Publishable Summary for 19NRM04 ISO-G-SCoPe Standardisation of structural and chemical properties of graphene

Overview

The overall aim of this project was to validate and standardise measurement and characterisation methods for the chemical and structural properties of graphene in powders and liquid dispersions for industrial applications. This was to overcome a "what is my material?" barrier for both users and producers leading to well-characterised highly tailored graphene, graphene oxide and chemically functionalised graphene. The project aimed to maximise innovation and competitiveness of European industries across the supply chains in multiple sectors including the energy sector, photovoltaics, lithium-ion batteries, flexible electronics, composites, consumer products, novel coatings, clothing, automotive and aerospace industries. The project has undertaken 4 VAMAS international interlaboratory studies of graphene related 2D materials (GR2M) using XPS, Raman spectroscopy SEM and AFM. The project led to the publication of the first ISO graphene measurement standard (ISO TS 21356-1) and has led or contributed to the development of a further 3 draft ISO standards (ISO TS 23359. ISO TS 23879 and ISO TS80004-13). These enable end users and producers to measure structural and chemical properties of GR2M in a standardised way.

Need

Graphene and related 2D materials are predicted to make a major impact in many technology areas, either through incremental advances via current material replacement or via disruptive changes. However, the uptake of these materials into commercial products is hindered as industrially produced "graphene" is often incompletely or not correctly characterised by the 100+ suppliers. This has been acknowledged as the single biggest issue by graphene companies, suppliers and standards bodies [e.g., ISO TC229 (nanotechnologies), BSI NTI/1 (nanotechnologies) and BSI UK-China JWG on graphene standardisation]. Issues include structural determination of the material as graphene or graphite, how many layers are present and what is the flake size distribution in different batches. Chemical determination issues include the amount of oxygen present (for graphene oxide and reduced graphene oxide), impurities and functionalisation. Before the project, there were no standard ways to measure these properties for the industry to take the material from the laboratory to large-scale production.

The industry is rushing to develop its own internal measurement procedures to obtain reproducible results for its process optimisation and for external sales. These remain poorly accepted by their peers and competitors. Before the project started, there were standardisation documents progressing through International Organization for Standardization (ISO) TC229/IEC TC113 and American Society for Testing and Materials (ASTM) E56, but these were either overview technical reports, focused on electrical measurements or other issues. They did not contain validated measurement protocols for structural and chemical properties. There were also no European Standards. European industries require international documentary standardisation of structural and chemical methods to characterise graphene validated via pre-normative Versailles Project on Advanced Materials and Standards(VAMAS) international interlaboratory testing. This allows end-users to compare technical datasheets of different commercially available ‘graphene’ products worldwide. This helps instil confidence and allows faster innovation and increased R&D productivity, as end-users will only need to test a few materials rather than hundreds. These end users will be able to match highly tailored 2D materials to performance requirements. Standardised characterisation procedures are also required for companies needing to comply with new nanomaterials regulations and in particular registering graphene nanoforms in the Registration, Evaluation, Authorisation and Restriction of Chemicals (REACH) register [ECHA, 2017 Appendix R.6-1] allowing reliable toxicity testing of different products on the market.

The EMPIR initiative is co-funded by the European Union's Horizon 2020 research and innovation programme and the EMPIR Participating States

The aim of this project was to build on the established work items at International Organization for Standardization (ISO) and International Electrotechnical Commission (IEC) in terminology and overview standards and add robust metrology, methodology and supply validated measurement methods and supporting data. This will lead to the publication of three industry-critical ISO standards via ISO TC229 (ISO Technical Committee 229 “Nanotechnologies”) which will become adopted as European standards via CEN TC352 (CEN Technical Committee 352 "Nanotechnologies").

Objectives

The overall objective of this project was to validate and standardise measurement and characterisation methods for the chemical and structural properties of graphene in powders and liquid dispersions for industrial applications.

