method is repeated until difference between two independent test results in a 240 h interval is no more than 0.6 mg. The difference between two independent test results separated by a short interval should be no more than 0.10 %.
SR ISO 6496:2001	Animal feeding stuffs – Determination of moisture and other volatile matter content	10 g liquid sample is mixed with sand or 5 g sample of other sample type is weighed to nearest 0.001 g in a pre-dried tube. Dried at 103 °C for 4 ± 0.1 hr in an oven, then cooled to ambient in a desiccator and weighed to the nearest 1 mg until the change between the weighings does not exceed 0.1 %.

Table 7 Moisture content measurement standard for polymer and plastics

Standard number	Title	Procedure
BS EN ISO 585:1999	Plastics —Unplasticized cellulose acetate —Determination of moisture content	Approximately 5 g of cellulose acetate is weighed to nearest 1 mg. Dried at 105 ± 2 °C in a thermostatic oven for 3 hr, then cooled in desiccator and weighed to the nearest 1 mg until the difference between two successive weighing is less than 0.1%
BS 3241:19	Specification for Toughened polystyrene for sheet extrusion	5 to 15 g of the material shall be weighed to the nearest 0.001 g. Heated in an oven at 80 ± 2 °C for 3 hr ± 5 min, then cooled in a desiccator and weighed to the nearest 1 mg.
SR EN ISO 585:2004	Plastics. Unsaturated cellulose acetate. Determination of moisture content	Approximately 5 g of cellulose acetate is weighed to nearest 1 mg. Dried at 105 ± 2 °C in a thermostatic oven for 3 hr, then cooled in desiccator and weighed to the nearest 1 mg until the difference between two successive weighing is less than 0.1%

Table 8 Moisture content measurement standard for building and construction materials

Standard number	Title	Procedure
BS 2972:1989	Methods of test for Inorganic thermal insulating materials	No more than 5 g sample is weighed as received or after conditioning at 20 ± 2 °C with 35 ± 5 %, 65 ± 5 % and 90 ± 5 % r.h. Dried in a desiccator for materials with an upper temperature limit less than 120 °C or at 105 to 110 °C in a ventilated oven for materials with an upper temperature limit above 120 °C , then weighed to constant mass
BS 812-109:1990	Testing aggregates —Part 109: Methods for determination of moisture content	Minimum mass of test portion from 0.5 – 1.5 kg for aggregate size from less than 5 to 63 mm is weighed. (1) Dried at 105 ± 5 °C in an oven for 16 to 24 hr, then cooled in an air-tight cabinet for 0.5 to 1 hr and weighed to the nearest 0.1 g until the difference between two successive weighings is less than 0.1 % (2) Microwave oven method for fine

		aggregates. The same as (1) except that the oven is replaced with microwave oven.
BS 4046:1991	Specification for Compressed straw building slabs	50 ± 10 g sample is weighed to the nearest 0.1 g. Dried at 103 ± 2 °C in an air circulating oven, then cooled in a desiccator and weighed to an accuracy of 0.1 g until the difference between two successive weighing is less than 0.1 %
BS EN 1353:1997	Determination of moisture content of autoclaved aerated concrete	The test specimen with a minimum dimension of at least 50 mm and a volume of at least 0.5×10^{-3} m^3 is weighed to an accuracy of 0.1 %. Dried at 105 ± 5 °C in an ventilated oven, then weighed to an accuracy of 0.1 % until the difference between two successive weighing is less than 0.2 %
BS ISO 13765-6:20	Refractory mortars —Part 6: Determination of moisture content of ready-mixed mortars	At least 50 g sample is weighed to the nearest 0.1 g. Dried at 110 ± 5 °C in an oven for 5 hr or until two successive weighings made 10 min apart and after 2 hr in the oven do not differ by more than 0.2 g or 0.5 %, then cooled to room temperature in a desiccator and weighed to 0.1 g
BS EN 772-10:1999	Methods of test for masonry units - Part 10: Determination of moisture content of calcium silicate and autoclaved aerated concrete units	A representative sample is weighed to an accuracy of at least 0.1 % of its mass. Dried at 105 ± 5 °C in a ventilated oven until in two subsequent weighings with a 24 h interval, the loss in mass between the two determinations is less than 0,2 % of the total mass, then weighed to an accuracy of at least 0,1 % of their mass. The mean value of the moisture content is calculated to the nearest 1 %.
BS EN ISO 1927-3:2012	Monolithic (unshaped) refractory products Part 3: Characterization as received	No less than 200 g sample is weighed to the nearest 0.1 g. Dried at 110 ± 5 °C in an oven to a constant mass, cooled to ambient temperature and weighed to ± 0.1 g.
BS EN ISO 12570:2000+A1:	Hygrothermal performance of building materials and products – Determination of moisture content by drying at elevated temperature	A representative sample is weighed with an accuracy of 0.1 % of its mass. Dried at 105 ± 2 °C, or 65 ± 2 °C, or 40 ± 2 °C depending on materials in an ventilated oven with RH < 10 % in warm until the change of mass between three consecutive weighings made 24 h apart is less than 0.1 % of the total mass, then cooled to 30 to 40 °C in a desiccator and weighed to 0.1 % (laboratory temperature during the test shall be 23 ± 6 °C).
SR 4474 – 2: 2000	Plaster and gypsum. Determination of moisture, hidration water, alkalinity regarding phenolphthalein, insoluble residue in hydrochloric acid and silicon dioxide	2 g sample is weighed to the nearest 0.1 g. Dried at 40 ± 4 °C in an oven for 2 h, to a constant mass, cooled to ambient temperature in a dessicator and weighed to ± 0.1 g until the difference between two successive weighing is less than 0.01 %

