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Accurate measurements of Primary Standard Gas Mixtures (PSMs) of CH4 in synthetic and scrubbed real air analyzed by two independent measuring techniques: CRDS and GC-FID

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In 2013 the BIPM organized the international comparison CCQM-K82 designed to evaluate the level of comparability between National Metrology Institutes (NMI) preparative capabilities for gravimetric methane in air primary reference mixtures in the range (1800-2200) nmol mol-1. This required the development of a measurement facility to compare standards, which was validated prior to the comparison with a suite of ten standards of methane in air prepared gravimetrically by NIST. The mixtures were intentionally prepared in two different air matrices, half in scrubbed real air and half in synthetic, to demonstrate that the use of synthetic air based standards did not introduced any bias for the measurement of atmospheric methane concentrations.
The BIPM facility is based on two analytical techniques used under repeatability conditions, namely, cavity ring-down spectroscopy (CRDS) and gas chromatography (GC-FID). GC-FID measurements were performed following a traditional protocol including ratios to a stable control cylinder, giving a typical relative uncertainty of 0.025%. CRDS measurements were performed with the same protocol, but also in a much shorter process that did not use any control cylinder, allowing the reduction of the relative uncertainty to 0.01%. Using the ten standards as references to construct a calibration line, all protocols resulted in a good linearity with very similar residuals. In particular, no effect of the air matrix was observed, as could be especially expected in CRDS due to different pressure broadening parameters, demonstrating the close matching between synthetic and scrubbed real air matrices.