NPL
National Physical Laboratory

NPL REPORT ENG 40

CONSULTATION ON THE USE OF CERTIFIED REFERENCE MATERIALS FOR THE CALIBRATION OF MOISTURE MEASUREMENT INSTRUMENTATION

Carroll, P A and Bell, S A

SEPTEMBER 2012

National
Measurement
System

CONSULTATION ON THE USE OF CERTIFIED REFERENCE MATERIALS FOR THE CALIBRATION OF MOISTURE MEASUREMENT INSTRUMENTATION

P A Carroll and S A Bell
Engineering Measurement Division

ABSTRACT

NPL has completed a study and consultation of users and instrument manufacturers in 2010 to 2011, in order to identify existing practices and needs when using certified reference materials (CRMs) to calibrate moisture measurement instrumentation.

The purpose of this consultation was to find out the calibration methods currently applied to the range of instrumentation being used for measurements of moisture (or water) content in materials across industrial sectors and to understand the areas where user needs for measurement traceability are not met.

It was found that the use of CRMs for calibration of moisture (or water) content instrumentation was not a widespread practice across all industrial sectors or instrumentation types used. Only respondents who used Karl Fischer titration were satisfied that suitable CRMs for were available. Other respondents generally expressed dissatisfaction with the suitability, stability, and other features of existing CRMs.

From analysis of responses, it was concluded that a powdered CRM applicable to foodstuff, polymer and pharmaceutical manufacturing would be of the greatest benefit to UK industry.

ISSN 1754-2987

National Physical Laboratory
Hampton Road, Teddington, Middlesex, TW11 0LW

Approved on behalf of NPLML by Graham Machin, Science Area Leader,
Temperature and Humidity group, Engineering Measurement Division

EXECUTIVE SUMMARY

This report describes work carried out within Deliverable 2 of Project TH6 of the National Measurement System Programme for Thermal Metrology.

NPL has completed a study and consultation of users and instrument manufacturers during 2010 to 2011 in order to identify existing practices when using certified reference materials (CRMs) to calibrate instrumentation for measurement of moisture in materials. The study covers all interpretations of moisture content (either water or all moisture including other volatiles).

The purpose of this consultation was to find out the calibration methods currently applied to the range of instrumentation being used for measurements of moisture content in materials across industrial areas and to understand the areas where user needs for measurement traceability are not met.

It was found that the use of moisture or water CRMs for calibration of instrumentation was not a widespread practice across all industries or instrument types used.

Only respondents who used Karl Fischer titration were satisfied that CRMs currently available were fully suitable for use with their measurement technique.

The survey results suggest that further needs for CRMs for moisture or water content include:

- CRMs compatible for use with microwave and electrical (resistance or capacitance) based instruments
- CRMs compatible with instruments which measure agricultural produce, construction and polymeric and composite materials
- CRMs that are representative of powders, pharmaceuticals and agricultural produce
- CRMs with longer lifespans and stabilities than those currently available, particularly representing powders, pharmaceuticals and foodstuffs
- Cheaper alternative water content CRMs for use with Karl Fischer titrators
- CRMs with defined surface, capillary and chemically bound water contents, of most interest to those measuring polymeric/composite and wood materials
- Development of CRMs with defined moisture profiles, of particular interest to electrical resistance/capacitance instrumentation users

In order to identify areas of greatest possible impact, the survey results were weighted according to the economic importance of the UK sectors concerned, to produce an approximate ranking to prioritise potential actions.

It was concluded that a powdered CRM applicable to foodstuff, polymer and pharmaceutical manufacturing would be of the greatest benefit to UK industry.

CONTENTS

1 INTRODUCTION

Certified reference materials (CRMs) available for moisture content are relatively few, and specialised to suit particular applications or measured products. Previous studies carried out by NPL [1, 2] concluded that support for moisture measurement traceability for use with physical measurement techniques could be provided by extending the range of CRMs available with certified moisture / water contents.

NPL has completed a consultation of users and instrument manufacturers in order to identify existing measurement practices where CRMs for moisture / water content are used to calibrate moisture measurement instrumentation.

This was in order to understand the industrial need for CRMs for water / moisture content, and to identify the characteristics that potential candidate materials for use as CRMs would need to have in order to meet the existing measurement needs.

The study applies to all interpretations of moisture content (either water or "all moisture" including other volatiles). Moisture instruments measure one or the other of these, and this is significant in measurement practice [1, 2]. The results reported here reflect the interests of users in both variants of the measurand, but the consultation did not depend on this distinction, and did not focus on it.

2 SELECTION OF PARTICIPANTS

Potential participants for the consultation were identified from a variety of sources. A list of contacts in the field of moisture measurement has been maintained and added to following previous consultations [1], [2] made by NPL on the subject of moisture measurement standards. External promotion of the consultation was also achieved through the websites www.TempSensorNEWS.com, www.MeasurementMedia.com, www.instmc.org.uk, www.npl.co.uk and through the *_connect* online knowledge transfer platform. Participants were mainly, but not exclusively, from the UK.

Further participants from moisture measurement instrumentation manufacturers and companies offering moisture content calibration services on the UKAS website were contacted after being identified through internet searches.

3 CONSULTATION PROCESS

The majority of participants answered questions from a detailed online questionnaire using the SurveyMonkey online survey tool. Some participants were interviewed in person on company visits or by telephone. The responses of participants interviewed in this way were added to the online survey on their behalf so that their answers could be included in the analysis of the overall results performed using the SurveyMonkey platform.

The questionnaire covered company details, materials measured, types of instrumentation used and if CRMs for moisture / water content are currently used as part of a calibration process. The factors preventing the use of CRMs for moisture / water content with certain techniques were investigated and information about the problems faced in practice by those using them was also obtained.

The results variously reflect the interests of users in measurements purely of water content, or of moisture content (including other volatiles).

The survey questions are shown at Appendix A.

4 SURVEY PARTICIPANTS

A total of 66 participants took part in the survey with various levels of response. The following charts are in the form directly output from the SurveyMonkey platform.