Table 9 Moisture content measurement standard for wood and forestry

Standard number	Title	Procedure
BS 1990-1:1984	Wood poles for overhead power and telecommunication lines —	Samples of 75 mm or the full depth of the sapwood, whichever is the greater, shall be taken

	Part 1: Specification for softwood poles	from the poles. Dried at 103 ± 2 °C in an oven until the mass is constant and weighed immediately after removal from the oven.
SR EN 13183 – 1:2003	Moisture content of a piece of sawn timber. Part 1: Determination by oven dry method	A sample is weighed to the nearest 0.1 g for sample > 100 g or to the nearest 0.01 g for sample < 100 g immediately after taken from representative boards or pieces of a quality of lumber. Dried at 103 ± 2 °C in an oven until the difference between the two consecutive weighings separated by an interval of 2 hr is less than 0.1 %.

Table 10 Moisture content measurement standard for pharmaceutical and personal care

Standard number	Title	Procedure
BS 1715-2.6:1989 ISO 672:1978	Analysis of soaps —Part 2: Quantitative test methods — Section 2.6 Method for determination of moisture and volatile matter content [ISO title: Soaps — Determination of moisture and volatile matter content — Oven method]	10 g sample is weighed to the nearest 0.01 g. Dried at 103 ± 2 °C in an oven for 1 hr, then cooled in a desiccator to ambient temperature until the difference in mass between two successive weighings is less than 0.01 g

