

CIL Analytical Standards

for US EPA Method 1668 Revision A

In 1995, CIL produced the standards used in the first generation of US EPA Method 1668, High Resolution GC/High Resolution MS using Isotope Dilution. Based on the results of the first study, the method and composition of standards was revised and the first Draft Method 1668 was published in October 1995. CIL produced revised standards that have been used since then for analysis and method validation.

In December 1999, Revision A to Method 1668 was cited in 64 FR 72045 as the "state of the art test method for the measurement of PCB congeners", as part of a proposed rule for 40 CFR part 503. A number of internal standards were added and the novel use of ¹³C-PCBs as window definers was also incorporated into the revised method.

The following set of standards is now available for Method 1668A. All standards have been prepared as defined by the method. CIL's highly accurate PCB Certified Standards were used to verify formulation of the ¹³C₁₂-PCBs used in the Method 1668A standards, and were also used to formulate the calibration solutions and native stock solution.

EC-4976

PCB Calibration Solutions

| Native Toxics/LOC | IUPAC | CS0.2 (Hi sens) | CS1 | Concentration (ng/ml) | | | |
|----------------------------|-------|--------------------|-----|-----------------------|--------------|-----|------|
| | | | | CS2 | CS3 (VER) | CS4 | CS5 |
| 2-MoCB | 1 | 0.2 | 1.0 | 5.0 | 50 | 400 | 2000 |
| 4-MoCB | 3 | 0.2 | 1.0 | 5.0 | 50 | 400 | 2000 |
| 2,2'-DiCB | 4 | 0.2 | 1.0 | 5.0 | 50 | 400 | 2000 |
| 4,4'-DiCB | 15 | 0.2 | 1.0 | 5.0 | 50 | 400 | 2000 |
| 2,2',6-TrCB | 19 | 0.2 | 1.0 | 5.0 | 50 | 400 | 2000 |
| 3,4,4'-TrCB | 37 | 0.2 | 1.0 | 5.0 | 50 | 400 | 2000 |
| 2,2',6,6'-TeCB | 54 | 0.2 | 1.0 | 5.0 | 50 | 400 | 2000 |
| 3,3',4,4'-TeCB | 77 | 0.2 | 1.0 | 5.0 | 50 | 400 | 2000 |
| 3,4,4',5-TeCB | 81 | 0.2 | 1.0 | 5.0 | 50 | 400 | 2000 |
| 2,2',4,6,6'-PeCB | 104 | 0.2 | 1.0 | 5.0 | 50 | 400 | 2000 |
| 2,3,3',4,4'-PeCB | 105 | 0.2 | 1.0 | 5.0 | 50 | 400 | 2000 |
| 2,3,4,4',5-PeCB | 114 | 0.2 | 1.0 | 5.0 | 50 | 400 | 2000 |
| 2,3',4,4',5-PeCB | 118 | 0.2 | 1.0 | 5.0 | 50 | 400 | 2000 |
| 2',3,4,4',5-PeCB | 123 | 0.2 | 1.0 | 5.0 | 50 | 400 | 2000 |
| 3,3',4,4',5-PeCB | 126 | 0.2 | 1.0 | 5.0 | 50 | 400 | 2000 |
| 2,2',4,4',6,6'-HxCB | 155 | 0.2 | 1.0 | 5.0 | 50 | 400 | 2000 |
| 2,3,3',4,4',5-HxCB | 156 | 0.2 | 1.0 | 5.0 | 50 | 400 | 2000 |
| 2,3,3',4,4',5'-HxCB | 157 | 0.2 | 1.0 | 5.0 | 50 | 400 | 2000 |
| 2,3',4,4',5,5'-HxCB | 167 | 0.2 | 1.0 | 5.0 | 50 | 400 | 2000 |
| 3,3',4,4',5,5'-HxCB | 169 | 0.2 | 1.0 | 5.0 | 50 | 400 | 2000 |
| 2,2',3,4',5,6,6'-HpCB | 188 | 0.2 | 1.0 | 5.0 | 50 | 400 | 2000 |
| 2,3,3',4,4',5,5'-HpCB | 189 | 0.2 | 1.0 | 5.0 | 50 | 400 | 2000 |
| 2,2',3,3',5,5',6,6'-OcCB | 202 | 0.2 | 1.0 | 5.0 | 50 | 400 | 2000 |
| 2,3,3',4,4',5,5',6-OcCB | 205 | 0.2 | 1.0 | 5.0 | 50 | 400 | 2000 |
| 2,2',3,3',4,4',5,5',6-NoCB | 206 | 0.2 | 1.0 | 5.0 | 50 | 400 | 2000 |
| 2,2',3,3',4,5,5',6,6'-NoCB | 208 | 0.2 | 1.0 | 5.0 | 50 | 400 | 2000 |
| DeCB | 209 | 0.2 | 1.0 | 5.0 | 50 | 400 | 2000 |

