

Method Detection Limit (MDL / LOD) Checklist * REV. 11/17/23

Based on NR 149 (2021), 40 CFR 136 Appendix B (2016)

Some questions may not be applicable to every lab. If applicable, all answers must be "yes" to be in compliance.

***This checklist was created for the aid of laboratories. It is only an internal audit guideline; it is not meant to be comprehensive of all regulatory requirements, to dictate DNR audit format, or to include all acceptable options. Laboratories must comply with all applicable code and method requirements whether listed on this checklist or not. Additional method and code requirements are on separate checklists. The department considers the MDL to be equivalent to the LOD.**

| | General | Y | N | Notes | Reference |
|---|--|---|---|-------|---|
| 1 | Do you perform an MDL for every analyte (excluding BOD, cBOD, titrimetric or gravimetric tests (HEM requires an MDL) and those tests where running a fortified sample is impossible or impractical), every method, every matrix, and every instrument? | | | | NR 149.48 (2)(a), (2)(b); 40 CFR Part 136 Appendix B (I)(1) |
| 2 | Do you determine the MDL using the 40 CFR 136 Appendix B method? | | | | NR 149.48 (2)(b) |
| 3 | Do all of the spiked blanks and method blanks go through all steps of the method? | | | | NR 149.48 (2)(b); 40 CFR Part 136 Appendix B (I)(1) |
| 4 | Were data removed only for gross failures (i.e., didn't remove statistical outliers)? | | | | 40 CFR Part 136 Appendix B (II)(2)(b), (II)(4)(b) |
| 5 | If the MDL spiked blanks or method blanks were in a batch with failed QC, do you exclude those results in the MDL determination? | | | | 40 CFR Part 136 Appendix B (II)(4)(d) |
| 6 | Is the MDL recalculated at least every 13 months? | | | | NR 149.48 (2)(b); 40 CFR Part 136 Appendix B (II)(4)(a) |
| 7 | Are all reported MDLs adjusted when the sample volumes or amounts used are different than those used for the MDL determination (i.e., for any pre- or post-dilutions with different liquid volumes and for any with different solids amount)? | | | | NR 149.48 (2)(d) |
| 8 | Are all and only those processing steps to which samples are subjected performed when determining the MDL? <i>(concentrating a sample to lower the MDL is not allowed unless the MDL determination was done the same way)</i> | | | | NR 149.48 (2)(b) |

| | Initial MDL | Y | N | Notes | Reference |
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| 9 | Is the initial MDL only performed for new methods, rarely used methods, if there was a significant change, or if the lab believes the sensitivity has changed significantly? | | | | NR 149.48 (2)(f); 40 CFR Part 136 Appendix B (II)(2), (II)(3)(d), (II)(4)(b) |
| 10 | Were at least 7 spiked blanks used? | | | | 40 CFR Part 136, Appendix B (II)(2)(b) |
| 11 | If a preparation step (e.g., distillation, digestion, extraction) is performed for the test, were the spiked blanks prepared in at least 3 batches on at least 3 different dates? | | | | 40 CFR Part 136 Appendix B (II)(2)(b) |
| 12 | Were the spiked blanks analyzed in at least 3 batches on 3 different dates? | | | | 40 CFR Part 136 Appendix B (II)(2)(b) |
| 13 | Were at least 7 method blanks used? | | | | 40 CFR Part 136 Appendix B (II)(2)(b) |