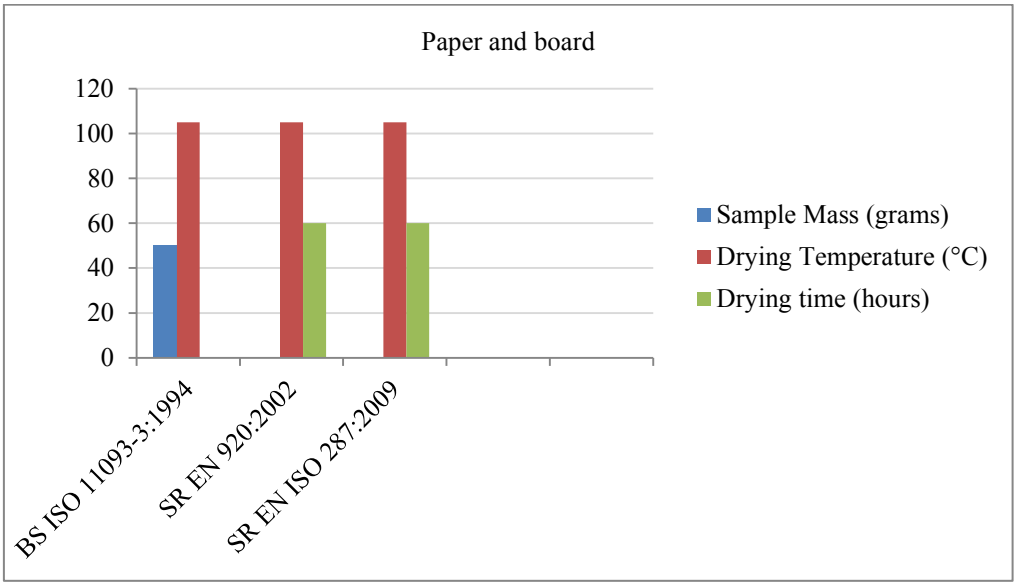

Figure 1 Comparison of measurement conditions for paper and board

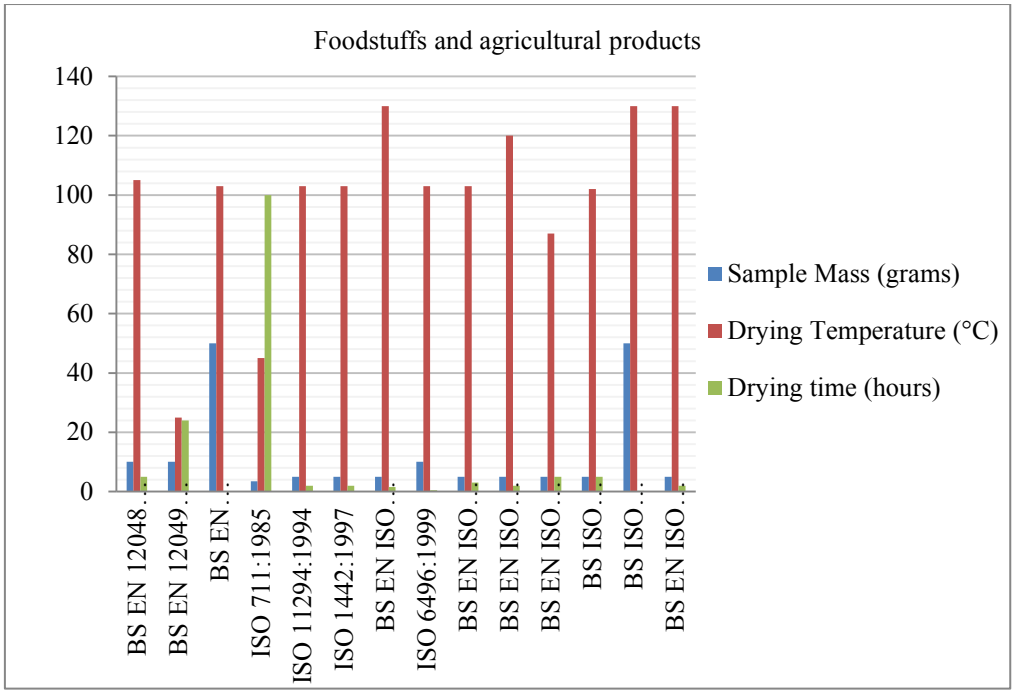

Figure 2 Comparison of measurement conditions for foodstuffs and agricultural products