The specific objectives of the project were:

1. To lead and provide a contribution to the publication “ISO TS 21356-1 Nanotechnologies – Structural characterisation of graphene: Part 1: Graphene from powders and dispersions”. The contribution will focus on measurement methods for the characteristics of graphene flakes in order to verify measurements made on commercial graphene flake products.
2. To lead and provide a contribution to the development of ISO/PWI 23359 "Nanotechnologies - Chemical characterisation for graphene in powders and suspensions" and to lead and provide a contribution to ISO/PWI 23879 "Nanotechnologies – Structural characterisation of graphene oxide flakes: thickness and lateral size measurement using Atomic Force Microscopy (AFM) and Scanning Electron Microscopy (SEM)”. The contribution will focus on developing validated quantitative measurement methods.
3. To provide a contribution to pre-normative international interlaboratory studies in VAMAS TWA 41 (graphene and related 2D materials), leading and participating in characterisation studies focused on structural and chemical properties using, X-ray Photoelectron Spectroscopy (XPS), AFM, Raman, and SEM techniques.
4. To work closely with the European and International Standards Developing Organisations, and pre-normative organisations, and the users of the Standards they develop, including the Graphene Flagship, to ensure that the outputs of the project are aligned with their needs and incorporated into Standards at the very earliest opportunity.

Progress beyond the state of the art

When the project started, there hadn’t been any published international standards on validated measurement and characterisation methods for the structural properties of graphene and no other source of validated standard operating procedures to use in industrial applications. There has been the first version of the terminology standard on graphene and related 2D materials (ISO TS 80004-13) and an overview ISO technical report (ISO/TR 19733;2019) that provides a matrix listing key properties of graphene to measurement techniques. The consortium helped to lead the update of the terminology standard including new terms. The consortium worked well with the European and International Standards Developing Organisations, pre-normative organisations, and the users of the Standards they develop, including the Graphene Flagship to address this lack of standards (objective 4) and to ensure that the outputs of the project were aligned with their needs and incorporated into Standards at the very earliest opportunity.

Regarding objective 1, the first version of ISO TS 21356-1 was published in March 2021, with the consortium providing final editing. This was a dual logo ISO TC229 and IEC TC113 publication. This document provided a flowchart of methods to characterise graphene. This along with the terminology standard (80004-13) allowed users to determine what was graphene and then measure it. However, critically the first version of 21356-1, did not provide validated measurement protocols but only example methods as informative annexes as they had not been validated through VAMAS interlaboratory studies at that time. The project provided the industry with its much-needed validated measurement methods for the structural characterisation of commercial powders and dispersions containing graphene flakes. These will form the centrepiece of an updated revision of 21456-1 that will when published be a full international standard and include the validated methods in order to determine essential properties that industries who are supplying, purchasing and using graphene require. This included average flake size and flake size distribution, number of layers, flake thickness and level of disorder. The methods were standardised using the techniques of SEM, Raman spectroscopy and AFM. The consortium led the process to get 21356-1 revision started and agreement from ISO TC229 for it to become a full ISO standard.

At the start of the project, there were no international standards to measure the chemical properties of graphene, functionalised graphene and graphene oxide (GO). There were also no international standards for characterising the structural properties of graphene oxide, as well as no graphene or 2D materials-specific surface chemical analysis standards for example using XPS. Functionalised 2D materials were readily available to purchase but the products were uncharacterised by validated methods and hence were very likely to have different chemical compositions to those that are expected. The consortium was world-leading in the development of the first international standards in a) chemical characterisation of graphene/functionalised graphene and graphene oxide (objective 2) and b) graphene oxide structural characterisation (objective 2). Methods have been developed by the consortium, tested in-house and have been validated via international interlaboratory studies (objective 3) on i) graphene oxide measurement using SEM, ii) graphene oxide and functionalised graphene using XPS. This along with the consortium's metrological knowledge in AFM was used to lead and input into the writing of the new work item proposals for the two ISO standards on graphene oxide measurement using SEM and AFM and chemical characterisation of graphene, graphene oxide and functionalised graphene. The chemical standard (ISO TS 23359) was led by the consortium and taken through the ISO balloting process up to the committee draft ballot stage. For ISO TS 23879, structural characterization of graphene oxide flakes: thickness and lateral size measurement using AFM and SEM the consortium provided strong contributions to the SEM method section and improved the whole document.

The consortium led and drove progress in 4 pre-standardisation interlaboratory studies in VAMAS TWA 41 (objective 3). These were designed so as to write, test and improve the protocols that have formed the basis of the ISO standards, as well as understand and reduce the uncertainties in all the measurements in the industry based on the key issues. The principal measurements of graphene nanoplatelets that AFM and SEM measure are the lateral flake size and thickness. At the start of the project uncertainties for measurements undertaken in industry were thought to be due to poor scanner calibration and image analysis protocol as well as sample preparation. The interlaboratory results showed that participant flake selection when looking at industrial samples was the main source of uncertainty. Hence in the international standards, additional information on flake choice was included.

