

## **Standards Preparation**

Integrated Atmospheric Deposition Network

O'Neill School of Public and Environmental Affairs

Indiana University

Bloomington, IN 47405

January 2024

For each compounds suite, there are seven types of standards that are used. A brief description of each one is provided below:

1. Stock solutions: used to prepare most of the working standards
2. Calibration standards: used for instrument calibration
3. Common reference standards (CRS): used for checking the instrument's calibration
4. Internal standards (IS): used for quantitation of target compounds
5. Surrogate standards (SS): used for evaluating recovery efficiency of each sample.
6. Matrix spike (MS) recovery standard: used for evaluating overall lab processing efficiency of target compounds
7. Linearity standards: used for determining instrument performance over a range of concentrations.
8. Instrument detection limit (IDL) standards: used for determining the detection limit of the instrument.

## PCB STANDARDS

**Table 1. Table of PCB custom and individual stock standards and their concentrations**

| Standard          | Vendor       | Mixture Type     | Purpose              | Concentration (µg/mL) |
|-------------------|--------------|------------------|----------------------|-----------------------|
| S-8074-AR1        | AccuStandard | Mixture          | PCB Calibration Std  | 1.0 ; 0.5             |
| S-8074-BR1        | AccuStandard | Mixture          | PCB Calibration Std  | 1.0 ; 0.5             |
| S-8074-CR1        | AccuStandard | Mixture          | PCB Calibration Std  | 1.0 ; 0.5             |
| C-IADN-01         | AccuStandard | Mixture          | Common Reference Std | 30.0                  |
| C-IADN-02         | AccuStandard | Mixture          | Common Reference Std | 30.0                  |
| C-IADN-03         | AccuStandard | Mixture          | Common Reference Std | 30.0                  |
| S-75951           | Agilent      | Mixture          | PCB Linearity        | 547.216               |
| S-97607           | Agilent      | Mixture          | PCB MS Vials         | 1.0 ; 1.5             |
| CUS 937           | Ultra        | Mixture          | PCB IDL              | 52.79                 |
| rpc-017s; PCB 14  | Agilent      | Individual Cong. | PCB Surrogate Std    | 100.0                 |
| rpc-028s; PCB 65  | Agilent      | Individual Cong. | PCB Surrogate Std    | 100.0                 |
| rpc-115s; PCB 166 | Agilent      | Individual Cong. | PCB Surrogate Std    | 100.0                 |
| rpc-020s; PCB 30  | Agilent      | Individual Cong. | PCB Internal Std     | 100.0                 |
| rpc-078s; PCB 204 | Agilent      | Individual Cong. | PCB Internal Std     | 100.0                 |
| rpc-010s; PCB 11  | Agilent      | Individual Cong. | PCB MS Vials         | 100.0                 |
| rpc-090s; PCB 169 | Agilent      | Individual Cong. | PCB MS Vials         | 100.0                 |
| rpc-102s; PCB 126 | Agilent      | Individual Cong. | PCB MS Vials         | 100.0                 |

**Table 2. Table of individual PCB congeners and their concentrations in custom stock mixtures S-8074-AR1, S-8074-BR1, and S-8074-CR1.**

