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Hydrogen Purity: Developing Low-Level Sulphur Speciation Measurement Capability

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Abstract : Fuel cell electric vehicles provide the potential to decarbonise road transport, create new economic opportunities, diversify national energy supply, and significantly reduce the environmental impacts of road transport. A potential issue, however, is that the catalyst used at the fuel cell cathode is susceptible to degradation by impurities, especially sulphur-containing compounds. A recent European Directive (2014/94/EU) stipulates that, from November 2017, all hydrogen provided to fuel cell vehicles in Europe must comply with the hydrogen purity specifications listed in ISO 14687-2; this includes reactive and toxic chemicals such as ammonia and total sulphur-containing compounds. This requirement poses great analytical challenges due to the instability of some of these compounds in calibration gas standards at relatively low amount fractions and the difficulty associated with undertaking measurements of groups of compounds rather than individual compounds. Without the available reference materials and analytical infrastructure, hydrogen refuelling stations will not be able to demonstrate compliance to the ISO 14687 specifications. The hydrogen purity laboratory at NPL provides world leading, accredited purity measurements to allow hydrogen refuelling stations to evidence compliance to ISO 14687. Utilising state-of-the-art methods that have been developed by NPL's hydrogen purity laboratory, including a novel method for measuring total sulphur compounds at 4 nmol/mol and a hydrogen impurity enrichment device, we provide the capabilities necessary to achieve these goals. An overview of these capabilities will be given in this paper. As part of the EMPIR Hydrogen co-normative project 'Metrology for sustainable hydrogen energy applications', NPL are developing a validated analytical methodology for the measurement of speciated sulphur-containing compounds in hydrogen at low amount fractions pmol/mol to nmol/mol) to allow identification and measurement of individual sulphur-containing impurities in real samples of hydrogen (opposed to a 'total sulphur' measurement). This is achieved by producing a suite of stable gravimetrically-prepared primary reference gas standards containing low amount fractions of sulphur-containing compounds (hydrogen sulphide, carbonyl sulphide, carbon disulphide, 2-methyl-2-propanethiol and tetrahydrothiophene have been selected for use in this study) to be used in conjunction with novel dynamic dilution facilities to enable generation of pmol/mol to nmol/mol level gas mixtures (a dynamic method is required as compounds at these levels would be unstable in gas cylinder mixtures). Method development and optimisation are performed using gas chromatographic techniques assisted by cryo-trapping technologies and coupled with sulphur chemiluminescence detection to allow improved qualitative and quantitative analyses of sulphur-containing impurities in hydrogen. The paper will review the state-of-the art gas standard preparation techniques, including the use and testing of dynamic dilution technologies for reactive chemical components in hydrogen. Method development will also be presented highlighting the advances in the measurement of speciated sulphur compounds in hydrogen at low amount fractions.

Keywords : gas chromatography, hydrogen purity, ISO 14687, sulphur chemiluminescence detector
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