| ¹³ C-Labeled Toxics/ LOC / Window-defining | IUPAC | CS0.2 (Hi sens) | CS1 | Concentration (ng/ml) | | CS4 | CS5 |
|---|-------|--------------------|-----|-----------------------|--------------|-----|-----|
| | | | | CS2 | CS3 (VER) | | |
| ¹³ C ₁₂ -2-MoCB | 1L | 100 | 100 | 100 | 100 | 100 | 100 |
| ¹³ C ₁₂ -4-MoCB | 3L | 100 | 100 | 100 | 100 | 100 | 100 |
| ¹³ C ₁₂ -2,2'-DiCB | 4L | 100 | 100 | 100 | 100 | 100 | 100 |
| ¹³ C ₁₂ -4,4'-DiCB | 15L | 100 | 100 | 100 | 100 | 100 | 100 |
| ¹³ C ₁₂ -2,2',6-TrCB | 19L | 100 | 100 | 100 | 100 | 100 | 100 |
| ¹³ C ₁₂ -3,4,4'-Tr-CB | 37L | 100 | 100 | 100 | 100 | 100 | 100 |
| ¹³ C ₁₂ -2,2',6,6'-TeCB | 54L | 100 | 100 | 100 | 100 | 100 | 100 |
| ¹³ C ₁₂ -3,3',4,4'-TeCB | 77L | 100 | 100 | 100 | 100 | 100 | 100 |
| ¹³ C ₁₂ -3,4,4',5-TeCB | 81L | 100 | 100 | 100 | 100 | 100 | 100 |
| ¹³ C ₁₂ -2,2',4,6,6'-PeCB | 104L | 100 | 100 | 100 | 100 | 100 | 100 |
| ¹³ C ₁₂ -2,3,3',4,4'-PeCB | 105L | 100 | 100 | 100 | 100 | 100 | 100 |
| ¹³ C ₁₂ -2,3,4,4',5-PeCB | 114L | 100 | 100 | 100 | 100 | 100 | 100 |
| ¹³ C ₁₂ -2,3',4,4',5-PeCB | 118L | 100 | 100 | 100 | 100 | 100 | 100 |
| ¹³ C ₁₂ -2',3,4,4',5-PeCB | 123L | 100 | 100 | 100 | 100 | 100 | 100 |
| ¹³ C ₁₂ -3,3',4,4',5-PeCB | 126L | 100 | 100 | 100 | 100 | 100 | 100 |
| ¹³ C ₁₂ -2,2',4,4',6,6'-HxCB | 155L | 100 | 100 | 100 | 100 | 100 | 100 |
| ¹³ C ₁₂ -2,3,3',4,4',5-HxCB | 156L | 100 | 100 | 100 | 100 | 100 | 100 |
| ¹³ C ₁₂ -2,3,3',4,4',5'-HxCB | 157L | 100 | 100 | 100 | 100 | 100 | 100 |
| ¹³ C ₁₂ -2,3',4,4',5,5'-HxCB | 167L | 100 | 100 | 100 | 100 | 100 | 100 |
| ¹³ C ₁₂ -3,3',4,4',5,5'-HxCB | 169L | 100 | 100 | 100 | 100 | 100 | 100 |
| ¹³ C ₁₂ -2,2',3,4',5,6,6'-HpCB | 188L | 100 | 100 | 100 | 100 | 100 | 100 |
| ¹³ C ₁₂ -2,3,3',4,4',5,5'-HpCB | 189L | 100 | 100 | 100 | 100 | 100 | 100 |
| ¹³ C ₁₂ -2,2',3,3',5,5',6,6'-OcCB | 202L | 100 | 100 | 100 | 100 | 100 | 100 |
| ¹³ C ₁₂ -2,3,3',4,4',5,5',6-OcCB | 205L | 100 | 100 | 100 | 100 | 100 | 100 |
| ¹³ C ₁₂ -2,2',3,3',4,4',5,5',6-NoCB | 206L | 100 | 100 | 100 | 100 | 100 | 100 |
| ¹³ C ₁₂ -2,2',3,3',4,5,5',6,6'-NoCB | 208L | 100 | 100 | 100 | 100 | 100 | 100 |
| ¹³ C ₁₂ -DeCB | 209L | 100 | 100 | 100 | 100 | 100 | 100 |