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| 14 | If pooling instruments for a combined MDL, were at least 3 separate preparations performed? (<i>the same prepared extract may be run on multiple instruments</i>) | | | | 40 CFR Part 136 Appendix B (II)(2)(b)(iii) |
| 15 | If pooling instruments for a combined MDL, were at least 2 spiked blanks and 2 method blanks prepared and analyzed on different calendar dates for each instrument being pooled? | | | | 40 CFR Part 136 Appendix B (II)(2)(b)(ii) |
| 16 | Are all spiked blanks greater than zero (and not ND)? | | | | 40 CFR Part 136 Appendix B (II)(2)(c) |
| 17 | Is the MDL _s calculated correctly: Student's <i>t</i> -value x sample standard deviation? | | | | 40 CFR Part 136 Appendix B (II)(2)(d)(ii) |
| 18 | If method blanks met chromatographic identification criteria, was the value included even if it was less than the current MDL? | | | | 40 CFR Part 136 Appendix B (II)(2)(d)(iii)(A) |
| 19 | If some method blanks were non-detects (i.e., did not meet the chromatographic identification criteria) but some were detects, is the MDL _b set to the highest method blank result or ≥99th percentile (if there are ≥100 method blank results)? | | | | 40 CFR Part 136 Appendix B (II)(2)(d)(iii)(B) |
| 20 | If all method blanks were numerical, is the MDL _b set to ≥99th percentile (if there are ≥100 method blank results) or is it calculated correctly: average + (Student's <i>t</i> -value x sample standard deviation)? | | | | 40 CFR Part 136 Appendix B (II)(2)(d)(iii)(C) |
| 21 | If the 99th percentile is used to determine the MDL _b , were 100 or more method blank results included? | | | | 40 CFR Part 136 Appendix B (II)(2)(d)(iii)(B), (II)(2)(d)(iii)(C) |
| 22 | Are negative results included in the MDL _b determination? | | | | 40 CFR Part 136 Appendix B (II)(2)(d)(iii)(A) |
| 23 | If the average of the method blanks is negative, is zero used in place of the average? | | | | 40 CFR Part 136 Appendix B (II)(2)(d)(iii)(C) |
| 24 | Is the correct Student's <i>t</i> -value, which is based on the number of replicates (n-1), used for both the MDL _s and for the MDL _b ? | | | | 40 CFR Part 136 Appendix B (II)(2)(d)(ii), (II)(2)(d)(iii)(C) |
| 25 | Were the spiked blanks and method blanks the most recent available for the initial MDL? | | | | 40 CFR Part 136 Appendix B (II)(2)(b) |
| 26 | Is the MDL set to the greater of the MDL _s or MDL _b ? | | | | 40 CFR Part 136 Appendix B (II)(2)(e) |
| 27 | Is the MDL below permit or regulatory limits? | | | | NR 149.48 (2)(c) |

| | Ongoing MDL | Y | N | Notes | Reference |
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| 28 | Is the MDL recalculated at least every 13 months? | | | | NR 149.48 (2)(b); 40 CFR Part 136 Appendix B (II)(4)(a) |
| 29 | If a preparation step (e.g., distillation, digestion, extraction) is performed for the test, were at least 2 spiked blanks prepared in separate batches each quarter (unless no field samples were analyzed in that quarter)? | | | | 40 CFR Part 136 Appendix B (II)(3)(a) |
| 30 | Were at least 2 spiked blanks analyzed in separate batches each quarter (unless no field samples were analyzed in that quarter)? | | | | 40 CFR Part 136 Appendix B (II)(3)(a) |
| 31 | If pooling MDLs, were at least 2 spiked blanks analyzed on each instrument in separate batches each quarter (unless no field samples were analyzed in that quarter)? | | | | 40 CFR Part 136 Appendix B (II)(3)(a) |

