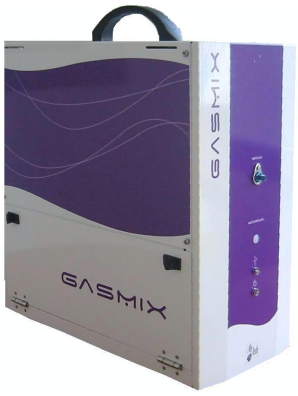
Application Note GasMix 06-03



MULTIPOINT CALIBRATION IS NOW OF HIGH INTEREST FOR GASES AS WELL

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Calibrating an analyzer at different concentration levels using only one gas standard is of high interest and now possible

The gas analyzer users are now under pressure to run their analyzers as described in the official methods. Round-Robin tests and validated environments impose more stringent procedures to lab managers to follow performances of their analyzers. Instruments need to be calibrated on a regular basis, following standards, such as ISO, EN, EPA etc.

Almost all analytical techniques request a periodical calibration of the analyzers and this calibration must be checked periodically. This can be done before, after, or even during a batch of analyses. Different ways of calibrating an analyzer are possible: External, internal, bracketed, normalized calibrations are among the most widely used. Standard added analysis is also used, mainly for trace analysis.

Four points are better than one!

In case a linear calibration curve has to be built, it is generally admitted that 4 points have to be established. Three points at least are needed in order to evaluate the linearity through the coefficient of linear regression. A fourth point is always better to confirm the performance of a detector in case one point is slightly out of the curve (see Figure 1). The fourth point will bring a confirmation (see Figure 2).

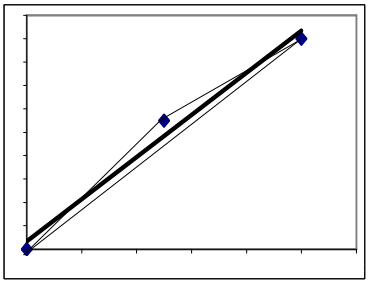


fig. 1, Three calibration points, which curve to choose?

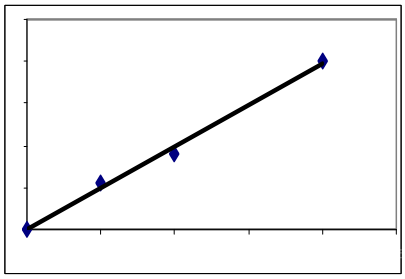
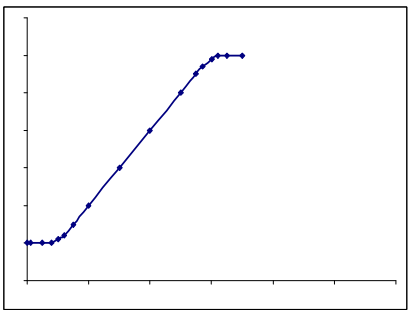


fig. 2, The fourth point confirms the trend

Linearity and dynamic property

Another important point to take into account is the S shape response shown by many detectors.



The concentration is reported on the x axis while the response of the detector is given on the y axis:

- for low concentrations, below the detection limit, the detector is ''blind'',
- then the response becomes linear; this happens for a concentration range which corresponds to the dynamic window of the detector,
- on the high range, detectors saturate: their response remains the same whatever the amount of analyte.

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Calibration curve and related errors

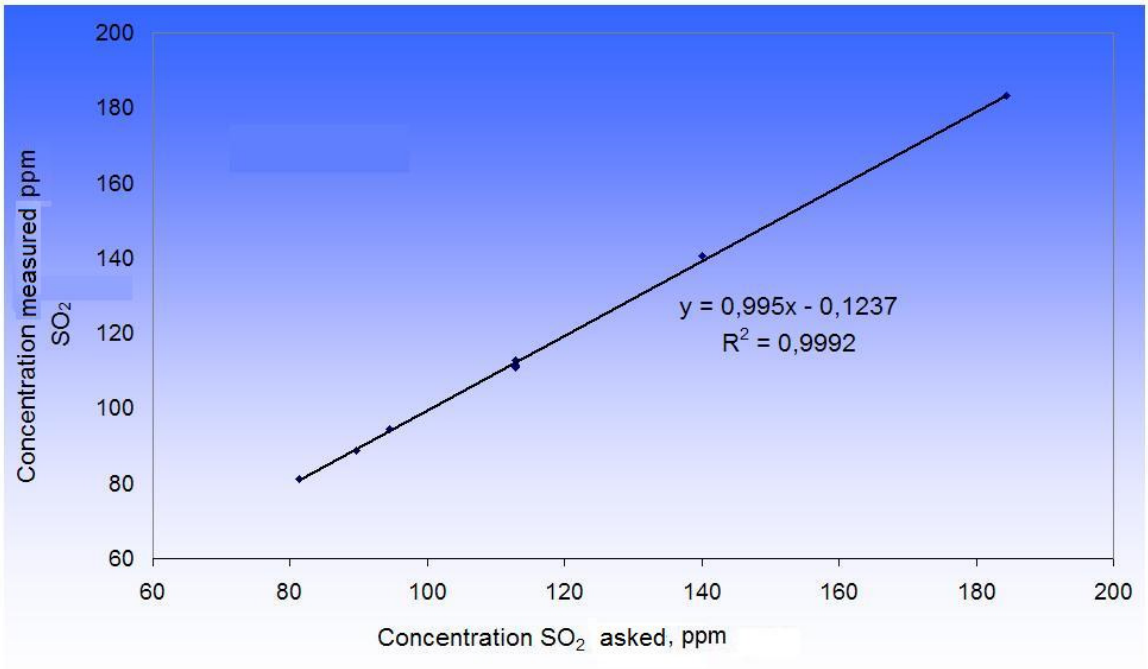
The error related to a concentration depends on the number of points used to plot the curve. This error decreases as number of measurement point increases.
If a calibration curve y = ax + b is plotted from one point only, the error on the slope may be high, and the y-intercept is fixed and considered to be equal to zero.

Limit of detection (LOD) and limit of quantification (LOQ)

To calculate the LOD and the LOQ, blank injections are performed. Their mean M and standard deviation σ are determined. These values M+3σ and M+10σ are then compared to the calibration curve to give the LOD and LOQ. Thus the importance of having a good and reliable calibration.

Experimental Conditions

GasMix™ allows us to deal with both questions by diluting standard gas mixtures in order to prepare on site different gas standards which will be used to build a calibration curve.
For example, starting from a standard with 505ppm of SO_2 in Nitrogen, a calibration curve is built between 80 and 180ppm SO_2. This calibration is made on an Antek 9000 Sulfur analyzer.



GasMix™ being completely automatic, this entire procedure was completely unattended. This allows a major human workload reduction for the laboratory manager. Preparing one gas injection or just a complete automatic sequence does not take any extra operator time.

Conclusion
A single point calibration may be the source of important errors on the metrology and validation of a gas analyzer: the slope and the y-intercept value may not be correct. The calculated LOD and LOQ values may therefore be inaccurate. All of this because laboratories have only one gas standard in stock.

GasMix™ makes the analyzer more reliable, allows a better use of the instrument within its entire dynamic range and all this without increasing the operator work load. GasMix™ is computer-controlled and ISO compliant.

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