Results

Objective 1: "ISO DTS 21356-1 Nanotechnologies – Structural characterisation of graphene: Part 1: Graphene from powders and dispersions".

This ISO technical specification specifies the sequence of methods for characterising the structural properties of graphene flakes using a range of measurement techniques. The properties covered are the number of layers/thickness, lateral flake size, the level of disorder, layer alignment and specific surface area. The first version of this standard did not include standardised methods and the consortium led the final editing and proof-reading of the document. ISO TS 21356-1 was published in March 2021 as the first graphene measurement standard. The consortium wrote a review paper summarising the standard with 10 international co-authors and published in the Nature Reviews Physics Journal. Importantly, in the first version of the standard, measurement protocols were only included as informative annexes, as they have not been validated via VAMAS interlaboratory studies. The consortium then developed these essential VAMAS interlaboratory studies, particularly involving SEM/AFM and Raman spectroscopy measurements. Samples were made, tested and then sent out to participants along with a protocol to follow. Based on the results of these interlaboratory studies (objective 3), the consortium developed and validated these methods whilst understanding better and reducing measurement uncertainties. Following completion of the VAMAS studies, the consortium presented the results to ISO TC229 and arranged for the standard to be revised in ISO TC229 and importantly for it to be converted to a full international standard (IS) with normative measurement methods sections developed by the project. The objective was fully achieved.

Objective 2: ISO/PWI 23359 'Nanotechnologies - Chemical characterisation for graphene in powders and suspensions' and ISO/PWI 23879 'Nanotechnologies – Structural characterisation of graphene oxide flakes: thickness and lateral size measurement using AFM and SEM'

Before the project started, these draft standards, led by the UK and UK /China respectively, were in the preliminary work stages and had been for approximately 18 months. Progress was hampered, due to the need for the methods to be validated using international interlaboratory studies. The project partners started writing the draft standards in the first year. For ISO/PWI 23359, this involved drafting the entire document, including a flow chart, general sections and measurement method sections on XPS, ICP-MS (Inductively coupled plasma mass spectrometry), TGA (Thermogravimetric Analysis) and FTIR (Fourier Transform Infrared Spectroscopy). ISO experts from China and Australia wrote the last three, (ICP-MS, TGA and FTIR) with significant editorial contributions by the consortium. The first draft was written, and ISO TC229/JWG2 gave the go-ahead for a new work item ballot. This ballot was held between 15 July to 8 October 2021. The proposal attracted strong support and received comments/support from countries around the globe including Canada, the US and France. Ballot comments were discussed at the ISO TC229/JWG2 meetings in November 2021 and May 2022. Further discussions and input were received from international experts and consortium partners at the November 2022 ISO TC229 meeting, which was hosted by NPL in Teddington, UK. Following the meeting, the document was re-circulated for additional comments. The document was then discussed at the May 2023 meeting of ISO TC229 and following that the consortium partners updated the XPS section and the whole of the document. This was then sent out for the Committee Draft (CD) consultation ballot, which is the main technical ballot for the document. Following the end of the project, it is expected that the ISO document will be published in 2024.

For ISO/PWI 23879, the draft was written in collaboration with NMI, China. This underwent new work item ballot and was approved as an ISO work item. The consortium progressed the documents through the ISO process in collaboration with NIM with presentations at the ISO TC229 meetings. The SEM measurement method section was developed following BAM and NPL mini-interlaboratory testing. The knowledge from this helped NPL to write the SEM section and improve the text of the whole document. The document is now waiting for the completion of the full VAMAS SEM interlaboratory study led by BAM, prior to undergoing technical and editorial ballots in ISO and then publication. The objective was fully achieved.

Objective 3: Pre-normative international interlaboratory studies in VAMAS TWA 41 (graphene and related 2D materials*)*

At the start of the project, within VAMAS TWA41, there were 6 planned or active VAMAS graphene interlaboratory studies, with only 4 on graphene flakes. This project provided the focus and drive to run 4 new interlaboratory studies in a short period of time on AFM/SEM and Raman spectroscopy of graphene flakes, SEM of graphene oxide (GO) and XPS of chemically functionalised graphene leading to validated methods and reduced uncertainties. For each measurement technique, the aim was to test the method for the ISO document and to understand and reduce uncertainties for industrial measurements.