¹³C-Labeled Clean-Up

| | | | | | | | |
|--|------|-----|-----|-----|-----|-----|-----|
| ¹³ C ₁₂ -2,4,4'-TrCB | 28L | 100 | 100 | 100 | 100 | 100 | 100 |
| ¹³ C ₁₂ -2,3,3',5,5'-PeCB | 111L | 100 | 100 | 100 | 100 | 100 | 100 |
| ¹³ C ₁₂ -2,2',3,3',5,5',6-HpCB | 178L | 100 | 100 | 100 | 100 | 100 | 100 |

¹³C-Labeled Injection Internal

| | | | | | | | |
|---|------|-----|-----|-----|-----|-----|-----|
| ¹³ C ₁₂ -2,5-DiCB | 9L | 100 | 100 | 100 | 100 | 100 | 100 |
| ¹³ C ₁₂ -2,2',5,5'-TeCB | 52L | 100 | 100 | 100 | 100 | 100 | 100 |
| ¹³ C ₁₂ -2,2',4,5,5'-PeCB | 101L | 100 | 100 | 100 | 100 | 100 | 100 |
| ¹³ C ₁₂ -2,2',3,4,4',5'-HxCB | 138L | 100 | 100 | 100 | 100 | 100 | 100 |
| ¹³ C ₁₂ -2,2',3,3',4,4',5,5'-OcCB | 194L | 100 | 100 | 100 | 100 | 100 | 100 |

| | | |
|--------------------|--|----------------|
| EC-4976 | PCB Calibration Solutions CS1 - CS5 (unlabeled/ ¹³ C ₁₂ , 99%) | Set 5 X 0.2 ml |
| EC-4976-0.2 | PCB Calibration Solution CS0.2, High Sensitivity (unlabeled/ ¹³ C ₁₂ , 99%) | 0.2 ml |
| EC-4976-3 | PCB Calibration Solution CS3, Verification (unlabeled/ ¹³ C ₁₂ , 99%) | 0.2 ml |

EC-4977**Labeled Toxics/LOC/Window Defining Solution (¹³C₁₂, 99%)**

1.2 ml

| ¹³ C-Labeled Congener | IUPAC | Concentration (µg/ml) |
|---|-------|-----------------------|
| ¹³ C ₁₂ -2-MoCB | 1L | 1.0 |
| ¹³ C ₁₂ -4-MoCB | 3L | 1.0 |
| ¹³ C ₁₂ -2,2'-DiCB | 4L | 1.0 |
| ¹³ C ₁₂ -4,4'-DiCB | 15L | 1.0 |
| ¹³ C ₁₂ -2,2',6-TrCB | 19L | 1.0 |
| ¹³ C ₁₂ -3,4,4'-TrCB | 37L | 1.0 |
| ¹³ C ₁₂ -2,2',6,6'-TeCB | 54L | 1.0 |
| ¹³ C ₁₂ -3,3',4,4'-TeCB | 77L | 1.0 |
| ¹³ C ₁₂ -3,4,4',5-TeCB | 81L | 1.0 |
| ¹³ C ₁₂ -2,2',4,6,6'-PeCB | 104L | 1.0 |
| ¹³ C ₁₂ -2,3,3',4,4'-PeCB | 105L | 1.0 |
| ¹³ C ₁₂ -2,3,4,4',5-PeCB | 114L | 1.0 |
| ¹³ C ₁₂ -2,3',4,4',5-PeCB | 118L | 1.0 |
| ¹³ C ₁₂ -2',3,4,4',5-PeCB | 123L | 1.0 |
| ¹³ C ₁₂ -3,3',4,4',5-PeCB | 126L | 1.0 |
| ¹³ C ₁₂ -2,2',4,4',6,6'-HxCB | 155L | 1.0 |
| ¹³ C ₁₂ -2,3,3',4,4',5-HxCB | 156L | 1.0 |
| ¹³ C ₁₂ -2,3,3',4,4',5'-HxCB | 157L | 1.0 |
| ¹³ C ₁₂ -2,3',4,4',5,5'-HxCB | 167L | 1.0 |
| ¹³ C ₁₂ -3,3',4,4',5,5'-HxCB | 169L | 1.0 |
| ¹³ C ₁₂ -2,2',3,4',5,6,6'-HpCB | 188L | 1.0 |
| ¹³ C ₁₂ -2,3,3',4,4',5,5'-HpCB | 189L | 1.0 |
| ¹³ C ₁₂ -2,2',3,3',5,5',6,6'-OcCB | 202L | 1.0 |
| ¹³ C ₁₂ -2,3,3',4,4',5,5',6-OcCB | 205L | 1.0 |
| ¹³ C ₁₂ -2,2',3,3',4,4',5,5',6-NoCB | 206L | 1.0 |
| ¹³ C ₁₂ -2,2',3,3',4,5,5',6,6'-NoCB | 208L | 1.0 |
| ¹³ C ₁₂ -DeCB | 209L | 1.0 |