| <b>S-8074-AR1</b> | <b>S-8074-AR1</b> | <b>S-8074-BR1</b> | <b>S-8074-BR1</b> | <b>S-8074-CR1</b> | <b>S-8074-CR1</b> |
|-------------------|-------------------|-------------------|-------------------|-------------------|-------------------|
| <b>PCB</b>        | <b>µg/mL</b>      | <b>PCB</b>        | <b>µg/mL</b>      | <b>PCB</b>        | <b>µg/mL</b>      |
| 4                 | 1.0               | 5                 | 1.0               | 6                 | 1.0               |
| 7                 | 1.0               | 8                 | 1.0               | 9                 | 1.0               |
| 10                | 1.0               | 12                | 1.0               | 13                | 1.0               |
| 15                | 1.0               | 16                | 0.5               | 17                | 0.5               |
| 18                | 0.5               | 19                | 0.5               | 22                | 0.5               |
| 28                | 0.5               | 26                | 0.5               | 31                | 0.5               |
| 32                | 0.5               | 33                | 0.5               | 37                | 0.5               |
| 41                | 0.5               | 42                | 0.5               | 44                | 0.5               |
| 45                | 0.5               | 47                | 0.5               | 48                | 0.5               |
| 52                | 0.5               | 49                | 0.5               | 53                | 0.5               |
| 56                | 0.5               | 60                | 0.5               | 64                | 0.5               |
| 66                | 0.5               | 70                | 0.5               | 71                | 0.5               |
| 74                | 0.5               | 76                | 0.5               | 77                | 0.5               |
| 81                | 0.5               | 83                | 0.5               | 84                | 0.5               |
| 85                | 0.5               | 87                | 0.5               | 89                | 0.5               |
| 91                | 0.5               | 92                | 0.5               | 95                | 0.5               |
| 97                | 0.5               | 99                | 0.5               | 100               | 0.5               |
| 101               | 0.5               | 105               | 0.5               | 110               | 0.5               |
| 114               | 0.5               | 118               | 0.5               | 119               | 0.5               |
| 123               | 0.5               | 126               | 0.5               | 128               | 0.5               |
| 131               | 0.5               | 132               | 0.5               | 135               | 0.5               |
| 138               | 0.5               | 144               | 0.5               | 149               | 0.5               |
| 153               | 0.5               | 156               | 0.5               | 163               | 0.5               |
| 167               | 0.5               | 169               | 0.5               | 170               | 0.5               |
| 171               | 0.5               | 172               | 0.5               | 174               | 0.5               |
| 180               | 0.5               | 190               | 0.5               | 194               | 0.5               |
| 200               | 0.5               | 199               | 0.5               | 202               | 0.5               |
| 205               | 0.5               | 206               | 0.5               | 207               | 0.5               |

**Table 3. Table of individual PCB congeners in custom stock mixtures C-IADN-01, C-IADN-02, and C-IADN-03 and their associated concentrations ( $\mu\text{g/mL}$ ).**

| <b>C-IADN-01</b> | <b>C-IADN-02</b> | <b>C-IADN-03</b> | <b>Conc., <math>\mu\text{g/mL}</math></b> |
|------------------|------------------|------------------|---|
| 4                | 5                | 6                | 30.0                                      |
| 7                | 8                | 9                | 30.0                                      |
| 10               | 12               | 13               | 30.0                                      |
| 15               | 16               | 17               | 30.0                                      |
| 18               | 19               | 22               | 30.0                                      |
| 28               | 26               | 31               | 30.0                                      |
| 32               | 33               | 37               | 30.0                                      |
| 41               | 42               | 44               | 30.0                                      |
| 45               | 47               | 48               | 30.0                                      |
| 52               | 49               | 53               | 30.0                                      |
| 56               | 60               | 64               | 30.0                                      |
| 66               | 70               | 71               | 30.0                                      |
| 74               | 76               | 77               | 30.0                                      |
| 81               | 83               | 84               | 30.0                                      |
| 85               | 87               | 89               | 30.0                                      |
| 91               | 92               | 95               | 30.0                                      |
| 97               | 99               | 100              | 30.0                                      |
| 101              | 105              | 110              | 30.0                                      |
| 114              | 118              | 119              | 30.0                                      |
| 123              | 126              | 128              | 30.0                                      |
| 131              | 132              | 135              | 30.0                                      |
| 138              | 144              | 149              | 30.0                                      |
| 153              | 156              | 163              | 30.0                                      |
| 167              | 169              | 170              | 30.0                                      |
| 171              | 172              | 174              | 30.0                                      |
| 180              | 190              | 194              | 30.0                                      |
| 200              | 199              | 202              | 30.0                                      |
| 205              | 206              | 207              | 30.0                                      |

**Table 4. Table of individual PCB congeners and their concentrations in the custom stock mixture S-75951 (total concentration of 547.216 µg/mL).**

