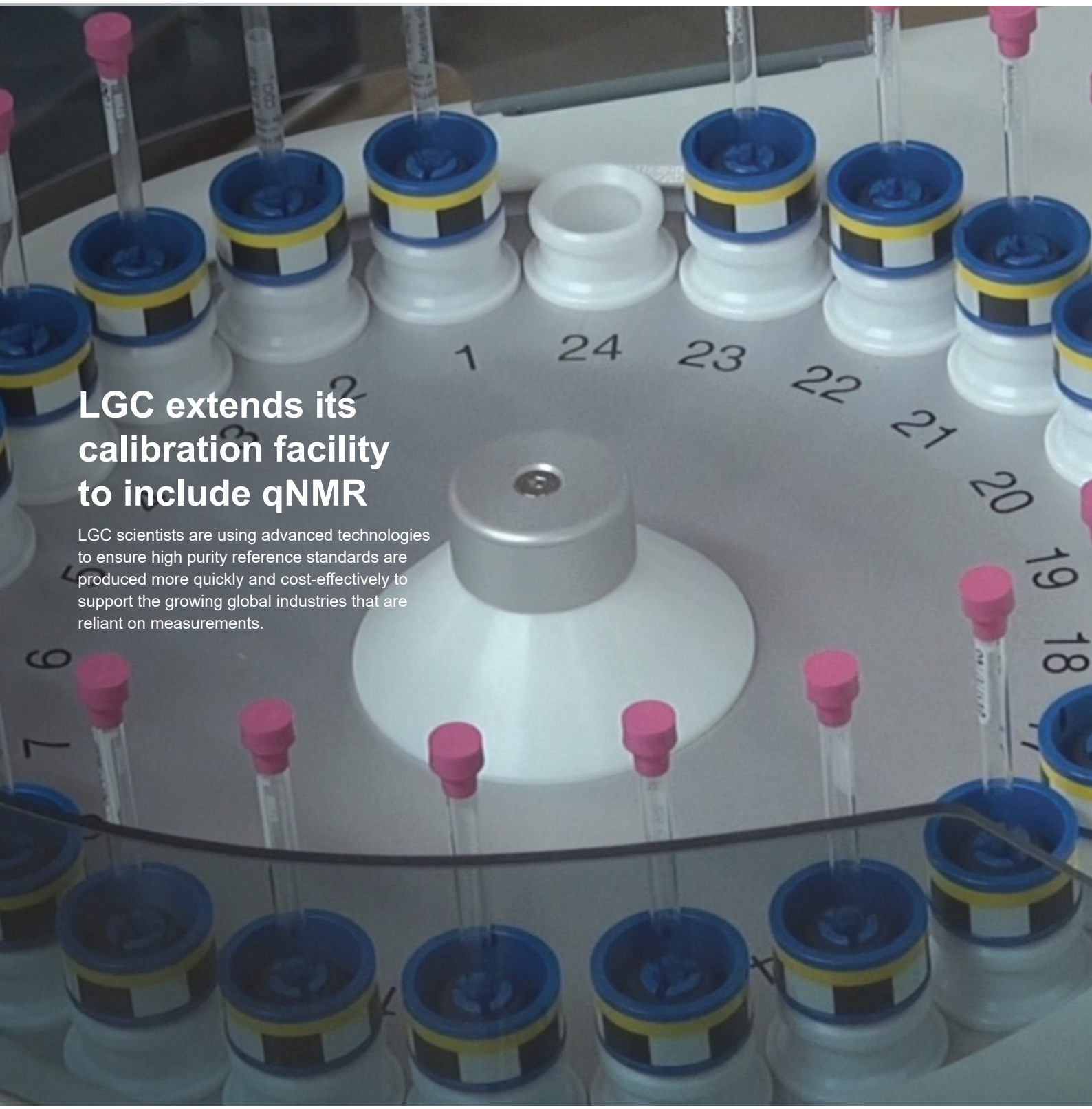

LGC extends its calibration facility to include qNMR

LGC scientists are using advanced technologies to ensure high purity reference standards are produced more quickly and cost-effectively to support the growing global industries that are reliant on measurements.

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The requirement

Thousands of chemical and biological measurements are made globally each day. These measurements support activities as varied as making a medical diagnosis, protecting the consumer against counterfeit products or safeguarding the quality of our water.

For governments, society and individuals to have confidence in the decisions based on these results they must have confidence in the measurements themselves: they must be accurate and reliable and – in our increasingly global society – this must be true regardless of where in the world they are made.

A series of reference materials are used to disseminate the required measurement confidence via calibration, from the highest (primary) level to those run on a day-to-day basis. The fundamental cornerstone of this reference material system is the provision of high purity reference standards as the primary calibrators. These are typically produced by National Measurement Institutes (NMIs) or Designated Institutes (DIs) and have assigned values with demonstrable traceability and low uncertainty.

Traditional methods for determining purity use an indirect approach, subtracting the measured known impurities from the maximum total of 100 %. This is time consuming, costly and, in some cases, even inaccurate, limiting the support NMI and DIs can provide to global industry.

As industry requirements become ever more varied and complex, an increasing number and range of high purity reference standards are required to support the challenging measurements of real-world samples. There is therefore a growing need for primary calibrators to have values assigned more quickly and at significantly lower cost.

The solution

To address this issue, scientists at LGC have been implementing quantitative nuclear magnetic resonance (qNMR) as an alternative to determine purity directly[a].

Using a number of small organic compounds where well-characterised reference materials exist to demonstrate proof of principle, such as amino acids, the use of qNMR for purity determination has been rigorously assessed and validated at LGC. The components of the measurement uncertainty have been evaluated and minimised, and the direct traceability of results to the International System of Units (SI) has been demonstrated.

The use of qNMR as a primary method has been standardised through comparison studies between NMIs. qNMR has been included in both LGC's ISO Guide 34 accreditation for the production of reference materials and ISO 17025 calibration accreditation for purity of organic materials, extending the scope of our calibration facility.

The potential of two dimensional qNMR, a technique traditionally used for qualitative work, has also been investigated for purity at LGC. Using small peptides as an example, 2D qNMR has been compared with the traditional approaches of high performance liquid chromatography (HPLC) and amino acid analysis (AAA). It dramatically reduced analysis times from a week to a single day, while also improving measurement uncertainty and preserving the sample for further analysis.

The impact

Purity determination by qNMR is faster than traditional methods and allows for direct analysis of the compound of interest rather than through subtraction of known and measurable impurities.

The 2D qNMR methods developed at LGC will allow for provision of ever more complex primary calibrators to new and emerging markets. This will affect an increasing number of fields, from the forensic analysis of adulterated powders to the production of pharmaceutical standards, all of which require pure reference standards to underpin daily measurement activities.

The efforts in this area will expand LGC's current capability to produce and value-assign high purity reference standards to meet the growing requirement for a greater diversity of pure materials. This will help support the >£300bn global industry reliant on chemical measurement.

Quantitative 1H NMR accreditation for purity

- *ISO 17025 - calibration accreditation for organic compounds of purity >90 % and molecular weight <1000 g/mol*
- *ISO Guide 34 - reference material production of pure organic chemicals*

[a] *Le Gresley A, Fardus F, Warren J. Crit Rev Anal Chem (2015) 45:300-10. doi:10.1080/10408347.2014.944971.*

For further information, contact:
LGC, Queens Road, Teddington, Middlesex TW11 0LY, UK

Tel: **+44 (0)20 8943 7393** Email: **nmshelp@lgcgroup.com** Web: **www.lgcgroup.com/nmi**

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