The participants fell into the following categories:

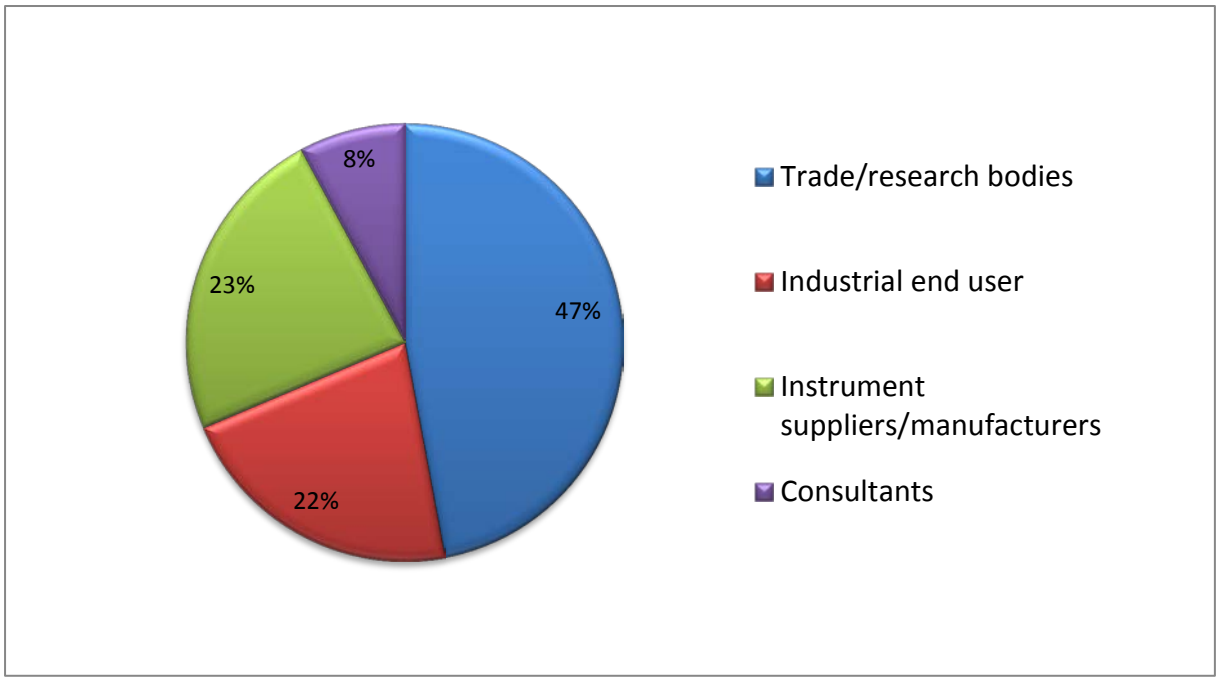


Figure 1 Pie chart showing categories of participants.

The majority of the participants were from trade / research bodies, possibly reflecting the fact that responses came from a number of scientist across NPL and from other NMIs worldwide.

5 RESPONSES TO SURVEY QUESTIONS

Question 1: (More than one answer selectable) 51 respondents of the 66 who supplied contact details went on to answer this question. **(n=51)**

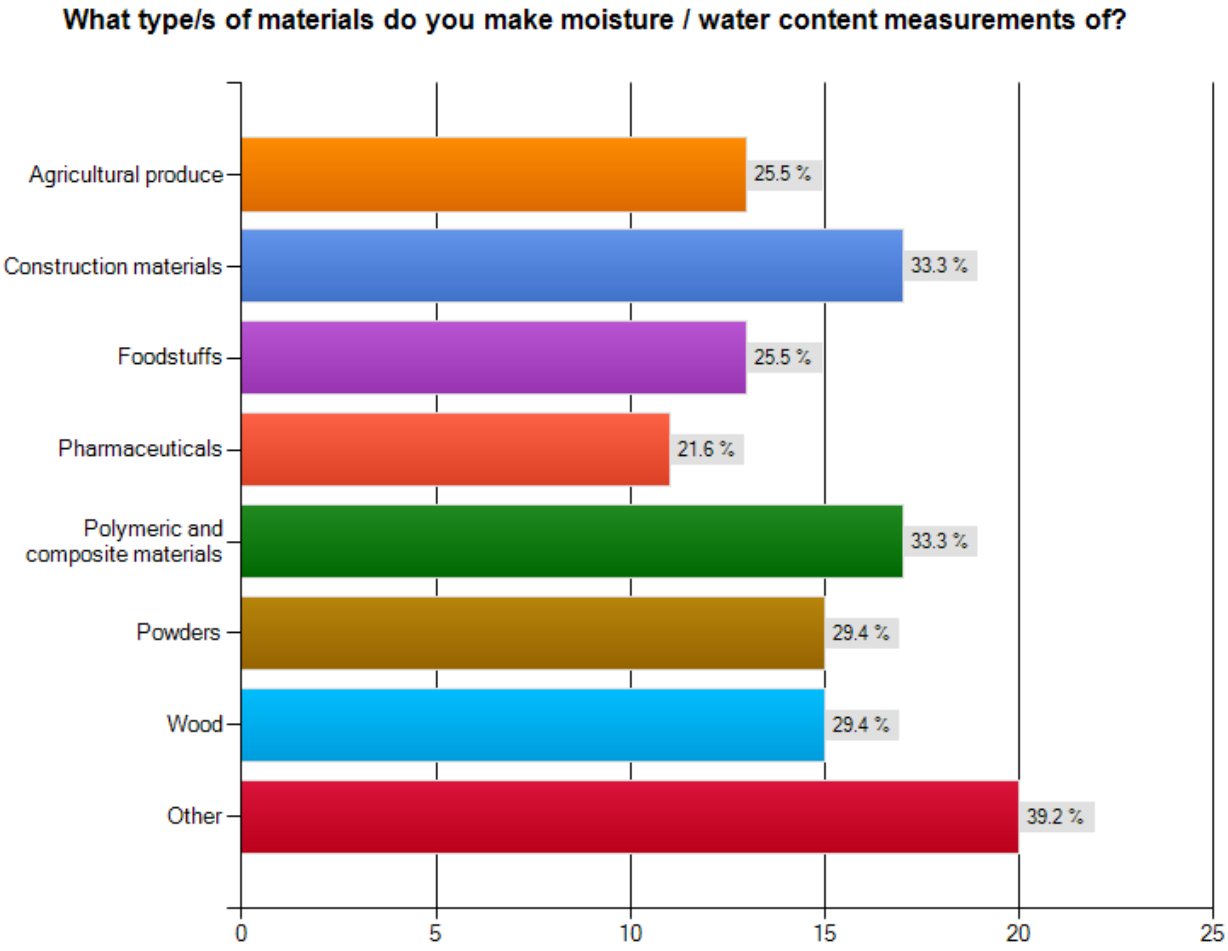


Figure 2 Bar chart showing material types measured by survey participants.

The broad spread of material types measured in the results of this question show that the survey results represent a range of industrial sectors, and that no single category of material is dominant.

The most popular answer to question was to suggest “other” materials which the respondent made moisture content measurements of. This demonstrates that there are many material types / industries to which measurements of moisture content are of importance. However the combined “other” responses did not greatly exceed the responses for each specific material, which suggests that the main relevant material types have been identified.

Other materials measured included: Soil, freeze-dried biomaterials, electronic components / circuit boards, tobacco, oils, paper, human hair, skin and processed wood (chips).

Construction and polymeric / composite materials were the most measured material types of those offered as selectable answers to the question.

