

The background of the slide is a blue gradient. In the upper half, there are faint, light-colored chemical structures and a globe, suggesting a scientific or laboratory setting. The main title is centered in the middle of the slide.

# Best Practices for Calibration and Calibration Verification

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# Volatiles GCMS

8260C / D

624.1

CLP

524.3

TNI



# Minimum number of Points

Method	Criterion
8260D	5, 6 for quadratic
624.1	5, 6 for quadratic, 3 allowed for “some analytes”
524.4	7
CLP	5
TNI	4, average 5, linear, 6 for quadratic

**Best Practice** | 5, 6 for quadratic

# Internal Standard

Method	Criterion
8260D	Implied
624.1	Required
524.4	Required
CLP	Required
TNI	Optional

**Best Practice** | **Required**

# Calibration types

Method	Criterion
8260D	Average, Linear, Quadratic, Cubic
624.1	Average, Linear, Quadratic
524.4	Linear or Quadratic
CLP	Average
TNI	Average, Linear or Quadratic (Implied)

**Best Practice** | **Average, Linear or Quadratic**

# Calibration criteria

## Average

Method	Criterion
8260D	Average: RSD $\leq$ 20%
624.1	Average: RSD < 35%
524.4	N/A
CLP	RSD < 20%, 30% or 40%, depending on analyte
TNI	Average: N/A

**Best Practice** | **20-30% range**

# Calibration criteria

## Regression

Method	Criterion
8260D	Linear, Quadratic: $R^2 \geq 0.990$ or $RSE \leq 20\%$
624.1	Linear, Quadratic: $R^2 \geq 0.920$ or $RSE \leq 35\%$
524.4	Linear, Quadratic: Readback <30% or 50% at MRL
CLP	N/A
TNI	Linear, Quadratic: N/A

**Best Practice** | **RSE < 20-30% or readback 30-50%**

“Very common mistakes in the analytical calibration process are the use of correlation and or determination coefficients...

*Evaluation of analytical calibration based on least squares linear regression for instrumental Techniques, Francisco Raposo, TrAC 77, Match 2016, Pages 167-185*



# Calibration criteria

## Fudge factors

Method	Criterion
8260C	10% may fail but must be reported as estimated
8260D	Same as 8260C but additional guidance
624.1	None
524.4	None
CLP	@ analytes may fail must be < 40% RSD
TNI	N/A

<b>Best Practice</b>	<b>10% may fail, combined with sensitivity demonstration for non-detects</b>
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# Weighting

Method	Criterion
8260D	Optional
624.1	Required (1/X)
524.4	Optional
CLP	Average only (i.e., required)
TNI	Optional

**Best Practice** | **1/X or 1/X<sup>2</sup> required**

# Recalc

Method	Criterion
8260D	Should, if using linear regression
624.1	N/A
524.4	Required
CLP	N/A
TNI	Required unless RSE

**Best Practice** | **Required unless RSE**

# Minimum Response Factor

Method	Criterion
8260D	Table 4 but with flexibility
624.1	N/A
524.3	N/A
CLP	Table 4
TNI	N/A

**Best Practice**

**Not needed**

# Initial Calibration Verification

Method	Criterion
8260C	Required, second source
624.1	N/A (but see FAQ)
524.3	N/A
CLP	Required, second source
TNI	Required

**Best Practice** | **Required, second source**

# Continuing Calibration Verification Criteria

Method	Criterion
8260D	80-120%, 20% of analytes allowed to fail, report as estimated unless sensitivity demonstration for NDs
624.1	Table 7 (mostly 65-135%, a few outliers, eg VC 5-195%). Retest allowed
524.3	70-130% if above MRL, 50-150% at MRL
CLP	20,25,30 or 35% depending on analyte
TNI	N/A

<b>Best Practice</b>	<b>70-130%, 10% allowed to fail, not more than 50-150%, report as estimated unless verification of sensitivity for NDs</b>
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# Continuing Calibration RT criteria

Method	Criterion
8260D	IS Within 30 sec of initial calibration
624.1	N/A
524.3	N/A
CLP	N/A
TNI	N/A

**Best Practice** | **Within 30 sec of initial calibration**

# Summary

Method	Criterion
Points	5, 6 for quadratic
IS	Within 30 sec of initial calibration
Cal types	Average, Linear, Quadratic
Acceptance criteria	20-30% RSD or RSE, or 30% each point, 50% at Quant limit
Fudge factors	10% fail, but within a limit, (50%) report as estimated unless sensitivity verification
Weighting	Required, 1/X or 1/X <sup>2</sup>
Recalc	Required (unless RSD or RSE)
Minimum RF	Not needed
ICV	Required, second source, 30%
CCV criteria	70-130%, 10% allowed to fail, not more than 50-150%, report as estimated unless verification of sensitivity for NDs