EC-4978**Labeled Clean-up Standard Solution (¹³C₁₂, 99%)**

1.2 ml

| ¹³ C-Labeled Congener | IUPAC | Concentration (µg/ml) |
|--|-------|-----------------------|
| ¹³ C ₁₂ -2,4,4'-TrCB | 28L | 1.0 |
| ¹³ C ₁₂ -2,3,3',5,5'-PeCB | 111L | 1.0 |
| ¹³ C ₁₂ -2,2',3,3',5,5',6-HpCB | 178L | 1.0 |

EC-4979**Labeled Injection Internal Standard Solution (¹³C₁₂, 99%)**

1.2 ml

| ¹³ C-Labeled Congener | IUPAC | Concentration (µg/ml) |
|---|-------|-----------------------|
| ¹³ C ₁₂ -2,5-DiCB | 9L | 5.0 |
| ¹³ C ₁₂ -2,2',5,5'-TeCB | 52L | 5.0 |
| ¹³ C ₁₂ -2,2',4,5,5'-PeCB | 101L | 5.0 |
| ¹³ C ₁₂ -2,2',3,4,4',5'-HxCB | 138L | 5.0 |
| ¹³ C ₁₂ -2,2',3,3',4,4',5,5'-OcCB | 194L | 5.0 |

EC-4989**Native Toxics/LOC Solution**

1.2 ml

| Native Congener | IUPAC | Concentration (µg/ml) |
|----------------------------|-------|-----------------------|
| 2-MoCB | 1 | 2.0 |
| 4-MoCB | 3 | 2.0 |
| 2,2'-DiCB | 4 | 2.0 |
| 4,4'-DiCB | 15 | 2.0 |
| 2,2',6-TrCB | 19 | 2.0 |
| 3,4,4'-TrCB | 37 | 2.0 |
| 2,2',6,6'-TeCB | 54 | 2.0 |
| 3,3',4,4'-TeCB | 77 | 2.0 |
| 3,4,4',5-TeCB | 81 | 2.0 |
| 2,2',4,6,6'-PeCB | 104 | 2.0 |
| 2,3,3',4,4'-PeCB | 105 | 2.0 |
| 2,3,4,4',5-PeCB | 114 | 2.0 |
| 2,3',4,4',5-PeCB | 118 | 2.0 |
| 2',3,4,4',5-PeCB | 123 | 2.0 |
| 3,3',4,4',5-PeCB | 126 | 2.0 |
| 2,2',4,4',6,6'-HxCB | 155 | 2.0 |
| 2,3,3',4,4',5-HxCB | 156 | 2.0 |
| 2,3,3',4,4',5'-HxCB | 157 | 2.0 |
| 2,3',4,4',5,5'-HxCB | 167 | 2.0 |
| 3,3',4,4',5,5'-HxCB | 169 | 2.0 |
| 2,2',3,4',5,6,6'-HpCB | 188 | 2.0 |
| 2,3,3',4,4',5,5'-HpCB | 189 | 2.0 |
| 2,2',3,3',5,5',6,6'-OcCB | 202 | 2.0 |
| 2,3,3',4,4',5,5',6-OcCB | 205 | 2.0 |
| 2,2',3,3',4,4',5,5',6-NoCB | 206 | 2.0 |
| 2,2',3,3',4,5,5',6,6'-NoCB | 208 | 2.0 |
| DeCB | 209 | 2.0 |



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