| <b>Congener</b> | <b>Conc. µg/mL</b> | <b>Congener</b> | <b>Conc. µg/mL</b> | <b>Congener</b> | <b>Conc. µg/mL</b> |
|-----------------|--------------------|-----------------|--------------------|-----------------|--------------------|
| 4+10            | 13.696             | 37              | 4.843              | 123+149         | 11.239             |
| 7+9             | 4.805              | 42              | 5.600              | 118             | 4.814              |
| 6               | 7.615              | 41+71           | 9.292              | 114             | 0.522              |
| 5+8             | 56.14              | 64              | 7.200              | 131             | 0.120              |
| 19              | 1.120              | 100             | 0.445              | 105+132+153     | 17.306             |
| 11              | 20.200             | 74              | 7.646              | 163+138         | 10.865             |
| 12              | 6.814              | 70+76           | 13.674             | 126             | 4.016              |
| 13              | 0.392              | 66              | 21.010             | 128             | 0.402              |
| 18              | 14.900             | 95              | 8.032              | 167             | 0.199              |
| 17+15           | 14.948             | 91              | 2.040              | 174             | 12.810             |
| 16              | 8.016              | 56+60           | 14.063             | 202+171         | 3.176              |
| 32              | 7.615              | 84+92           | 7.236              | 156             | 0.264              |
| 26              | 2.817              | 89              | 0.410              | 172             | 2.242              |
| 31              | 18.820             | 101             | 7.222              | 180             | 24.400             |
| 28              | 18.820             | 99              | 2.966              | 199             | 1.725              |
| 33              | 13.210             | 119             | 0.110              | 169             | 4.024              |
| 53              | 2.600              | 83              | 0.602              | 170+190         | 6.810              |
| 22              | 11.600             | 97              | 2.260              | 201             | 16.830             |
| 45              | 3.600              | 81              | 0.642              | 207             | 0.374              |
| 52              | 18.110             | 87              | 4.000              | 194             | 7.214              |
| 49              | 9.292              | 85              | 2.806              | 205             | 0.441              |
| 47              | 4.004              | 77              | 0.935              | 206             | 2.747              |
| 48              | 4.024              | 110             | 7.638              |                 |                    |
| 44              | 17.290             | 135+144         | 3.564              |                 |                    |

**Table 5. Table of individual PCB congeners and their concentrations in the custom stock mixture S-97607 (total concentration of 90.5 µg/mL).**

| PCB | Conc. µg/mL | PCB | Conc. µg/mL | PCB | Conc. µg/mL |
|-----|-------------|-----|-------------|-----|-------------|
| 4   | 1.5         | 49  | 1.0         | 118 | 1.0         |
| 5   | 1.5         | 52  | 1.0         | 119 | 1.0         |
| 6   | 1.5         | 53  | 1.0         | 123 | 1.0         |
| 7   | 1.5         | 56  | 1.0         | 126 | 1.0         |
| 8   | 1.5         | 60  | 1.0         | 128 | 1.0         |
| 9   | 1.5         | 64  | 1.0         | 131 | 1.0         |
| 10  | 1.5         | 66  | 1.0         | 132 | 1.0         |
| 11  | 1.5         | 70  | 1.0         | 135 | 1.0         |
| 12  | 1.5         | 71  | 1.0         | 138 | 1.0         |
| 13  | 1.5         | 74  | 1.0         | 144 | 1.0         |
| 15  | 1.5         | 76  | 1.0         | 149 | 1.0         |
| 16  | 1.0         | 77  | 1.0         | 153 | 1.0         |
| 17  | 1.0         | 81  | 1.0         | 156 | 1.0         |
| 18  | 1.0         | 83  | 1.0         | 163 | 1.0         |
| 19  | 1.0         | 84  | 1.0         | 167 | 1.0         |
| 22  | 1.0         | 85  | 1.0         | 169 | 1.0         |
| 26  | 1.0         | 87  | 1.0         | 170 | 1.0         |
| 28  | 1.0         | 89  | 1.0         | 171 | 1.0         |
| 31  | 1.0         | 91  | 1.0         | 172 | 1.0         |
| 32  | 1.0         | 92  | 1.0         | 174 | 1.0         |
| 33  | 1.0         | 95  | 1.0         | 180 | 1.0         |
| 37  | 1.0         | 97  | 1.0         | 190 | 1.0         |
| 41  | 1.0         | 99  | 1.0         | 194 | 1.0         |
| 42  | 1.0         | 100 | 1.0         | 199 | 1.0         |
| 44  | 1.0         | 101 | 1.0         | 201 | 1.0         |
| 45  | 1.0         | 105 | 1.0         | 202 | 1.0         |
| 47  | 1.0         | 110 | 1.0         | 205 | 1.0         |
| 48  | 1.0         | 114 | 1.0         | 206 | 1.0         |
|     |             |     |             | 207 | 1.0         |

**Individual PCB Stock Solutions**

**Instructions:** Dilute 1 mL of a selected individual PCB standard to 50 mL in hexane as shown for PCB congeners 11, 14, 65, 126, 155, and 169 as shown in Table 6. Dilute 1 mL of a selected individual PCB standard to 100 mL in hexane as shown for PCB congeners 30, 166, and 204 as shown in Table 6. Each solution is prepared separately.