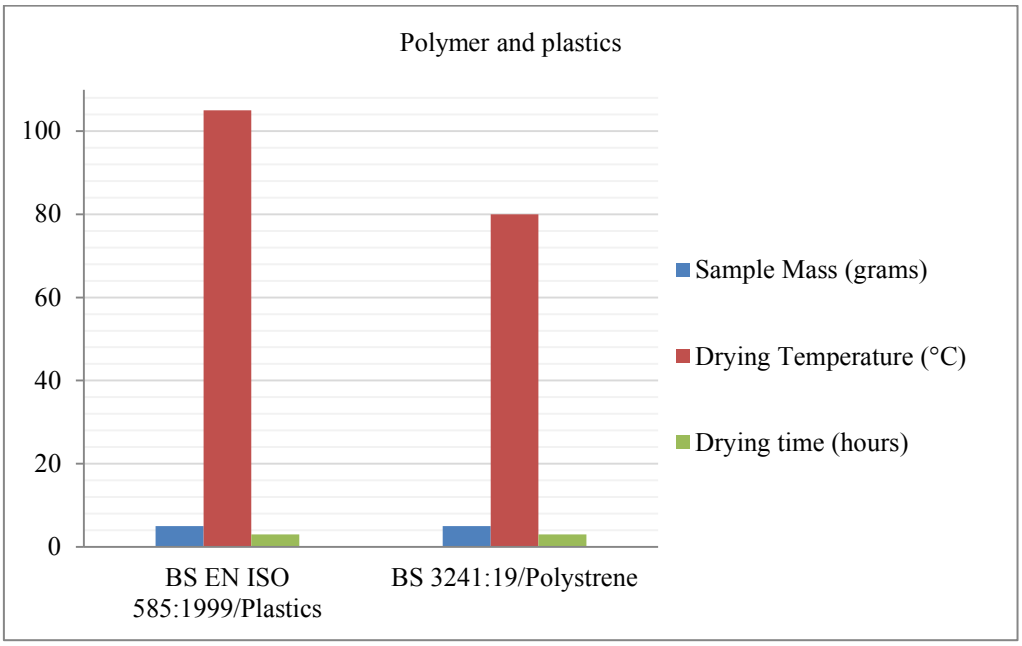

Figure 3 Comparison of measurement conditions for polymer and plastics

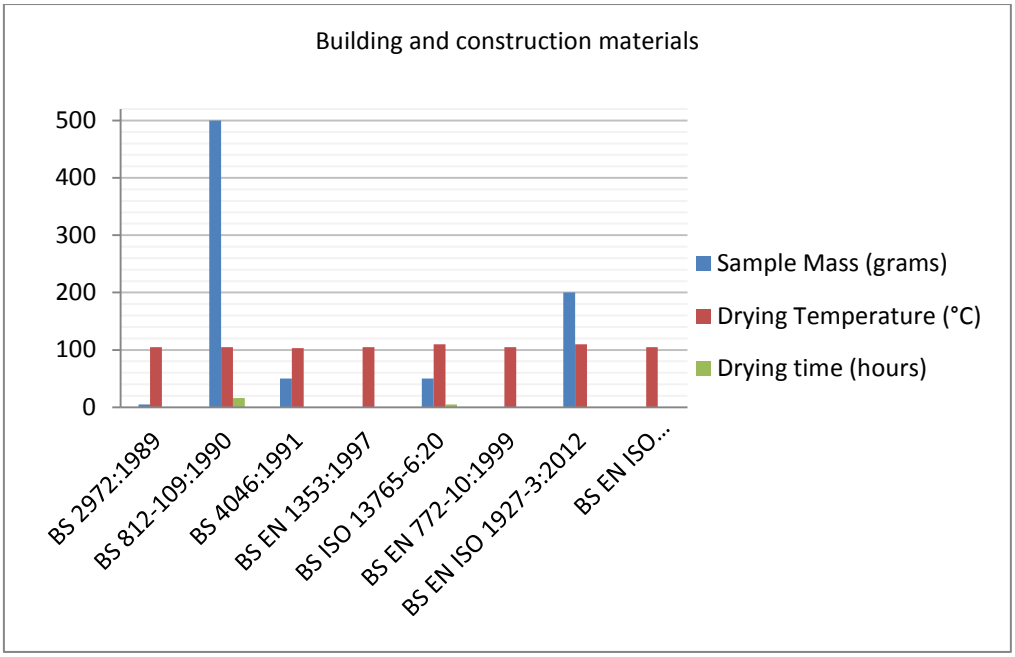

Figure 4 Comparison of measurement conditions for building and construction materials