Question 2: (More than one answer selectable) **n=50**

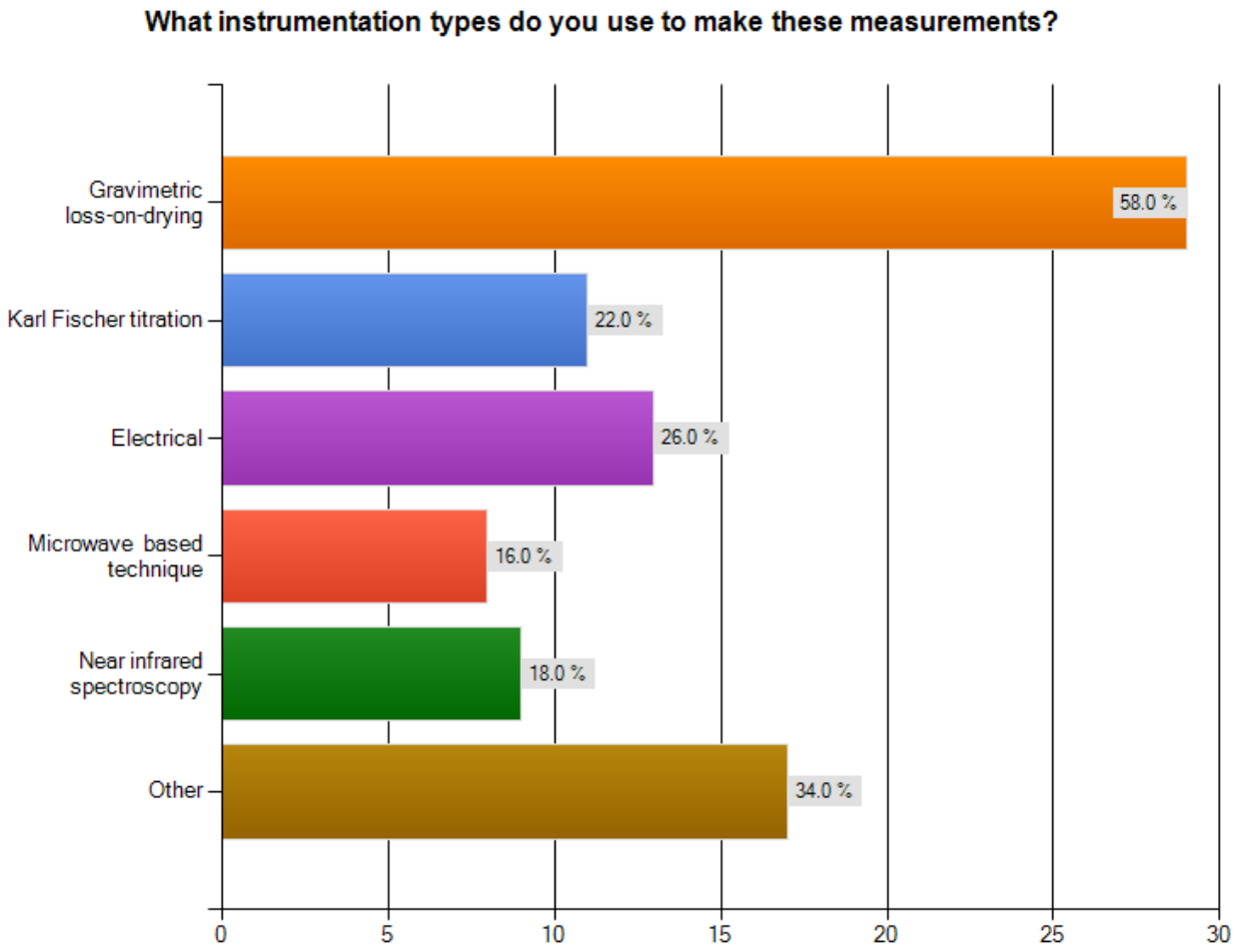


Figure 3 Bar chart showing the reported instrumentation types used by participants to measure moisture content.

In answer to this question, 29 of the 50 respondents reported using gravimetric loss-on-drying to measure moisture / water content, making it the most popular measurement technique.

Of the 17 respondents who reported “other” instrumentation types, two were in fact users of gravimetric loss-on-drying, and three were users of electrical (resistance/capacitance) meters – making electrical instrumentation the second most popular instrument type used by those surveyed.

Other measurement principles used to measure moisture content included: Dynamic vapour sorption (DVS), equilibrium relative humidity (ERH), nuclear magnetic (NMR) relaxometry, thermo-gravimetric analysis with evolved gas analysis, tuneable laser diode systems, coulometric analysis of evolved vapour and isothermal gas perfusion calorimetry.