**Table 6. Table of individual PCB stock solutions.**

| Stock   | Conc.<br>µg/mL | Aliquot,<br>mL | Final Vol.,<br>mL | Final conc.,<br>ng/mL |
|---------|----------------|----------------|-------------------|-----------------------|
| PCB 11  | 100.0          | 1.0            | 50.0              | 2,000                 |
| PCB 14  | 100.0          | 1.0            | 50.0              | 2,000                 |
| PCB 30  | 100.0          | 1.0            | 100.0             | 1,000                 |
| PCB 65  | 100.0          | 1.0            | 50.0              | 2,000                 |
| PCB 126 | 100.0          | 1.0            | 50.0              | 2,000                 |
| PCB 155 | 100.0          | 1.0            | 50.0              | 2,000                 |
| PCB 166 | 100.0          | 1.0            | 100.0             | 1,000                 |
| PCB 169 | 100.0          | 1.0            | 50.0              | 2,000                 |
| PCB 204 | 100.0          | 1.0            | 100.0             | 1,000                 |

**PCB Internal Standard (IS)**

**Instructions:** Mix aliquot volumes of stock PCB-30 and -204 standards (Table 6) together as shown in Table 12 and dilute to 100 mL in hexane.

**Table 7. Table of working PCB internal standard.**

| Stock   | Stock conc.,<br>ng/mL | Aliquot,<br>mL | Final Vol.,<br>mL | Final Conc.,<br>ng/mL |
|---------|-----------------------|----------------|-------------------|-----------------------|
| PCB-30  | 1,000                 | 8.0            | 100               | 80                    |
| PCB-204 | 1,000                 | 6.0            | 100               | 60                    |

**PCB / Pesticide Surrogate Recovery Standard (SS)**

**Instructions:** Mix aliquot volumes individual PCB and Pesticide stock standards (Tables 6 and 25) together as shown in Table 8 and dilute to 100 mL in hexane. Information on preparation of pesticide stock solutions can be found in the Pesticide section (Table 25).

**Table 8. Table of PCB / Pesticide surrogate recovery standard working mixture.**

| Stock   | Stock Conc.,<br>ng/mL | Aliquot,<br>mL | Final Vol.,<br>mL | Final Conc.,<br>ng/mL |
|---------|-----------------------|----------------|-------------------|-----------------------|
| PCB-14  | 2,000                 | 10.0           | 100               | 200                   |
| PCB-65  | 2,000                 | 2.5            | 100               | 50                    |
| PCB-166 | 1,000                 | 5.0            | 100               | 50                    |
| DBC     | 2,000                 | 10.0           | 100               | 200                   |
| δ-HCH   | 2,000                 | 10.0           | 100               | 200                   |
| ε-HCH   | 1,000                 | 10.0           | 100               | 200                   |

**PCB Common Calibration Standard**

**Instructions:** Mix indicated aliquot volumes of selected PCB and Pesticide standards together and dilute to 100 mL in hexane as shown in Table 9. For preparation of individual pesticide stock solutions, see Table 25.

Table 9. Table of PCB Common Calibration Standard.

