

Addendum 1

Standard Hot Carrier Gas Extraction Method

This addendum adds a third standard method of analysis to AWS A4.3:1993 and should be used in the same context as the two other standard methods of analysis prescribed in Clauses 7 and 8.

The hot carrier gas extraction method is where a welded specimen is heated to a relatively high temperature [400°C (750°F)] to facilitate the extraction of diffusible hydrogen from the specimen. The evolved hydrogen gas is measured continuously using instrumentation that utilizes an inert carrier gas and thermal conductivity detectors (TCD).

It is not the objective of this document to specify each hot carrier gas extraction system that is available for the measurement of hydrogen in metals. However, it is important to note that all hot carrier gas extraction systems shall provide a proper correlation, in terms of accuracy and reproducibility. Particularly, the calibration procedures may be different for the individual measurement systems; therefore, the manufacturer's instructions shall be followed for the gas extraction equipment.

Prior to analysis, proper calibration procedures shall be performed that generate acceptable results per recommended instrument manufacturer procedure requirements or adequate linearity per internal standard operating procedures. Also, sufficient testing shall be conducted to determine the ramp time necessary to reach the desired specimen test temperature and that time at temperature is sufficient to release substantially all diffusible hydrogen. Substantially all is achieved when the detector has met the time at temperature requirements and when the rate of analyte change has minimized and returned to baseline.

Determination of the required ramp time can be best performed by measuring the temperature of an instrumented specimen that is of comparable thermal mass (i.e., geometry and thermal conductivity) to the test specimens during a heating cycle identical to that which will be used to carry out the testing.

The only acceptable specimen test temperature is 400°C ± 3°C (750°F ± 5°F). The minimum extraction time is 0.35 h (21 min) and begins once the specimen has reached test temperature. Test time is defined as a minimum, with no maximum limit given, to ensure substantially all diffusible hydrogen has been collected. Weld deposits containing a large amount of hydrogen may require extraction times much longer than the required minimum to evolve substantially all hydrogen.

Depending on the equipment design, the instrument may produce substantial thermal variations between different locations of the specimen (top, bottom, front, back, etc.). Characterization of the detected thermal gradients informs selection of an appropriate temperature set point for the apparatus.

The apparatus shall be assembled and operated following the instructions of the manufacturer. Other methods of calibration than what is described in the paragraph below are allowed per instrument instruction.

The calibration shall be carried out by supplying a known hydrogen (or other suitable calibration gas with applied gas factor by reason of difference in thermal conductivity) volume to the hot carrier gas extraction system. Depending on the analysis instrument, a sufficiently long gas flow time shall be assured for stabilization of the TCD prior to analysis. The detector shall be calibrated over the entire working area of interest by incorporating different gas dose volumes.

During normal instrument use, any existing calibration shall be verified by gas dosing before analysis is attempted. Checks shall be made at the start and completion of a series of analyses to ensure that no drift has occurred in the instrument calibration. The interval between drift checks shall not exceed 24 h.

The manufacturer's calibration procedure shall be utilized, and the defined acceptance limits shall be followed when prescribed. If calibration acceptance limits are not defined, the linearity R^2 values must be determined.

The linearity of response of the instrument may be judged by using linear regression analysis to fit the calibration data and then calculating the correlation, R^2 . Values of R^2 close to unity indicate a high degree of correlation. If the manufacturer has not defined acceptable calibration limits, the R^2 values must be determined and comply with established internal operating procedures. Calibration gas injection shall cover the applicable range to confirm linearity of response.