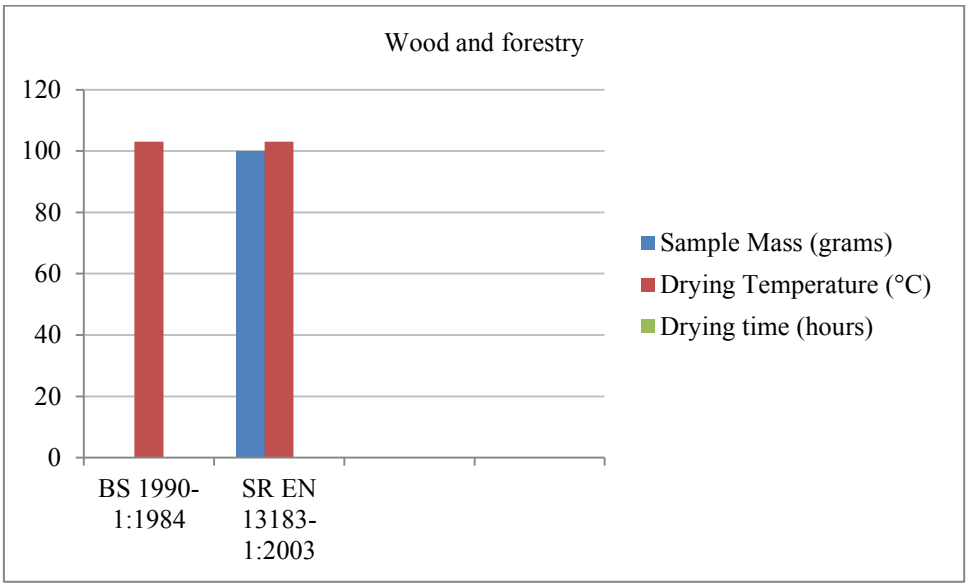

Figure 5 Comparison of measurement conditions for wood and forestry

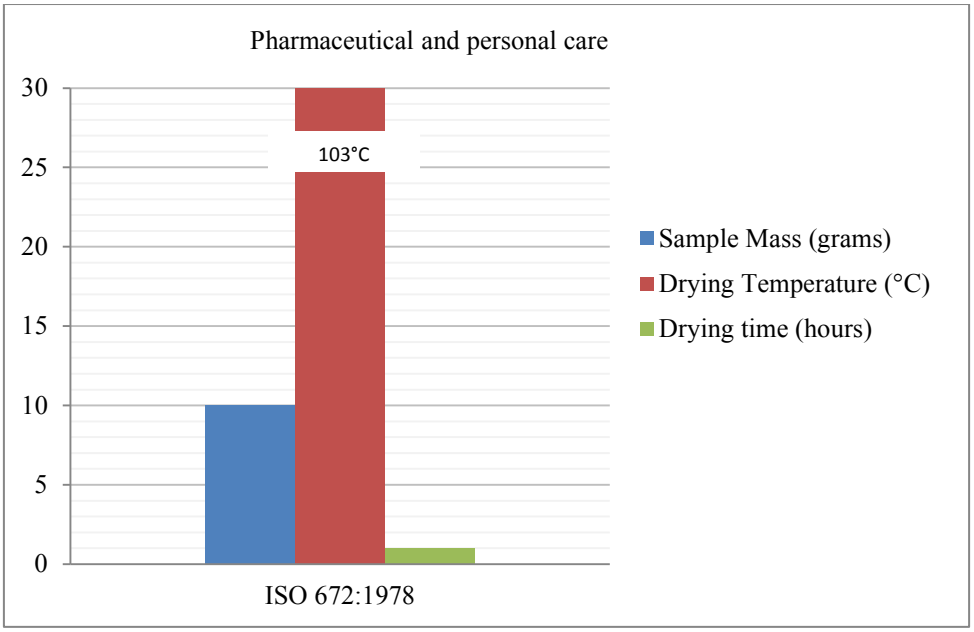

Figure 6 Comparison of measurement conditions for pharmaceutical and personal care

6 PROBLEMS WITH LOSS ON DRYING METHOD

The loss on drying method (LoD) is a most widely recognized method for moisture determination for its simplicity and fundamentality, therefore it is not only used as a moisture content measurement method, but also as a calibration method against other faster and more practical indirect techniques such as electrical conductance, electric capacitance, optical absorption, time domain reflectometry (TDR), microwaves and x-rays, infrared (IR) [9-13], etc. or as a reference and comparison method [14-20]. However, in practice, there are some problems associated with this method.

First of all, the oven drying method is not specific to water. Other volatiles, such as organic solvents, oils etc. may be included in any measured weight loss, or may interfere with driving off the moisture. Some bound water may be also driven off with free water if the drying temperature is too high. This will lead to error in the measurement. In many industries like food, agriculture and pharmaceutical, water content is their main concern. In this case, LoD method must be used with care for determining water content. To avoid confusion, the results should be compared with those obtained by water specific techniques such as KF titration etc.