| Stock             | PCB / Pesticide | Stock Conc.,<br>µg/mL | Aliquot,<br>mL | Final Vol.,<br>mL | Final Conc.,<br>ng/mL |
|-------------------|-----------------|-----------------------|----------------|-------------------|-----------------------|
| <i>S-8074-AR1</i> |                 |                       | <i>1.0</i>     | <i>100</i>        |                       |
|                   | 4               | 1.0                   | 1.0            | 100               | 10.0                  |
|                   | 7               | 1.0                   | 1.0            | 100               | 10.0                  |
|                   | 10              | 1.0                   | 1.0            | 100               | 10.0                  |
|                   | 15              | 1.0                   | 1.0            | 100               | 10.0                  |
|                   | 18              | 0.5                   | 1.0            | 100               | 5.0                   |
|                   | 28              | 0.5                   | 1.0            | 100               | 5.0                   |
|                   | 32              | 0.5                   | 1.0            | 100               | 5.0                   |
|                   | 41              | 0.5                   | 1.0            | 100               | 5.0                   |
|                   | 45              | 0.5                   | 1.0            | 100               | 5.0                   |
|                   | 52              | 0.5                   | 1.0            | 100               | 5.0                   |
|                   | 56              | 0.5                   | 1.0            | 100               | 5.0                   |
|                   | 66              | 0.5                   | 1.0            | 100               | 5.0                   |
|                   | 74              | 0.5                   | 1.0            | 100               | 5.0                   |
|                   | 81              | 0.5                   | 1.0            | 100               | 5.0                   |
|                   | 85              | 0.5                   | 1.0            | 100               | 5.0                   |
|                   | 91              | 0.5                   | 1.0            | 100               | 5.0                   |
|                   | 97              | 0.5                   | 1.0            | 100               | 5.0                   |
|                   | 101             | 0.5                   | 1.0            | 100               | 5.0                   |
|                   | 114             | 0.5                   | 1.0            | 100               | 5.0                   |
|                   | 123             | 0.5                   | 1.0            | 100               | 5.0                   |
|                   | 131             | 0.5                   | 1.0            | 100               | 5.0                   |
|                   | 138             | 0.5                   | 1.0            | 100               | 5.0                   |
|                   | 153             | 0.5                   | 1.0            | 100               | 5.0                   |
|                   | 167             | 0.5                   | 1.0            | 100               | 5.0                   |
|                   | 171             | 0.5                   | 1.0            | 100               | 5.0                   |
|                   | 180             | 0.5                   | 1.0            | 100               | 5.0                   |
|                   | 200             | 0.5                   | 1.0            | 100               | 5.0                   |
|                   | 205             | 0.5                   | 1.0            | 100               | 5.0                   |
| <i>S-8074-BR1</i> |                 |                       | <i>1.0</i>     | <i>100</i>        |                       |
|                   | 5               | 1.0                   | 1.0            | 100               | 10.0                  |
|                   | 8               | 1.0                   | 1.0            | 100               | 10.0                  |
|                   | 12              | 1.0                   | 1.0            | 100               | 10.0                  |
|                   | 16              | 0.5                   | 1.0            | 100               | 5.0                   |
|                   | 19              | 0.5                   | 1.0            | 100               | 5.0                   |
|                   | 26              | 0.5                   | 1.0            | 100               | 5.0                   |
|                   | 33              | 0.5                   | 1.0            | 100               | 5.0                   |
|                   | 42              | 0.5                   | 1.0            | 100               | 5.0                   |
|                   | 47              | 0.5                   | 1.0            | 100               | 5.0                   |
|                   | 49              | 0.5                   | 1.0            | 100               | 5.0                   |
|                   | 60              | 0.5                   | 1.0            | 100               | 5.0                   |
|                   | 70              | 0.5                   | 1.0            | 100               | 5.0                   |
|                   | 76              | 0.5                   | 1.0            | 100               | 5.0                   |