For each of the 4 studies, measurement protocols were developed, tested and improved. Importantly the samples were sourced, and sample preparation optimised. Excellent repeatable samples are the key to a successful interlaboratory study, so time was spent to produce the best samples possible. This has included the manufacture and testing of specially produced substrates with fiducial markers and a calibration grid for SEM and AFM calibration checking. Much work has been undertaken on optimising the deposition procedure for graphene flakes and graphene oxide flakes onto these substrates to have well-separated flakes. This was solved and samples were produced including those on the special substrates produced by C2N.

For the AFM and SEM study on graphene flakes, the protocol was drafted based on the informative annex of ISO TS21356-1. The protocol was finalised, and participants were invited. The study used two samples I) graphene flakes deposited on a special grid with markers and a calibration grid for AFM and ii) graphene deposited onto silicon wafer for SEM analysis and supplied along with a commercial calibration sample. The samples and protocol were tested in an inter-consortium ILC (inter-laboratory comparison) with NPL, the University of Manchester and LNE before launching the main study. The samples and protocols were sent to 26 participants from Africa, Australia, America, Asia and Europe. From those 17 participants submitted results. The results showed the factors that most contribute to the uncertainty in this type of measurement. In particular, this study has shown that user bias in flake selection is a major source of uncertainty and variation between the different laboratories. Hence additional guidance on flake selection will be included in the revision of ISO TS 21356-1.

For XPS analysis, work was undertaken to understand the differences between the analysis of pellet samples and those of loose powder. A paper on this topic was published in 2023. In early 2022, the protocols and samples were tested in small test interlaboratory studies involving project partners. This was to check and improve the protocols. New test material was produced and supplied and differences between high energy (HAXPS) and normal (XPS) were investigated by BAM and NPL on the material. These important differences were published in a paper in 2023. BAM prepared the samples and protocols. Four graphene powders were used in the study were supplied along with 2 calibration samples. 27 participants from 14 different countries from Europe, Asia, and North and South America agreed to participate with 21 participants sending results. Powder sample preparation was found to be much more popular than using the pellet method.

There were three outliers from one laboratory. This was found to be due to humidity in the lab. For the results, 20 % relative uncertainty covered more than 50 % of the results. It was found that reliable quantification with XPS is possible for industrial graphene nanoplatelets. It was found that it is important to provide information about the sample preparation (pellet, powder in recess). The learning from this study aided the development of ISO TS23359.

For Raman spectroscopy, initially, multiple issues with the initial test material were found. After a few iterations, new material was produced by NPL (chemically exfoliated graphene) and by the University of Manchester (mechanically exfoliated few-layer graphene). Both of these were measured by INRIM using AFM and Raman spectroscopy. A mini-ILC involving INRIM, NPL and IMDEA took place which improved the protocol and resolved issues. The main ILC was launched and involved ten participants from Europe, USA, Brazil, China and Japan. The measurements based on Raman spectroscopy for the identification of a number of layers in graphene were found to be consistent in the ILC. Here, the identification of single-layer graphene had the highest consistency. When the results were not consistent, it was found that it was clearly a problem of location of the same exact area between participants. The results taken on graphene samples with more than one layer showed some variations in the ILC results It was found that the nature of layer stacking should be also considered while analysing the samples with Raman spectroscopy as twisted graphene shows signatures (2D peak width) similar to single-layer graphene. Hence optical microscopy and or other techniques should be used in conjunction as a complementary tool to determine the number of layers. The position of the 2D peak can also be used as an alternative criterion for determining the number of layers. There could be variations in results because of the different fitting methods used to fit the 2D peak. The ILC was completed successfully.

