

The Dropping of Mid-level Calibration Points



- *An outlier calibration point* (other than a high point or a low point) may be eliminated only after an investigation has been performed and the reasons for the problem have been documented (e.g., statistical test, or review of standard preparation logs). At no time may calibration point/s be eliminated solely to meet or improve performance relative to calibration curve acceptance criteria.



Corrective action may include elimination of the outlier, re-analysis of the outlier in duplicate (consecutive) with results of both re-analysis resulting in acceptable final curves, or re-analysis of the entire calibration curve. For example, poor calibration standard injection or purge would be documented in the run log and a re-analysis of the specific problematic runs could be simply repeated in the analytical batch sequence.



If instrument maintenance is required or the calibration standards are re-prepared, then re-analysis of the entire curve must be performed. At no time shall the level used for the Calibration Verification Standard (CLC) be eliminated from the original calibration curve.

Ref: USEPA Region III Laboratory Quality Manual, Version 3, Section 10.2.2.



It is prohibited to remove data points from within a calibration range while still retaining the extreme ends of the calibration range.

Ref: SW-846, Method 8000C, Section 11.5.5.2



NOTE: Reanalyzing or replacing a single standard must NOT be confused with the practice of discarding individual calibration results for specific target compounds in order to pick and choose a set of results that will meet the RSD or correlation criteria for the linear model.



The practice of discarding individual calibration results is addressed as a fourth alternative option and is very specific as to how a set of results are chosen to be discarded. If a standard is reanalyzed or a new standard is analyzed, then ALL of the results from the original analysis of the standard in question must be discarded.



Further, the practice of running additional standards at other concentrations and then picking only those results that meet the calibration acceptance criteria is **EXPRESSLY PROHIBITED**, since the analyst has generated data that demonstrate that the linear model does not apply to all of the data.

Ref: SW-846, Method 8000C, Section 11.5.5.2



In our opinion, the only valid technical reason to discard a standard in the middle of the curve is when there has been an obvious problem such as a bent injection needle on an autoinjector that yields very low responses for all the compound because the injection was not completed, or a single standard that has gone so bad that the difference is obvious to the naked eye. In either of these cases, the appropriate response would be to reinject a standard at that concentration and use it along with the other results to develop the initial calibration.

Ref: EHSO MICE, Email, April 2000



Discarding whole standards in the middle of the curve is simply an admission that something is wrong with the curve. The analyst has the evidence that the response does not fit the model, yet he ignores it to the point of throwing out the data. From our understanding of the perspective of your agency, this could amount to falsification.

Ref: EHS&G MICE, Email, April 2000



We have heard of analyst discarding results for individual compounds in this manner, throwing out one or two from this standard, several from another standard, etc. We see no justification for that approach at all. Either the whole standard goes, or it all stays.

Ref: EHSO MICE, Email, April 2000



*RESPONSE FACTOR REPORT GC MS #2

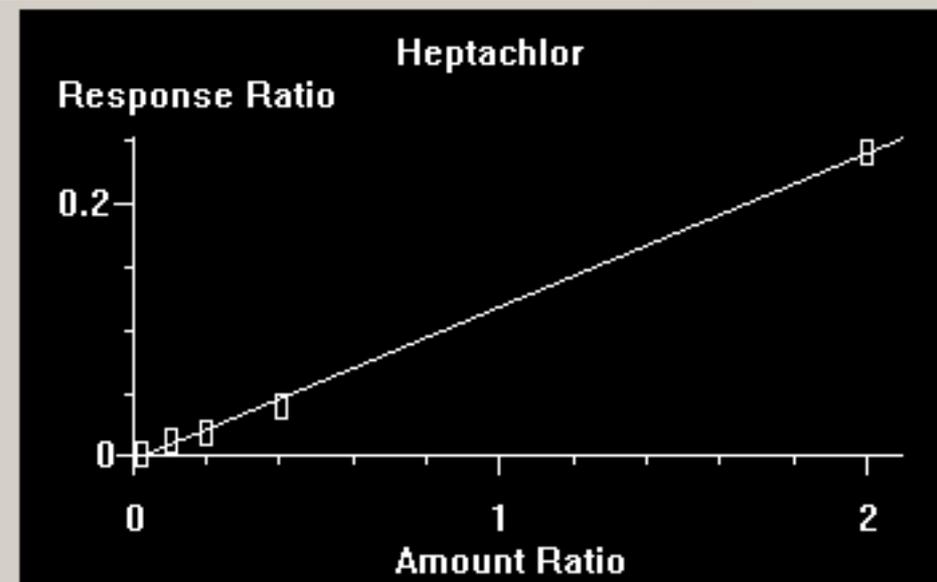
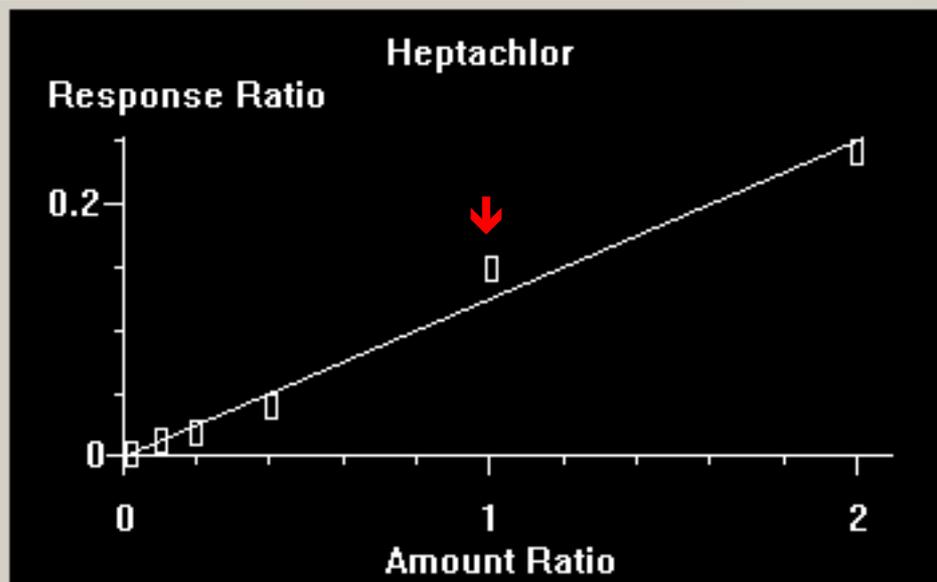
Method Path : C:\MSDchem\1\METHODS\
 Method File : 032702.M
 Title : method 525
 Last Update : Wed Mar 27 14:03:40 2002
 Response Via : *INITIAL CALIBRATION