|  |                          |     |            |            |      |
|--|--------------------------|-----|------------|------------|------|
|  | 83                       | 0.5 | 1.0        | 100        | 5.0  |
|  | 87                       | 0.5 | 1.0        | 100        | 5.0  |
|  | 92                       | 0.5 | 1.0        | 100        | 5.0  |
|  | 99                       | 0.5 | 1.0        | 100        | 5.0  |
|  | 105                      | 0.5 | 1.0        | 100        | 5.0  |
|  | 118                      | 0.5 | 1.0        | 100        | 5.0  |
|  | 126                      | 0.5 | 1.0        | 100        | 5.0  |
|  | 132                      | 0.5 | 1.0        | 100        | 5.0  |
|  | 144                      | 0.5 | 1.0        | 100        | 5.0  |
|  | 156                      | 0.5 | 1.0        | 100        | 5.0  |
|  | 169                      | 0.5 | 1.0        | 100        | 5.0  |
|  | 172                      | 0.5 | 1.0        | 100        | 5.0  |
|  | 190                      | 0.5 | 1.0        | 100        | 5.0  |
|  | 199                      | 0.5 | 1.0        | 100        | 5.0  |
|  | 206                      | 0.5 | 1.0        | 100        | 5.0  |
|  | <i>S-8074-CR1</i>        |     | <i>1.0</i> | <i>100</i> |      |
|  | 6                        | 1.0 | 1.0        | 100        | 10.0 |
|  | 9                        | 1.0 | 1.0        | 100        | 10.0 |
|  | 13                       | 1.0 | 1.0        | 100        | 10.0 |
|  | 17                       | 0.5 | 1.0        | 100        | 5.0  |
|  | 22                       | 0.5 | 1.0        | 100        | 5.0  |
|  | 31                       | 0.5 | 1.0        | 100        | 5.0  |
|  | 37                       | 0.5 | 1.0        | 100        | 5.0  |
|  | 44                       | 0.5 | 1.0        | 100        | 5.0  |
|  | 48                       | 0.5 | 1.0        | 100        | 5.0  |
|  | 53                       | 0.5 | 1.0        | 100        | 5.0  |
|  | 64                       | 0.5 | 1.0        | 100        | 5.0  |
|  | 71                       | 0.5 | 1.0        | 100        | 5.0  |
|  | 77                       | 0.5 | 1.0        | 100        | 5.0  |
|  | 84                       | 0.5 | 1.0        | 100        | 5.0  |
|  | 89                       | 0.5 | 1.0        | 100        | 5.0  |
|  | 95                       | 0.5 | 1.0        | 100        | 5.0  |
|  | 100                      | 0.5 | 1.0        | 100        | 5.0  |
|  | 110                      | 0.5 | 1.0        | 100        | 5.0  |
|  | 119                      | 0.5 | 1.0        | 100        | 5.0  |
|  | 128                      | 0.5 | 1.0        | 100        | 5.0  |
|  | 135                      | 0.5 | 1.0        | 100        | 5.0  |
|  | 149                      | 0.5 | 1.0        | 100        | 5.0  |
|  | 163                      | 0.5 | 1.0        | 100        | 5.0  |
|  | 170                      | 0.5 | 1.0        | 100        | 5.0  |
|  | 174                      | 0.5 | 1.0        | 100        | 5.0  |
|  | 194                      | 0.5 | 1.0        | 100        | 5.0  |
|  | 202                      | 0.5 | 1.0        | 100        | 5.0  |
|  | 207                      | 0.5 | 1.0        | 100        | 5.0  |
|  | <i>Individual Stocks</i> |     |            |            |      |
|  | 11                       | 2.0 | 0.50       | 100        | 10.0 |
|  | 14                       | 2.0 | 0.50       | 100        | 10.0 |
|  | 30                       | 1.0 | 0.80       | 100        | 8.0  |

|  |                   |     |      |     |      |
|--|-------------------|-----|------|-----|------|
|  | 65                | 2.0 | 0.25 | 100 | 5.0  |
|  | 166               | 1.0 | 0.50 | 100 | 5.0  |
|  | 204               | 1.0 | 0.60 | 100 | 6.0  |
|  | <i>o,p'</i> -DDT  | 1.0 | 0.50 | 100 | 5.0  |
|  | <i>p,p'</i> -DDT  | 1.0 | 0.50 | 100 | 5.0  |
|  | <i>p,p'</i> -DDE  | 2.0 | 0.50 | 100 | 10.0 |
|  | Hexachlorobenzene | 1.0 | 2.0  | 100 | 20.0 |
|  | Trans-nonachlor   | 1.0 | 0.50 | 100 | 5.0  |
|  | Aldrin            | 1.0 | 0.50 | 100 | 5.0  |
|  | Octachlorostyrene | 2.0 | 0.25 | 100 | 5.0  |

**Table 10. Concentrations for individual PCB congeners and selected pesticides in the PCB Common Calibration Standard.**

| PCB / Pesticide | Conc., ng/mL | PCB / Pesticide  | Conc., ng/mL | PCB / Pesticide  | Conc., ng/mL |
|-----------------|--------------|------------------|--------------|------------------|--------------|
| 4+10            | 20           | 44               | 5            | 135+144          | 10           |
| 7+9             | 20           | 37               | 5            | 123+149          | 10           |
| 6               | 10           | 42               | 5            | 118              | 5            |
| 8+5             | 20           | 41+71            | 10           | 114              | 5            |
| HCB             | 20           | 64               | 5            | 131              | 5            |
| 14              | 10           | 100              | 5            | <i>o,p'</i> -DDT | 5            |
| 19              | 5            | OCS              | 5            | 132+153+105      | 15           |
| 30              | 8            | 74               | 5            | <i>p,p'</i> -DDT | 5            |
| 12              | 10           | 70+76            | 10           | 163+138          | 10           |
| 13              | 10           | 66               | 5            | 126              | 5            |
| 18              | 5            | 95               | 5            | 166              | 5            |
| 15+17           | 15           | 91               | 5            | 128              | 5            |
| 16              | 5            | 56+60            | 10           | 167              | 5            |
| 32              | 5            | 92+84            | 10           | 174              | 5            |
| 26              | 5            | 89               | 5            | 202+171          | 10           |
| 31              | 5            | 101              | 5            | 156              | 5            |
| 28              | 5            | 99               | 5            | 204              | 6            |
| 33              | 5            | <i>t</i> -Nona   | 5            | 172              | 5            |
| 53              | 5            | 119              | 5            | 180              | 5            |
| 22              | 5            | 83               | 5            | 199              | 5            |
| 45              | 5            | 97               | 5            | 169              | 5            |
| 52              | 5            | 81               | 5            | 170+190          | 10           |
| 49              | 5            | 87               | 5            | 201              | 5            |
| Aldrin          | 5            | 85               | 5            | 207              | 5            |
| 47              | 5            | <i>p,p'</i> -DDE | 10           | 194              | 5            |
| 48              | 5            | 77               | 5            | 205              | 5            |
| 65              | 5            | 110              | 5            | 206              | 5            |

