

CHAPTER 8

Initial Calibration Verification (ICV)

8-1. Introduction.

The **initial calibration verification (ICV)** is evaluated to assess the accuracy of the initial calibration standards¹.

8-2. Criteria.

The ICV must be performed after the initial calibration via the analysis of a mid-level standard. The working calibration standards and the ICV standard must be from *independent* sources (e.g., from two different manufacturers). The recovery of the ICV should be within 90% to 110% for inorganic analyses and within 80% to 120% for organic analyses.

8-3. Evaluation.

a. Review the standard preparation logs to verify that the ICV and initial calibration standards were prepared from independent NIST-traceable standards. Review the instrument printouts and run log sheets to verify that the ICV was analyzed after the initial calibration within its expiration date. Using the standard preparation log sheets and the ICV summary form, recalculate an ICV recovery and compare the calculated value with the reported value. If an ICV standard was not prepared, review the standard preparation log to determine whether or not any CCVs or LCSs were prepared from an independent-source standard

b. It should be noted that an ICV failure does not definitively demonstrate a source problem for the initial calibration standards. For example, failures may occur because of problems with the initial calibration curve (e.g., a poor fit) and analytical blunder. Prior to qualifying the data, it may be desirable to investigate the source of the failure (e.g., by requesting additional information from the laboratory).

¹One could argue that an acceptable ICV does not definitively demonstrate the accuracy of the standards used for the initial calibration. For example, the spiking concentrations for both the initial calibration and ICV standards could be biased low (relative to the actual analyte concentrations in the standards). However, since both standards should be traceable to a reliable source (e.g., NIST), an acceptable ICV supports the conclusion that the standards are accurate. It is more likely than not that two different traceable standards that are in agreement are accurate.

8-4. Qualification.

8-4.1. Frequency.

a. When an ICV is not performed (i.e., when initial calibration standards are not verified with an independent-source standard) at a minimum, qualify all detections with the J flag and all nondetections with the UN flag. Alternatively, the data review report must state that all the results are potentially estimated. Rejection of the data may be appropriate when the data is being used to support critical decisions.

b. The CCVs or the LCS may have been prepared from an independent-source standard. If CCVs or LCSs are prepared from independent-source stock standards and the recoveries are acceptable, then the data must not be qualified. If an independent-source standard for the ICV is not commercially available, a standard from the same source material but a different preparation lot (e.g., different manufacturer's lot number) may be used for the ICV standard.

8-4.2. Percent Recovery.

8-4.2.1. Inorganics.

If the ICV recovery is unacceptable but falls within 80% to 120%, qualify detections with the J flag and nondetections with the UN flag. If the ICV recovery does not fall within 80% to 120%, then qualify the results with the X flag.

8-4.2.2. Organics.

If the ICV recovery is unacceptable but falls within 70% to 130%, qualify detections with the J flag and nondetections with the UN flag. If the ICV recovery does not fall within 70% to 130%, then qualify the results with the X flag.

8-4.3. Qualification for Bias.

When the ICV recovery is unacceptable or an independent-source standard is not used to verify the initial calibration standard (e.g., an ICV is not performed), *the direction of bias is unknown for the entire analytical process.* The recoveries of other QC samples (e.g., laboratory control samples and matrix spikes) must not be used to make inferences about the direction of bias (e.g., unless the uncertainty is much greater than that arising from the ICV noncompliance.)

Table 8-1
Data Qualification for ICV Results ¹

ICV %R	Method	Remarks	Sample (y)	Flag	
$90\% \leq \%R \leq 110\%$	Inorganics	Acceptable %R	$MRL < MQL < y$	None	
			$MRL < y < MQL$	J	
$80\% \leq \%R \leq 120\%$	Organics		$y < MRL$	U	
			$y > MRL$	J	
$110\% \leq \%R \leq 120\%$, $80\% \leq \%R \leq 90\%$	Inorganics	Marginal Failure	$y > MRL$	J	
			$y < MRL$	UN	
$120\% \leq \%R \leq 130\%$, $70\% \leq \%R \leq 80\%$	Organics		Gross Failure	$y < MRL$	UN
				$y > MRL$	X
$\%R > 120\%$, $\%R < 80\%$	Inorganics	Gross Failure		$y > MRL$	X
				$y < MRL$	X
$\%R > 130\%$, $\%R < 70\%$	Organics		Gross Failure	$y < MRL$	X
				$y > MRL$	X

Notes: 1. %R, MRL, MQL, and y denote the percent recovery of the target analyte in the ICV, method reporting limit, method quantitation limit, and concentration of the target analyte in an associated field sample, respectively. (The MRL is assumed to be greater than the MDL but less than the MQL.)