

Elementar rapidOXY cube (O) Methodology

XRF Laboratory

Solid State and Elemental Analysis Unit
UNSW Mark Wainwright Analytical Centre

Instrumentation

rapidOXY cube

Sample Analysis

Following the running of blanks and standards for daily corrections, approximately 1 to 50 mg of sample material is weighed into a silver foil, dependent on the program and type of sample. Additives may be used depending on the sample. The weight is recorded and the appropriate calibration program is selected. The silver foil is pressed and placed in the loading assembly for analysis. The sample undergoes pyrolysis, the resulting gases are analysed and the results checked by staff before release.

Theory of Operation:

After atmospheric gases have been flushed from the instrument system and the sample has dropped into the crucible, pyrolysis begins (pyrolysis occurs at 1400 °C). The gases pass through the pyrolysis tube and H₂, CO, CH₄ and various radicals are produced and halogens form volatile halogen compounds. Gases pass through an absorption tube, leaving only CO to pass through to be measured by a thermal conductivity detector (TCD) (EA, 2017: 48-51).

The TCD “consists of two chambers through which the analysis gas mixture (measuring cell) and the pure carriage gas (reference cell) simultaneously flow through during the analysis. The chambers form a measuring bridge. The thermal conductivity of the carrier gas reacts sensitively on the addition of small amounts of foreign gases such as the measuring components N₂, CO₂, H₂O and SO₂. If one of these measuring components exists in the analysis gas mixture, this leads to a disturbance of the measuring bridge that is registered in the form of an electrical signal. The TCD measuring signal is digitized, integrated and output by the instrument to the PC, where it is registered in the form of a measuring peak dependent on time, and the integral is correlated with the absolute quantity of the respective measuring component by calibration functions.” (EA, 2017: 48)

Reference

Elementar Analysensysteme GmbH (EA), 2017, *Operating instructions rapid OXY cube Elemental Analyzer*. Langenselbold, Germany: Elementar Analysensysteme GmbH.