* CALIBRATION FILES

1 =CAL1.D 2 =CAL2.D 3 =CAL3.D 4 =CAL4.D 5 =CAL5.D 6 =CAL6.D

* COMPOUND 1 2 3 4 5 6 * AUG %RSD

		1	2	3	4	5	6	* AUG	%RSD
1) I	p-Terphenyl-d14	-----ISTD-----							
2)	Acenaphthene-d10	-----ISTD-----							
3)	Hexachlorocycl...	0.331		0.267	0.248	0.242	0.226	0.263	15.56
4)	Propachlor	0.623	0.559	0.510	0.462	0.454	0.528	0.523	12.12
5)	Hexachlorobenzene	0.510	0.493	0.501	0.507	0.494	0.546	0.509	3.87
6) I	Chrysene-d10	-----ISTD-----							
7)	Simazine	0.285	0.277		0.140	0.153	0.113	0.194	42.01
8)	Atrazine	0.418	0.446	0.299	0.292	0.359	0.282	0.349	20.01
9)	Pentachlorophenol	0.202	0.164	0.078	0.040	0.037	0.028	0.092	80.85
10)	Lindane	0.250	0.272	0.208	0.211	0.292	0.256	0.248	13.51
11)	Metribuzin	0.261	0.278	0.176	0.128	0.141	0.101	0.181	40.35
12)	Alachlor	0.209		0.177	0.172	0.209	0.166	0.187	11.18
13)	Heptachlor	0.120		0.097	0.097	0.127	0.107	0.110	12.31
14)	Metolochlor	0.618	0.680	0.504	0.468	0.549	0.489	0.552	14.97
15)	Aldrin	0.122	0.146	0.119	0.125	0.171	0.142	0.137	14.45
16)	Heptachlor Epo...	0.087		0.084	0.089	0.116	0.114	0.098	15.76
17)	Butachlor	0.273	0.286	0.207	0.190	0.200	0.161	0.219	22.48
18)	Nonachlor	0.140		0.128	0.136	0.180	0.153	0.148	13.87
19)	4,4-DDE	0.234	0.257	0.221	0.222	0.285	0.315	0.256	14.84
20)	Dieldrin	0.140	0.150	0.143	0.148	0.190	0.202	0.162	16.49
21)	Endrin	0.042		0.034	0.032	0.039	0.037	0.037	10.53



Amount Ratio	Response Ratio
2.00000000	0.24074557
1.00000000	0.14861137
0.40000000	0.03886507
0.20000000	0.01937307
0.10000000	0.01265755
0.02000000	0.00213925

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Resp Ratio = 1.25e-001 * Amt - 6.80e-004
 Coef of Det (r²) = 0.983 Curve Fit: Linear

Resp Ratio = 1.21e-001 * Amt - 3.29e-003
 Coef of Det (r²) = 0.998 Curve Fit: Linear



Mid-point Calibration Dropping