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| 32 | Was the same spike concentration used for all spiked blanks? | | | | 40 CFR Part 136 Appendix B (II)(3)(a) |
| 33 | For the annual MDL determination, were at least 7 spiked blanks used from the last 2 years? | | | | 40 CFR Part 136 Appendix B (II)(3)(b) |
| 34 | If more than 5% of the spiked blanks were negative or non-detects, was the initial MDL performed at a higher concentration? | | | | 40 CFR Part 136 Appendix B (II)(3)(c)(i) |
| 35 | Were all spiked blanks from the last 2 years (no more, no less) used in the MDL _s determination, including the initial MDL data if within 2 years of the recalculation date? | | | | 40 CFR Part 136 Appendix B (II)(4)(b), (II)(4)(c) |
| 36 | Is the MDL _s calculated correctly: Student's <i>t</i> -value x sample standard deviation? | | | | 40 CFR Part 136 Appendix B (II)(2)(d)(ii), (II)(4)(a) |
| 37 | Were all method blanks from the last 2 years used in the MDL _b determination (or were the last six months or last 50 method blanks used, whichever of these two options yielded more data)? | | | | 40 CFR Part 136 Appendix B (II)(4)(e) |
| 38 | If method blanks met chromatographic identification criteria, was the value included even if it was less than the current MDL? | | | | 40 CFR Part 136 Appendix B (II)(2)(d)(iii)(A), (II)(4)(a) |
| 39 | If some method blanks were non-detects (i.e., did not meet the chromatographic identification criteria) but some were detects, is the MDL _b set to the highest method blank result or ≥99th percentile (if there are ≥100 method blank results)? | | | | 40 CFR Part 136 Appendix B (II)(2)(d)(iii)(B), (II)(4)(a) |
| 40 | If all method blanks were numerical, is the MDL _b set to ≥99th percentile or is it calculated correctly: average + (Student's <i>t</i> -value x sample standard deviation)? | | | | 40 CFR Part 136 Appendix B (II)(2)(d)(iii)(C), (II)(4)(a) |
| 41 | If the 99th percentile is used to determine the MDL _b , were 100 or more method blank results included? | | | | 40 CFR Part 136 Appendix B (II)(2)(d)(iii)(B), (II)(2)(d)(iii)(C) |
| 42 | Are negative results included in the MDL _b determination? | | | | 40 CFR Part 136 Appendix B (II)(2)(d)(iii)(A), (II)(4)(a) |
| 43 | If the average of the method blanks is negative, is zero used in place of the average? | | | | 40 CFR Part 136 Appendix B (II)(2)(d)(iii)(C), (II)(4)(a) |
| 44 | Is the correct Student's <i>t</i> -value, which is based on the number of replicates (n-1), used for both the MDL _s and for the MDL _b ? | | | | 40 CFR Part 136 Appendix B (II)(2)(d)(ii), (II)(2)(d)(iii)(C), (II)(4)(a) |
| 45 | Is the MDL set to the greater of the MDL _s or MDL _b ? | | | | 40 CFR Part 136 Appendix B (II)(4)(f) |
| 46 | If the new, recalculated MDL is not within 0.5 - 2 times the existing MDL or ≥ 3% of the method blanks were above the existing MDL, was the MDL set to the newly determined MDL? | | | | 40 CFR Part 136 Appendix B (II)(4)(f) |
| 47 | Is the MDL below permit or regulatory limits? | | | | NR 149.48 (2)(c) |

| | Adding Instrument to MDL Pool | Y | N | Notes | Reference |
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| 48 | Were at least 2 spiked blanks and 2 method blanks run on the new instrument? | | | | 40 CFR Part 136 Appendix B (II)(3)(e) |

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| 49 | Were the 2 spiked blanks added to the existing spiked blank results and the MDL _s recalculated? | | | | 40 CFR Part 136 Appendix B (II)(3)(e) |
| 50 | If the recalculated MDL _s was not within a factor of 2 from the existing MDL, was a new initial MDL determined? | | | | 40 CFR Part 136 Appendix B (II)(3)(e) |
| 51 | If both method blanks were not below the existing MDL, was a new initial MDL determined? | | | | 40 CFR Part 136 Appendix B (II)(3)(e) |

| Documentation and Records - Are all of the following documented or recorded, if applicable? | | Y | N | Notes | Reference |
|--|---|----------|----------|--------------|----------------------------------|
| 52 | Analytical method used | | | | 40 CFR Part 136 Appendix B (III) |
| 53 | Units | | | | 40 CFR Part 136 Appendix B (III) |
| 54 | Matrix | | | | 40 CFR Part 136 Appendix B (III) |
| 55 | Rationale for removal of outliers | | | | 40 CFR Part 136 Appendix B (III) |
| 56 | If pooling MDLs, instrument identification | | | | NR 149.45 |
| 57 | Dates of prep (distillation, digestion, extraction) for each result | | | | NR 149.45 |
| 58 | Dates of analysis for each result | | | | NR 149.45 |
| 59 | MDL calculation date | | | | NR 149.45 |

| Other Observations | |
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| WI DNR MDL/LOD Resources |
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| DNR Website (which includes the resources below): Laboratory Certification Wisconsin DNR |
| DNR MDL spreadsheet - basic version |
| DNR MDL spreadsheet - advanced version (includes information for chromatography) |
| DNR MDL spreadsheet - advanced multi-parameter version |
| DNR MDL spreadsheet instructions |
| Lab Accreditation Program staff - contact any staff with questions or concerns, especially if there are ongoing QC issues |