Even for moisture determination, there are still some practical issues affecting the uncertainties involved in the LoD method, for example, sample preparation and handling, sample size, temperature, pressure, humidity, air velocity and drying duration in oven, loss or gain of sample moisture in ambient environment, possible spillage in oven, balance uncertainty and sensitivity, burning, melting and chemical decomposition and so on. Among these factors, the sample preparation and handling may be the greatest source of the uncertainty for this method.

The major weakness of standard oven methods is their unsatisfactory reproducibility. It is evident that the residual water content in materials after drying is related to the temperature

and relative humidity in the oven [8]. In addition, during sample preparation and handling, the humidity and temperature in the laboratory will play a crucial role in moisture gain or loss of the sample. In most cases, samples are cooled in a desiccator after they are removed from oven before re-weighing, but occasionally, the samples are cooled in ambient, which may lead to mass gain, hence an error in dry mass weighing

Apart from temperature and humidity, pressure in oven also influences the drying process. A reduced pressure in the oven is in favour of the material drying. Although in theory, the pressure dependent buoyancy of the sample also influences the gravimetric measurements, in practice, this influence should be negligible for most cases. Air flow in the oven is another parameter affecting measurements, for example, the convection and, at low pressures, the thermal gas flow effects.

The physical properties of a sample also have influence on drying process. During the drying of large samples, shrinking of porous materials (collapse of pores) or caking of fine materials will result in obstruction to moisture evaporating from the interior of the samples. For some bio-samples, thermal drying may lead to the formation of a thin film on the surface of the samples, which also prevents moisture from evaporation from the interior of the samples. All these changes in physical properties of the samples distort the kinetics of the drying process.

For micro or nano-porous materials, thermal drying is proven to be inefficient, e.g. for single wall carbon nano tube (SWCNT) samples, water cannot be completely liberated or detected if the drying temperature is not high enough. But in this particular case, a higher drying temperature is not suitable for the samples because the samples may register mass gain after initial moisture loss if prolonged drying time or elevated temperatures (120 °C) are used [21]. This mass gain is probably due to the oxidation of catalytic metal content in the SWCNT. Therefore the oven may need to be purged with dry nitrogen to minimise this effect. In general, a higher drying temperature may lead to chemical decomposition of a sample. So the drying temperature must be carefully chosen. Alternatively, a vacuum drying process can be considered for micro or nano-porous materials.

Finally, although LoD method is the most widely recognised method for moisture determination, it is time consuming since the heating period can last several hours, even several days. Therefore, LoD is practically unsuitable for the purpose of automatic control purpose or in-situ monitoring.

7 SUMMARY

The presence of moisture in materials can impact on almost any product in a vast range of sectors and research fields. Therefore moisture content measurement in solids is important in terms of property, processing, quality control and storage of the materials. The literature survey on British Standards (BS), European Standards (EN), Romanian Standards (SR), International Standards (ISO) and relevant journal papers reveals that up to now, majority of moisture content measurement is based on oven drying method (LoD) due to its simplicity and fundamentality. This method is also widely used as a calibration or reference method against other indirect techniques, such electric conductance, capacitance, IR, microwave methods etc.

From the literature survey, it appears that there is no significant development in this technique in recent years. Only minor modifications to the drying process were found, e.g. in some cases, a vacuum oven and a microwave oven were used to replace the traditional oven. In another case, the sample was packed in a column in the oven and a stream of dried air was passed through the column to remove humidity influence on the drying process. Another finding in the survey is that no traceability of the LoD method is addressed in the standards, even for those standards used as a reference method.

Although LoD technique is the most widely used method for moisture determination, a number of problems associated with this technique have been identified. For example, this measurement is affected by factors like sample preparation and handling, temperature, pressure, humidity and drying duration in the oven, temperature and humidity in ambient environment etc. In addition, LoD method cannot differentiate water vapour from other volatile substances.

In search for a water specific technique to be used as a primary water content measurement standard, vapour evolution technique has shown some promising potentials for its high sensitivity, non-destructive approach and minimum effort required from analysts. However before this technique is accredited for this purpose, a lot of validation work and uncertainty analysis need to be done, which will be the part of the future work of Metefnet project.

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