**PCB Common Reference Standard**

*Stock Mixture*

**Instructions:** Dilute 0.5 mL of three PCB custom standard mixtures to 50 mL in hexane as shown in Table 11.

Table 11. Table of PCB Common Reference Stock mixture.

| Stock            | PCB Congener | Stock conc., $\mu\text{g/mL}$ | Aliquot, mL | Final Vol., mL | Final Conc., $\mu\text{g/mL}$ |
|------------------|--------------|-------------------------------|-------------|----------------|-------------------------------|
| <i>C-LADN-01</i> |              |                               | <b>0.5</b>  | <b>50</b>      |                               |
|                  | 4            | 30.0                          | 0.5         | 50             | 0.3                           |
|                  | 7            | 30.0                          | 0.5         | 50             | 0.3                           |
|                  | 10           | 30.0                          | 0.5         | 50             | 0.3                           |
|                  | 15           | 30.0                          | 0.5         | 50             | 0.3                           |
|                  | 18           | 30.0                          | 0.5         | 50             | 0.3                           |
|                  | 28           | 30.0                          | 0.5         | 50             | 0.3                           |
|                  | 32           | 30.0                          | 0.5         | 50             | 0.3                           |
|                  | 41           | 30.0                          | 0.5         | 50             | 0.3                           |
|                  | 45           | 30.0                          | 0.5         | 50             | 0.3                           |
|                  | 52           | 30.0                          | 0.5         | 50             | 0.3                           |
|                  | 56           | 30.0                          | 0.5         | 50             | 0.3                           |
|                  | 66           | 30.0                          | 0.5         | 50             | 0.3                           |
|                  | 74           | 30.0                          | 0.5         | 50             | 0.3                           |
|                  | 81           | 30.0                          | 0.5         | 50             | 0.3                           |
|                  | 85           | 30.0                          | 0.5         | 50             | 0.3                           |
|                  | 91           | 30.0                          | 0.5         | 50             | 0.3                           |
|                  | 97           | 30.0                          | 0.5         | 50             | 0.3                           |
|                  | 101          | 30.0                          | 0.5         | 50             | 0.3                           |
|                  | 114          | 30.0                          | 0.5         | 50             | 0.3                           |
|                  | 123          | 30.0                          | 0.5         | 50             | 0.3                           |
|                  | 131          | 30.0                          | 0.5         | 50             | 0.3                           |
|                  | 138          | 30.0                          | 0.5         | 50             | 0.3                           |
|                  | 153          | 30.0                          | 0.5         | 50             | 0.3                           |
|                  | 167          | 30.0                          | 0.5         | 50             | 0.3                           |
|                  | 171          | 30.0                          | 0.5         | 50             | 0.3                           |
|                  | 180          | 30.0                          | 0.5         | 50             | 0.3                           |
|                  | 200          | 30.0                          | 0.5         | 50             | 0.3                           |
|                  | 205          | 30.0                          | 0.5         | 50             | 0.3                           |
| <i>C-LADN-02</i> |              |                               | <b>0.5</b>  | <b>50</b>      |                               |
|                  | 5            | 30.0                          | 0.5         | 50             | 0.3                           |
|                  | 8            | 30.0                          | 0.5         | 50             | 0.3                           |
|                  | 12           | 30.0                          | 0.5         | 50             | 0.3                           |
|                  | 16           | 30.0                          | 0.5         | 50             | 0.3                           |
|                  | 19           | 30.0                          | 0.5         | 50             | 0.3                           |
|                  | 26           | 30.0                          | 0.5         | 50             | 0.3                           |
|                  | 33           | 30.0                          | 0.5         | 50             | 0.3                           |
|                  | 42           | 30.0                          | 0.5         | 50             | 0.3                           |
|                  | 47           | 30.0                          | 0.5         | 50             | 0.3                           |
|                  | 49           | 30.0                          | 0.5         | 50             | 0.3                           |
|                  | 60           | 30.0                          | 0.5         | 50             | 0.3                           |
|                  | 70           | 30.0                          | 0.5         | 50             | 0.3                           |
|                  | 76           | 30.0                          | 0.5         | 50             | 0.3                           |