For GO analysis using SEM, commercial GO was sourced as the reference material. BAM investigated improving the GO deposition procedure in order to produce well-separated GO flakes of appropriate density for SEM measurements. A mini-ILC between BAM and NPL was undertaken. Here, 2 samples were analysed using Scanning Electron Microscopy (SEM) by both institutes analysing at least 200 flakes on each of the two samples. Detailed image and data analysis of the flakes' lateral dimension was undertaken through image processing software involving the comparison of two methods. It was found that the influence of the SEM operator as well as the possible local inhomogeneities of the GO flakes with different sizes were observed and different flakes chosen to analyse were found to be the main source of uncertainties. The image analysis methods produced similar results The full VAMAS international interlaboratory study was launched with samples and protocols sent to 10 international participants. These interlaboratory studies all help improve the ISO standards. The objective was fully achieved.

Impact

The consortium put a strong emphasis on impact from the outset and throughout the duration of the project. There was strong engagement with key stakeholders both in Europe and internationally via the interlaboratory studies and the accompanying standards development. This included international metrology institutes (for example from USA, Canada, Japan, China, Brazil and India) as well as large and SME industry companies and academia. The consortium partnered with the Graphene Flagship project and industry associations such as the Graphene Council and the nanotechnologies industry association as well as industry, standards bodies and other key players. This included joint workshops, conferences and YouTube videos. Strong engagement and input into the project were received by these at the stakeholder's advisory group meeting.

Sustained awareness and impact have been achieved throughout the project via 51 presentations at international, European and national conferences and workshops including many invited talks. Consortium members were extremely active in key standardisation committees including ISO TC229. Members have participated in 46 meetings, including general participation, presentation of the project, and leading the development of new standards.

A website was created to provide general information and to promote news stories from the project. The consortium had Twitter and LinkedIn accounts, with consortium members also posting updates of the project on Linkedin using their own accounts. The first publication highlighting the publication of the first graphene measurement standard (ISO TS21356-1 led by the consortium) was published in Nature Review Physics. A second publication on Raman spectroscopy of graphene has also been published. A third publication on the preparation of graphene material has been published. Two other papers on XPS of functionalised graphene were published. Four of the publications were joint publications between different consortium members.

Impact on industrial and other user communities

At the start of the project, there were over 100 commercial ‘graphene’ producers worldwide, including leading graphene producers in Europe, with an 'on-paper' offering of materials with vastly different properties and types. However, many suppliers (and buyers) were hindered due to uncharacterised material that can be more often graphite rather than graphene or have batch-to-batch variations. With this project and the resultant standards, the entire supply chain of graphene from manufacturers to application builders and final consumers will profit from clear material specifications and globally accepted characterisation standards. This will lead to suppliers seeing a stable demand for their high-value high-quality products.

Application builders will profit from reliable and traceable materials supply as well as comparability and traceability of various parameters to metrological standards. This will further enhance the development of advanced graphene applications and will ultimately profit ordinary consumers with greater choices of technology as well as reliable functionality due to well-specified materials used in the products. The impact is therefore on the entire supply chain and it does not depend on the graphene price or application hype in the market.

Once the material itself is characterised reproducibly and in a way that allows cross-comparison, real-world products can be tailored and improved using specific types of graphene and related 2D materials. These products include those in solar cell and battery production, composites and coatings, aerospace and automotive products (that need strong lightweight components), along with advanced clothing and consumer products. Transparent and consensus-driven metrology of graphene will enable companies that are producing and using graphene in these and other future applications to have a clear and sustainable business, reliable products and a safer environment.

Impact on the metrology and scientific communities

Based on the project's results, validated methods for characterising the structural and chemical properties of graphene using SEM, AFM, XPS and Raman spectroscopy were and will be made available to the metrology and scientific communities via peer-reviewed publications and European and international standards. This will create a large impact on measurement and test laboratories which will be able to measure the increasing number of materials being produced by companies worldwide to an accredited standard. In addition, academics will be able to better reproduce scientific results, assess the applicability of results to different technology areas under investigation and understand how the material properties affect the performance of lab-scale products for different application areas.

The uncertainties for measuring the properties of graphene such as flake size, chemical composition, thickness, number of layers, etc were up to 50 % currently and will reduce typically by at least a factor of two. The metrology and scientific communities had the opportunity to take part in international interlaboratory studies enabling them to benchmark themselves against peers, along with a chance to improve their abilities to measure graphene more accurately. On a broader scope, the project has strengthened the collaboration of European NMIs in the area of nanomaterials and will increase their competitiveness and consistency in graphene characterisation in a competitive international market.