Question 3: (More than one answer selectable) **n=51**

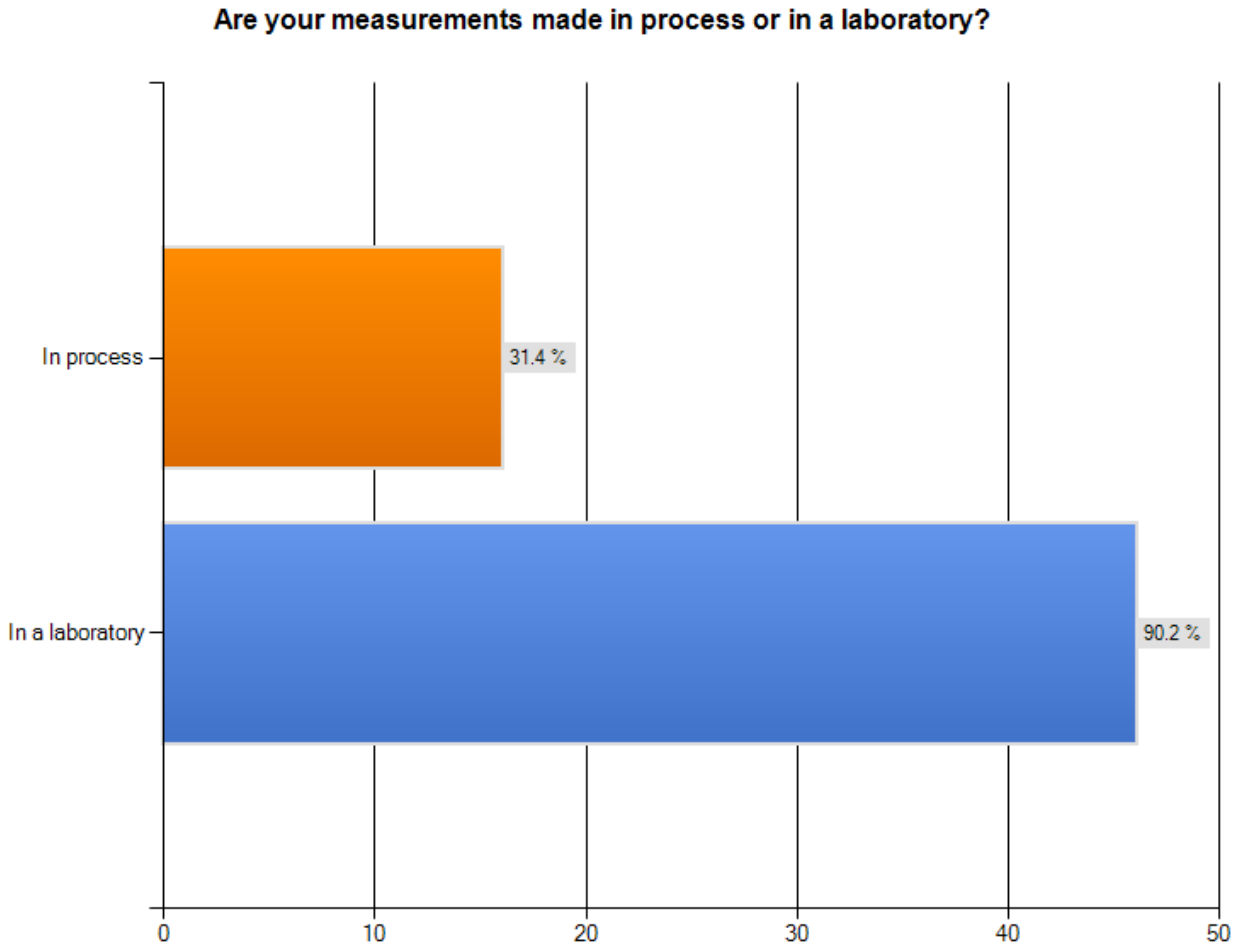


Figure 4 Bar chart showing where participants are making moisture measurements.

Concerning place of use, 46 of the 51 respondents made laboratory based measurements with 11 of these also making process based measurements. Only 5 respondents made process based measurements only.

Techniques used only in laboratories included: Karl Fischer titration, thermal gravimetric analysis (TGA) with evolved gas analysis, dynamic vapour sorption (DVS), tuneable diode laser systems, NMR relaxometry, Isothermal gas perfusion calorimetry and dynamic mechanical analysis.

Techniques used both in laboratories and in process situations included: gravimetric loss on drying, electrical (resistance / capacitance) instruments, microwave based techniques, near infrared (NIR) spectroscopy and ERH / water activity measurement.

NIR spectroscopy, electrical and gravimetric loss-on-drying were the main techniques used by those only making process based measurements.

Question 4: (More than one answer selectable) **n=51**

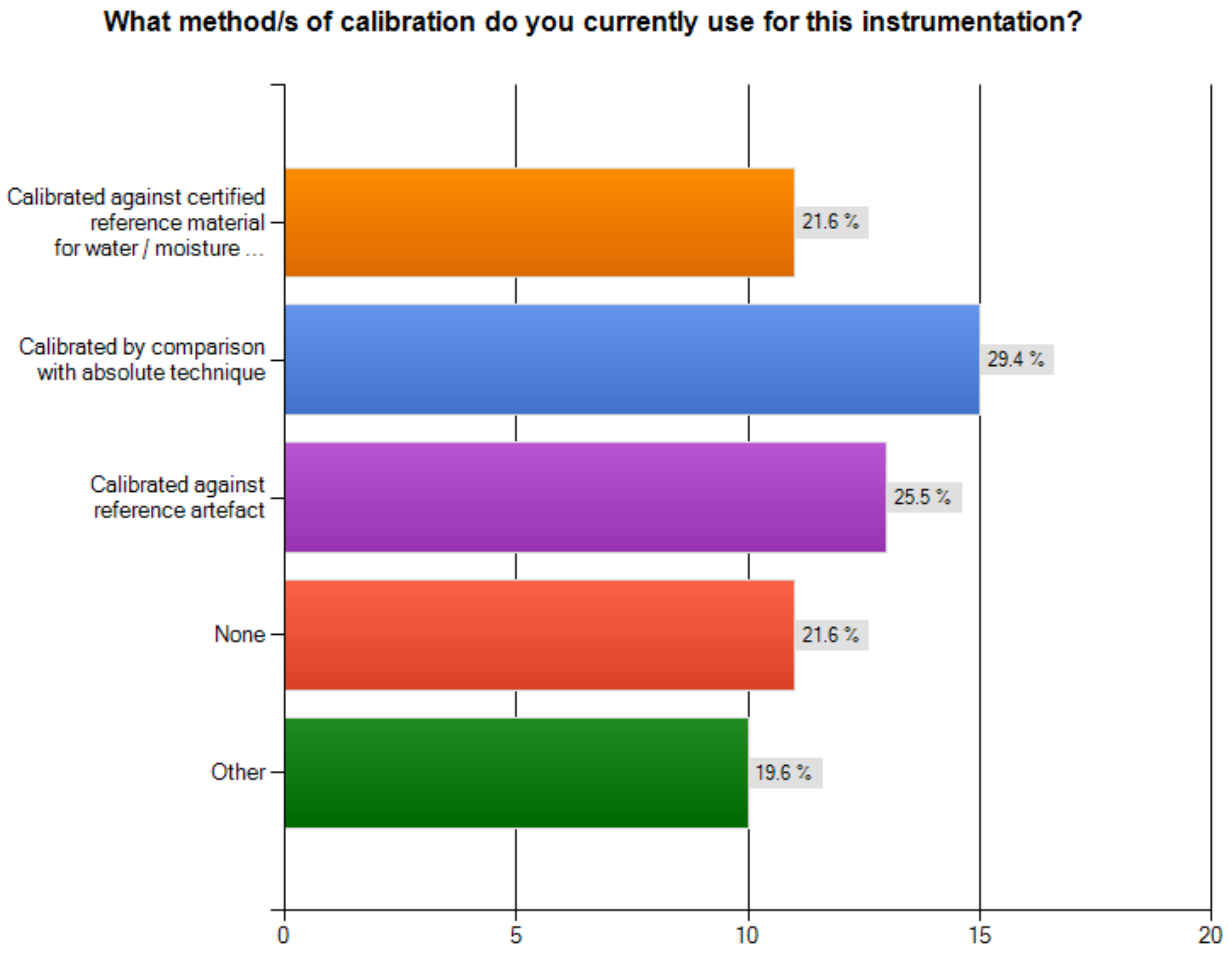


Figure 5 Bar chart showing how participants calibrate their instrumentation.

Other methods of calibration included the use of saturated salt solutions for those using ERH instrumentation. Seven of the "other" answers given could be included in the calibration against reference artefacts answer (e.g. calibration of balance against mass standards).

This would make calibration against a reference artefact the most popular calibration method, which might be expected given that the most popular measurement technique used was gravimetric loss-on-drying. This may reflect a view of a reference artefact being a reference mass (weight), rather than a reference object of known water or moisture content

The second most popular method of calibration, which applied to all instrumentation types, was to compare the response of the instrument measurement of the same material by so-called absolute techniques. The absolute techniques used for comparison (where stated) were gravimetric loss–on–drying and Karl Fischer titration.

Question 5: (More than one answer selectable) **n=40**

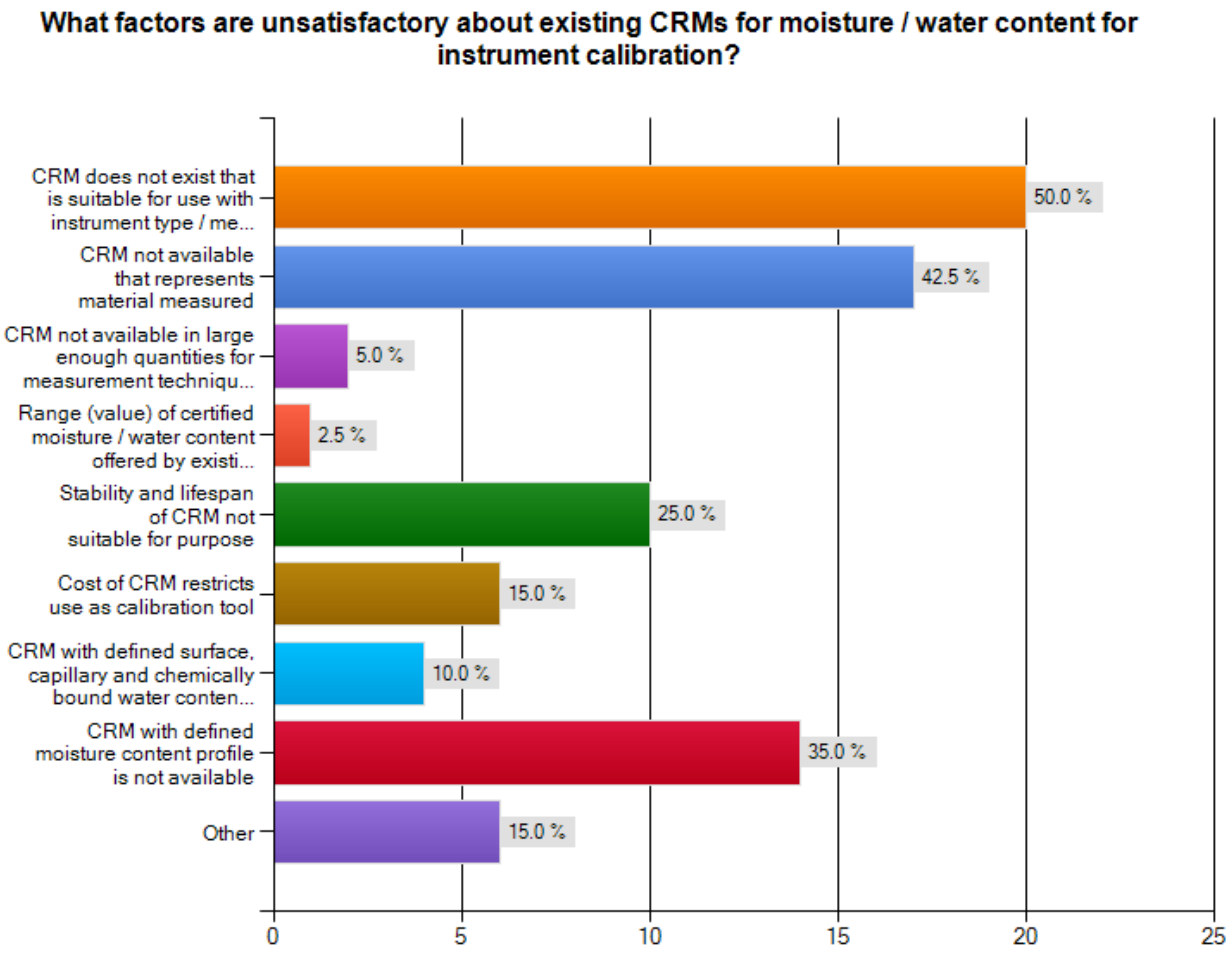


Figure 6 Bar chart representing factors that make CRMs for moisture / water content unsuitable for participants to use as calibrants.

Most of the "other" responses could be considered as "CRM not available that represents material measured" selections making this and "CRM does not exist that is suitable for use with my instrument type / measurement technique" the dominant reasons that respondents found CRMs for moisture / water content unsuitable for use when calibrating their instruments.

Karl Fischer titration users were the only respondents happy that suitable CRMs for moisture / water content for their technique were already in existence.

Availability of CRMs with a longer lifespan / stability and with a defined moisture profile were the next most important factors that would make CRMs for moisture / water content more attractive to the respondents for use as calibrants.

The measurement technique most used by those with a need for a CRM with a defined moisture profile was electrical instrumentation; with construction materials the most measured material type by this measurement technique.

Question 6: (More than one answer selectable) **n=29**

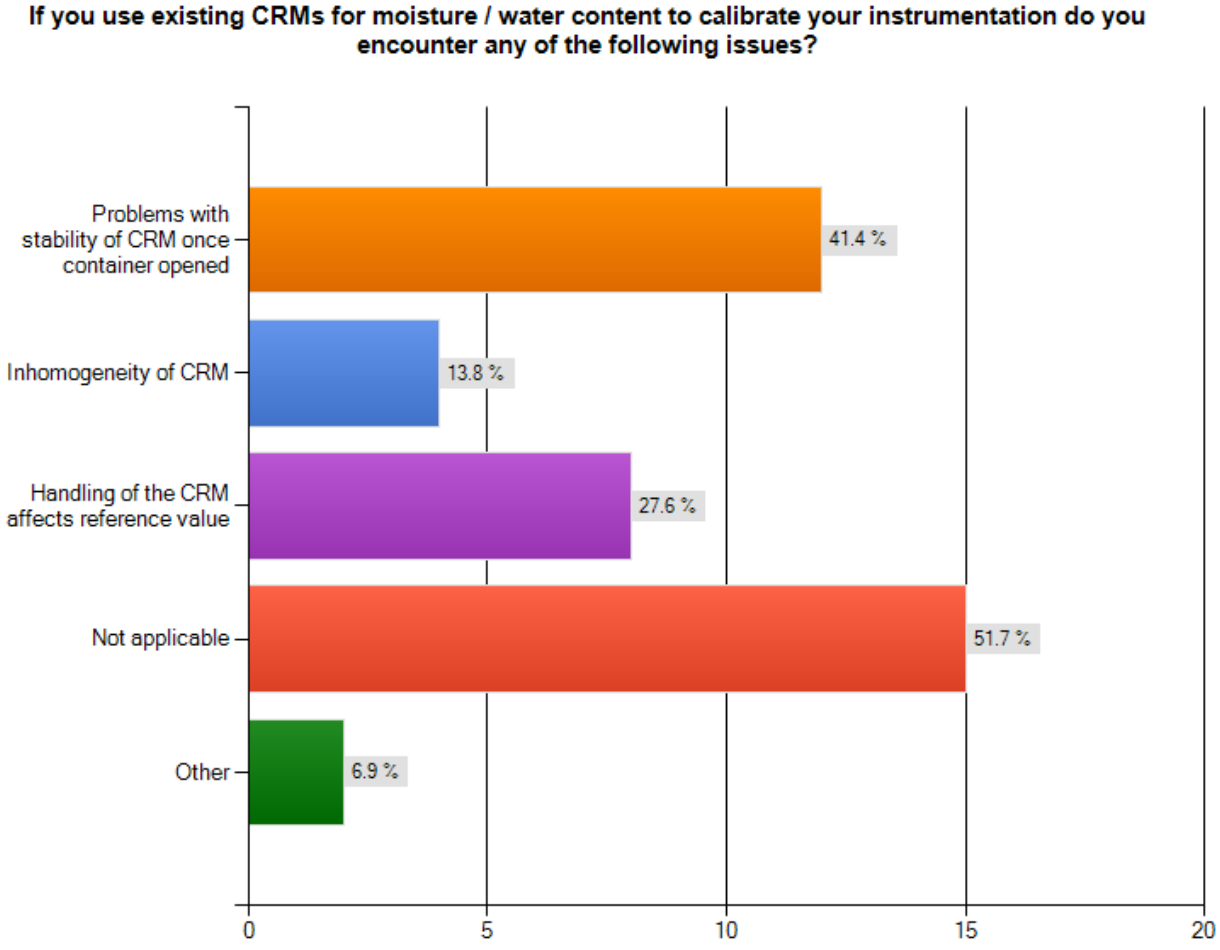


Figure 7 Bar chart representing difficulties faced by participants that currently use CRM's for moisture calibration.