Impact on relevant standards

Standards cannot be developed without proven and verified methodology. Normative documentary standards are based on methods or procedures developed through interlaboratory studies and verified internationally. Before the project commenced there were no graphene standards available beyond terminology and an overview technical report, neither of which contains such verified methodology. This project targeted the current gaps by leading four international interlaboratory studies and leading the development of three international standards in ISO TC229 and IEC TC113. This project will directly lead to the faster publication of these standards internationally and as European standards via CEN TC352. To this end, the first graphene measurement standard ISO TS 21356-1 on structural characterization of graphene was published in March 2021 led by the consortium with the other standards led by the consortium due to be published in the next year or two.

Longer-term economic, social and environmental impacts

Standards in general, but graphene standards in particular, aim to provide a level-playing field for the graphene industry where the main beneficiary is the customer – the innovative product developer and the final consumer. Without clear standards and accepted specifications, it is impossible to develop such a sustainable supply chain. Some previous nanomaterials have bypassed specification developments with companies arguing their unique properties and have subsequently found low acceptance and application of their "wonder material" by the global market. Without this project, these standards would have taken substantially longer to publish and would not have a larger amount of informative content, both of which would have hampered the industry and led to the possibility that companies would have developed different internal processes. This would have led to a substantial amount of time and investment wasted and require further effort to rectify. As most companies in this industry are SMEs, this could have led to otherwise very profitable companies struggling to survive financially. This would have been a substantial loss to the market overall and hamper advances in technology.

Many industries that use graphene including flexible electric/photonics, solar cells, and various medical, chemical and industrial processes will benefit from the well-characterised graphene that will be able to be obtained as a result of this project. The purchasers of graphene will also benefit from the confidence of being able to purchase graphene with known properties and less batch-to-batch variability. Without overcoming this metrological barrier, the potential of this disruptive material will not be truly realised and thus advancements in many important grand challenges, such as the aging population, the internet of things, light-weighting, and improvements in energy storage, will be slower.

List of Publications

1. Clifford, C.A., Martins Ferreira, E.H., Fujimoto, T. et al. The importance of international standards for the graphene community. Nat Rev Phys 3, 233–235 (2021). https://doi.org/10.1038/s42254-021-00278-6
2. Sacco A.,Portesi C.,Giovannozzi A.M., Rossi A.M. Graphene edge method for three-dimensional probing of Raman microscopes focal volumes. Journal of Raman Spectroscopy 52(10) 1671-1684 (2021) https://doi.org/10.1002/jrs.6187
3. Chemello G., Radnik J., Hodoroaba V-D. Analysis of Industrial Graphene-Based Flakes – First Results on Morphological Characterization, Sample Preparation and Chemical Composition. Microscopy and Microanalysis, 28(S1), 1006–1008 (2022), https://doi.org/10.1017/S1431927622004342
4. Reed B. P., Marchesini S., Chemello G, Morgan D.J., Vyas N., Howe T., Radnik J., Clifford C.A., Pollard A.J., The influence of sample preparation on XPS quantification of oxygen-functionalised graphene nanoplatelets, Carbon 211, 118054 (2023) https://doi.org/10.1016/j.carbon.2023.118054
5. Chemello G., Knigge X., Ciornii D, Reed B.P., Pollard A.J., Clifford C.A., Howe T., Vyas N., Hodoroaba V.-D., RadnikJ. Influence of the Morphology on the Functionalization of Graphene Nanoplatelets Analyzed by Comparative Photoelectron Spectroscopy with Soft and Hard X-Rays, Advanced materials interfaces, 10(20) 2300116 (2023) https://doi.org/10.1002/admi.202300116

This list is also available here: https://www.euramet.org/repository/research-publications-repository-link/

<table>
<tr><td colspan="2">Project start date and duration:</td><td>01 September 2020, 36 months</td></tr>
<tr><td colspan="3">Coordinator: Charles Clifford, NPL, Tel: +44 20 8943 6620 E-mail: charles.clifford@npl.co.uk
Project website address: http://empir.npl.co.uk/isogscope</td></tr>
<tr><td colspan="2">Chief Stakeholder Organisation: ISO TC229 / JWG2</td><td>Chief Stakeholder Contact: Naoyuki Taketoshi</td></tr>
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3. INRIM, Italy
4. LNE, France</td><td>External Funded Partners:
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7. Hay, United Kingdom
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