Of the 51 survey respondents who went on to answer the majority of survey questions only 29 answered this question. This could be taken to mean that the response of the remaining 22 respondents found this question 'not applicable'. If this was believed to be the case, then only 14 of the respondents had experience of using CRMs for moisture / water content with their instrumentation.

The most common measurement problems encountered by those who used CRMs were their stability once opened and the fact that handling the CRM affects its reference value.

Those currently using CRMs for moisture / water content were evenly spread across material types measured and instrumentation used.

Question 7:

Do you have any suggestions for candidate materials that could be trialled by NPL as potential certified reference material where an industrial need exists?

Suggested candidate materials included:

Wood, paper, silica gel beads, cellulose and microcrystalline cellulose (as used in BCR-302, a certified reference material for water sorption analysis).

Question 8:

Can you suggest any other contacts who NPL should be talking to about this subject?

Seven contacts were suggested. NPL already had established links with four of these (TRADA, LGC, TEWS Elektronik, and TA Instruments). The survey was sent to three additional measurement user companies.

Question 9:

Would you like NPL to contact you about measurement of moisture/water content?

There were 15 positive responses to this question. The respondents who indicated that they would like to be contacted by NPL have been contacted and they include:

- A university
- Electrical (resistance/capacitance) moisture/water content instrument manufacturers
- An in process instrument manufacturer
- A research body specialising in the measurement of transport and thermodynamic properties of fluids
- A national measurement institute
- A biological standards institute
- An environmental consultancy firm
- A timber manufacturer
- An environmental protection institute

The leads generated by the survey have been followed up with personal visits to companies to discuss measurement problems and offers of test work / consultancies where applicable.

Question 10:

In addition NPL can: Visit you free of charge to discuss your moisture measurement problems. Make measurements of the moisture content of materials. Offer the opportunity of extended help via a subsidised consultancy through NPL's Technology Innovation Fund Details of specific needs:

There were five responses to this question. The respondents have been contacted to discuss their requirements further.

Needs included:

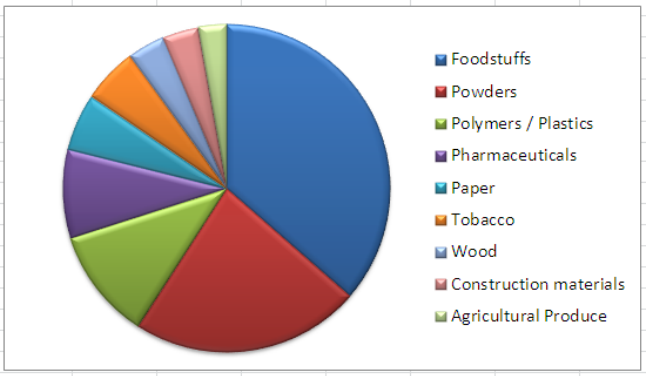
- Calibration of moisture meters to obtain certificates that can be presented to our clients as verification of our measurements

- Measurements of the moisture content of materials (foodstuffs, agricultural produce, powders)
- Reference material preparation consultancy
- Characterisation methods of varying moisture contents
- Offer of collaboration on projects involving composite moisture measurement.

6 SECTOR IMPORTANCE TO UK MANUFACTURING

Table 1 Data collated from UK Office for National Statistics Annual Business Survey 2009R – Section C: Manufacturing Results for 2009, released June 2011. GVA is "gross value added".

Survey sector	2009 turnover £millions	2009 GVA £millions	% of UK manufacturing turnover
All	448,517	130,227	
Foodstuffs	67,789	18,498	15.11
Powders	42,221	15,735	9.41
Polymers / Plastics	20,557	6,448	4.58
Pharmaceuticals	16,261	7,828	3.63
Paper	10,475	2,688	2.34
Tobacco	10,132	1,677	2.26
Wood	6,938	2,059	1.55
Construction materials	6,582	1,522	1.47
Agricultural Produce	5,236	1,223	1.17



Information from the UK National Office of Statistics was used to weight the sector responses in order of the percentage of UK manufacturing turnover. The most recent available figures were for 2009, made available in June 2011.

The data shown in Table 1 is only indicative of the importance of each of the survey sector categories. Some sectors were hard to directly match to industries as classified in the Annual Business Survey results by the UK Standard Industrial Classification 2007, so best approximate categorisations were made..

Foodstuffs were by far the largest contributing sector to UK manufacturing turnover of those surveyed. Powdered materials are shown to be widely used in UK manufacturing with polymer / plastic and pharmaceutical production ranked the next most important sectors.

The data highlights that tobacco manufacturing contributes more to UK manufacturing turnover than the wood, construction and agricultural produce sectors.

In order to consider how to derive most UK impact from selective development of new moisture CRMs, the UK manufacturing turnover results were used to weight the survey results relative to sectors as shown in Tables 2 and 4.

Table 2 (From answers to survey questions 1 and 5) CRM use problems experienced by sector / material type measured as percentage of total response, and weighted by importance of sector to UK manufacturing turnover

	CRM does not exist that is suitable for use with instrument type	CRM not available that represents material measured	CRM not available in large enough quantities for measurement technique	Range (value) of certified moisture / water content offered by existing CRMs not applicable for my process	Stability and lifespan of CRM not suitable for purpose	Cost of CRM restricts use as calibration tool	CRM with defined surface, capillary, and chemically bound water contents not available	CRM with defined moisture content profile is not available	Other
SECTOR (Number of respondents)									
As percentages of response									
Agricultural Produce (12)	67	42	8	0	8	0	17	33	8
Construction Materials (15)	47	27	7	7	33	20	13	33	27
Foodstuffs (11)	45	18	9	0	45	18	9	18	18
Pharmaceuticals (11)	45	36	0	0	45	18	9	27	18
Polymeric and Composite Materials (15)	53	27	13	0	33	7	20	33	13
Powders (13)	46	38	8	0	46	15	8	31	15
Wood (13)	46	31	8	0	23	0	23	31	15
Other (17)	35	41	0	0	29	18	12	35	18
Responses weighted with factor derived from sector % of UK manufacturing turnover	Weighted by UK T % "Impact factor"	Weighted by UK T % "Impact factor"	Weighted by UK T % "Impact factor"	Weighted by UK T % "Impact factor"	Weighted by UK T % "Impact factor"	Weighted by UK T % "Impact factor"	Weighted by UK T % "Impact factor"	Weighted by UK T % "Impact factor"	Weighted by UK T % "Impact factor"
Agricultural Produce (12) 1.2 % UK Turnover	80	50	10	0	10	0	20	40	10
Construction Materials (15) 1.5 % UK Turnover	70	40	10	10	50	30	20	50	40
Foodstuffs (11) 15.1 % UK Turnover	686	275	137	0	686	275	137	275	275
Pharmaceuticals (11) 3.6 % UK Turnover	164	131	0	0	164	65	33	98	65
Polymeric and Composite Materials (15) 4.6 % UK Turnover	245	123	61	0	153	31	92	153	61
Powders (13) 9.4 % UK Turnover	434	362	72	0	434	145	72	289	145
Wood (13) 1.6 % UK Turnover	74	49	12	0	37	0	37	49	25
Other (17) 4.6 % UK Turnover	162	189	0	0	135	81	54	162	81

Table 3 (From answers to questions 2 and 5) CRM problems experienced by instrument type used, as percentage of total response

	CRM does not exist that is suitable for use with instrument type	CRM not available that represents material measured	CRM not available in large enough quantities for measurement technique	Range (value) of certified moisture / water content offered by existing CRMs not applicable for my process	Stability and lifespan of CRM not suitable for purpose	Cost of CRM restricts use as calibration tool	CRM with defined surface, capillary, and chemically bound water contents not available	CRM with defined moisture content profile is not available	Other
FILTER BY INSTRUMENT TYPE (number of respondents)									
As percentages of response									
Gravimetric loss on drying (24)	54	50	4	4	33	17	13	46	4
Karl Fischer Titration (8)	0	50	13	0	50	38	13	13	13
Electrical (11)	64	45	9	9	45	9	9	55	9
Microwave based Technique (8)	75	25	13	0	38	13	25	38	13
Near Infrared Spectroscopy (9)	33	33	11	0	33	11	11	22	33
Other (14)	50	43	7	0	29	14	14	29	21

Table 4 (From answers to survey questions 1 and 2) instrument type used for measurement in each sector as percentage of total response, and weighted by importance of sector to UK manufacturing turnover

Instrumentation Type Used		Gravimetric loss on drying	Karl Fischer Titration	Electrical	Microwave based Technique	Near Infrared Spectroscopy	Other
As percentages of response							
Agricultural Produce (13)		77	15	38	31	38	31
Construction Materials (17)		71	29	47	24	29	29
Foodstuffs (13)		77	31	23	31	31	46
Pharmaceuticals (11)		55	36	18	18	36	64
Polymeric and Composite Materials (16)		69	31	31	25	31	38
Powders (15)		67	33	20	20	27	27
Wood (15)		73	20	40	27	27	47
Other (19)		53	37	26	11	11	37
Responses weighted with factor							
derived from sector % of UK manufacturing		Weighted by UK T %	Weighted by UK T %	Weighted by UK T %	Weighted by UK T %	Weighted by UK T %	Weighted by UK T %
turnover	**% UK T**	"Impact factor"	"Impact factor"	"Impact factor"	"Impact factor"	"Impact factor"	"Impact factor"
Agricultural Produce (12) 1.2 % UK Turnover	1.2	92	18	46	37	46	37
Construction Materials (15) 1.5 % UK Turnover	1.5	106	44	71	35	44	44
Foodstuffs (11) 15.1 % UK Turnover	15.1	1162	465	348	465	465	697
Pharmaceuticals (11) 3.6 % UK Turnover	3.6	196	131	65	65	131	229
Polymeric and Composite Materials (15) 4.6 % UK Turnover	4.6	316	144	144	115	144	173
Powders (13) 9.4 % UK Turnover	9.4	627	313	188	188	251	251
Wood (13) 1.6 % UK Turnover	1.6	117	32	64	43	43	75
Other (17) 4.6 % UK Turnover	4.6	242	169	121	48	48	169

7 DISCUSSION

7.1 MOISTURE TRACEABILITY PRACTICES

The survey confirmed that moisture content measurements are being made across a wide range of industries. If an improvement in traceability (CRM) provision in this area is to be pursued, with limited resources, then any new development should either focus on a specific industry where high impact can be achieved, or on trying to develop a multi-purpose material that has more than one application.

The measurement technique most used by respondents was gravimetric loss-on-drying. Users of this approach could use a certified reference material for water / moisture content, but they typically do not. It is widely considered sufficient to calibrate the balance used against reference weights with traceability to national standards for mass, and that this underpins an absolute technique. As such, loss on drying is often used as the method against which other secondary (inferential) measurement methods are calibrated, and is also a measurement method commonly used when certifying materials in terms of their moisture content. However, this approach remains “non-standard” in the sense that it is often method-dependent - with a different temperature-time protocol for every material. If suitable CRMs were available, there would be benefit in using them in support of loss-on-drying measurements. In particular, any errors due to incomplete drying or other imperfections in method might not come to light through traceable weighing, but could well be revealed by using CRMs.

Karl Fischer titration also is considered an absolute technique but in this case certified reference materials for water content are frequently used to check or validate the performance of the instruments. [3]

Inferential (indirect) measurement techniques such as electrical (resistance / capacitance), microwave and near infrared spectroscopy measurements represented 34 % of the instrumentation types used. Techniques such as these use checks against reference artefacts (resistance checks / optical reflectance checks) as well as CRMs to validate the performance of the instrument.

Among respondents, those using CRMs for moisture / water content to calibrate their instrumentation would be most likely to be measuring pharmaceuticals and powders, with Karl Fischer titration the most likely instrument type to be calibrated this way.

Those using NIR spectroscopy, microwave based techniques and Karl Fischer titration were not amongst those answering that they had no current method of calibration. However, the use of NIR optical checks does not provide measurement traceability in terms of the measured quantity.

With only 14 of the 66 respondents reporting use of CRMs for moisture / water content to calibrate their instruments, it is clear that this is currently not a common calibration method.

Major reasons cited for the unsuitability of CRM use for instrument calibration were that a CRM representative of the material measured was not available and that a CRM did not exist that was compatible with the instrument type used.

Stability of CRM moisture values once their containers were opened and subsequent material handling affecting the reference value were main problems encountered by those using CRMs for moisture / water content.

Tables 2, 3 and 4 show a more detailed breakdown of which factors about existing CRMs made them unsuitable for use in different sectors and with different instrument types, derived from responses to Questions 1, 2 and 5.

Table 4 shows a breakdown of the material types measured by each instrument type, derived from responses to Questions 1 and 2. From this analysis it can be concluded that:

- Gravimetric loss-on-drying is most commonly used to measure agricultural produce, foodstuffs, construction materials and wood.
- Karl Fischer titration is most commonly used to measure powdered materials and pharmaceuticals.
- Electrical (resistance or capacitance) instrumentation is most commonly used to measure agricultural produce, construction materials and wood.
- Microwave based instrumentation is most commonly used to measure agricultural produce, foodstuffs, polymeric / composite materials and wood.
- NIR spectroscopy is most commonly used to measure agricultural produce, pharmaceuticals and polymeric / composite materials.
- Instrumentation types other than those listed are most commonly used to measure pharmaceuticals (e.g. those listed in the responses to question 2).

A few respondents answered the questions of the survey from a moisture in gas (humidity) measurement perspective. Measurement standards for water content of air, with provision of calibration of hygrometers, are long established at NPL, and development of humidity calibrations in other gases is a current area of research. The survey was not meant to cover needs related to moisture measurement in gases –this could perhaps have been emphasised in the questions of the survey.

It should be noted that immediate responses of participants at the time of survey might not always be a complete or accurate reflection of their practices or requirements.

7.2 IDENTIFIED NEEDS FOR CRMS BEYOND WHAT IS AVAILABLE

From the response summaries shown in Tables 2 and 3 specific requirements that can be identified are:

- **Development of CRMs compatible for use with microwave, electrical and gravimetric loss-on-drying instrument types** as these users felt most strongly that suitable CRMs were not available for them.
- **CRMs suitable for use with the measurement techniques used in measuring agricultural produce, construction and polymeric / composite materials**
- **CRMs that represent powders, pharmaceuticals and agricultural produce** which were the materials that users felt were least represented by those currently available.
- **Development of CRMs with longer lifespan and stability**, particularly for those measuring powders, pharmaceuticals and foodstuffs that are applicable for use with Karl Fischer titration, electrical and microwave based instrumentation.
- **A cheaper alternative CRM for water content for use with Karl Fischer titration.**
- **A polymeric / composite or wood based CRM with defined surface, capillary and chemically bound water content.**
- **A CRM (or reference artefact) with a defined moisture profile**, of interest to all the material types covered by the survey and particularly users of electrically sensing instrumentation.

From weightings by UK economic importance, as shown in Tables 2 and 4, priority areas of impact can be identified:

1. First priority – more or better CRMs suitable for instrument type – especially for food, powders, polymers (and fairly strongly for pharmaceuticals).
2. Second priority – better stability and/or lifespan of CRMs – especially for food and powders (and fairly strongly for pharmaceuticals and polymers)
3. Joint third priority:
    a. CRMs appropriately representing materials to be measured – especially for food and powders (and fairly strongly for pharmaceuticals, polymers)
    b. CRMs giving moisture profile information – especially for food, powders (and fairly strongly for polymers and composites). It would need to be investigated what constitutes useful moisture profile information for a powder.

8 CONCLUSION

From the results of the survey we have gained a greater understanding of:

- measurement techniques for which calibration using certified reference materials (CRMs) for water / moisture content is currently not used.
- reasons why such CRMs are perceived to be unsuitable for the calibration of certain instruments.
- types of materials for which CRMs for water / moisture content do not currently exist.
- existing problems encountered by those using these CRMs for calibration of moisture / water content measurement instrumentation.
- potential candidate reference materials that could be considered, to address the measurement issues raised in the survey responses.

We have also:

- raised the profile of the moisture/water content measurements currently undertaken.
- made initial contact with several new organisations, and followed up with visits or technical advice.
- introduced NPL's Technology Innovation Fund to contacts who may not have previously been aware of it.

The consultation revealed different CRM requirements across the sectors surveyed. Material type, amount of material and moisture content range requirements were all found to vary depending on industrial sector.

Ideally the most beneficial CRMs to develop for UK industry would be those that could be used widely across main industrial sectors and instrument types.

Foodstuff manufacturing is by far the largest value turnover of the sectors surveyed. Powders, generally, are also an area of high impact. From this perspective, the development of a powdered CRM with as wide a range of moisture contents as possible that was compliant with food safety and hygiene regulations would appear to be of the most benefit. If this powdered material could also be used by those in the polymer and pharmaceutical manufacturing sectors then this would be of significant added benefit.

The project will now continue with an investigation into materials that can be characterised to meet the needs identified in the survey and have the potential to be developed as a future certified reference material for moisture / water content.

Calibration using CRMs is not the only possible approach to improve traceability and quality assurance of moisture measurements, and other mechanisms, such as instrument transfer standards, may also be usefully considered.

9 ACKNOWLEDGEMENTS

This work was funded within Project TH6 of the National Measurement System Programme for Thermal Metrology. The authors gratefully acknowledge the help of Ray Peacock and the Institute of Measurement and Control in publicising the survey, and Richard Dockree of University of Southampton for processing some of the data.

10 REFERENCES

[1] BELL, S.A. A study of the requirement for standards for the measurement of moisture in solid materials, *NPL Report CMAM 20*. 1998.

[2] CARROLL, P.A. BELL, S.A. Needs for NMS support for measurements of moisture in materials, *NPL report ENG 7*, 2008.

[3] JOSEPH, B and LE GOFF, T. Validation of the Metrohm 774 Oven Sample Processor and 831 Karl Fischer Coulometer for Moisture Determination. *LGC report LGC/AT/2006/*019, 2006.

APPENDIX A – SURVEY QUESTIONS

Certified reference materials for moisture / water content consultation:

Materials measured and instrumentation used

Q1. What type/s of materials do you make moisture / water content measurements of?

- Agricultural produce
- Construction materials
- Foodstuffs
- Pharmaceuticals
- Polymeric and composite materials
- Powders
- Wood
- Other

Q2. What instrumentation types do you use to make these measurements?

- Gravimetric loss-on-drying
- Karl Fischer titration
- Electrical
- Microwave based technique
- Near infrared spectroscopy
- Other

Q3. Are your measurements made in process or in a laboratory?

- In process
- In a laboratory

Q4. What method/s of calibration do you currently use for this instrumentation?

- Calibrated against certified reference material for water / moisture content
- Calibrated by comparison with absolute technique
- Calibrated against reference artefact
- None
- Other

Q5. What factors are unsatisfactory about existing CRMs for moisture / water content for instrument calibration?

- CRM does not exist that is suitable for use with instrument type / measurement technique
- CRM not available that represents material measured
- CRM not available in large enough quantities for measurement technique employed
- Range (value) of certified moisture / water content offered by existing CRMs not applicable for my process
- Stability and lifespan of CRM not suitable for purpose
- Cost of CRM restricts use as calibration tool
- CRM with defined surface, capillary and chemically bound water contents not available
- CRM with defined moisture content profile is not available
- Other

Q6. If you use existing CRMs for moisture / water content to calibrate your instrumentation do you encounter any of the following issues?

- Problems with stability of CRM once container opened
- Inhomogeneity of CRM
- Handling of the CRM affects reference value

- Not applicable
- Other

Q7. Do you have any suggestions for candidate materials that could be trialled by NPL as potential certified reference materials where an industrial need exists?

Q8. Can you suggest any other contacts who NPL should be talking to about this subject?