|                  |     |      |            |           |     |
|------------------|-----|------|------------|-----------|-----|
|                  | 83  | 30.0 | 0.5        | 50        | 0.3 |
|                  | 87  | 30.0 | 0.5        | 50        | 0.3 |
|                  | 92  | 30.0 | 0.5        | 50        | 0.3 |
|                  | 99  | 30.0 | 0.5        | 50        | 0.3 |
|                  | 105 | 30.0 | 0.5        | 50        | 0.3 |
|                  | 118 | 30.0 | 0.5        | 50        | 0.3 |
|                  | 126 | 30.0 | 0.5        | 50        | 0.3 |
|                  | 132 | 30.0 | 0.5        | 50        | 0.3 |
|                  | 144 | 30.0 | 0.5        | 50        | 0.3 |
|                  | 156 | 30.0 | 0.5        | 50        | 0.3 |
|                  | 169 | 30.0 | 0.5        | 50        | 0.3 |
|                  | 172 | 30.0 | 0.5        | 50        | 0.3 |
|                  | 190 | 30.0 | 0.5        | 50        | 0.3 |
|                  | 199 | 30.0 | 0.5        | 50        | 0.3 |
|                  | 206 | 30.0 | 0.5        | 50        | 0.3 |
| <b>C-LADN-03</b> |     |      | <b>0.5</b> | <b>50</b> |     |
|                  | 6   | 30.0 | 0.5        | 50        | 0.3 |
|                  | 9   | 30.0 | 0.5        | 50        | 0.3 |
|                  | 13  | 30.0 | 0.5        | 50        | 0.3 |
|                  | 17  | 30.0 | 0.5        | 50        | 0.3 |
|                  | 22  | 30.0 | 0.5        | 50        | 0.3 |
|                  | 31  | 30.0 | 0.5        | 50        | 0.3 |
|                  | 37  | 30.0 | 0.5        | 50        | 0.3 |
|                  | 44  | 30.0 | 0.5        | 50        | 0.3 |
|                  | 48  | 30.0 | 0.5        | 50        | 0.3 |
|                  | 53  | 30.0 | 0.5        | 50        | 0.3 |
|                  | 64  | 30.0 | 0.5        | 50        | 0.3 |
|                  | 71  | 30.0 | 0.5        | 50        | 0.3 |
|                  | 77  | 30.0 | 0.5        | 50        | 0.3 |
|                  | 84  | 30.0 | 0.5        | 50        | 0.3 |
|                  | 89  | 30.0 | 0.5        | 50        | 0.3 |
|                  | 95  | 30.0 | 0.5        | 50        | 0.3 |
|                  | 100 | 30.0 | 0.5        | 50        | 0.3 |
|                  | 110 | 30.0 | 0.5        | 50        | 0.3 |
|                  | 119 | 30.0 | 0.5        | 50        | 0.3 |
|                  | 128 | 30.0 | 0.5        | 50        | 0.3 |
|                  | 135 | 30.0 | 0.5        | 50        | 0.3 |
|                  | 149 | 30.0 | 0.5        | 50        | 0.3 |
|                  | 163 | 30.0 | 0.5        | 50        | 0.3 |
|                  | 170 | 30.0 | 0.5        | 50        | 0.3 |
|                  | 174 | 30.0 | 0.5        | 50        | 0.3 |
|                  | 194 | 30.0 | 0.5        | 50        | 0.3 |
|                  | 202 | 30.0 | 0.5        | 50        | 0.3 |
|                  | 207 | 30.0 | 0.5        | 50        | 0.3 |

### ***Working Mixture***

**Instructions:** Mix aliquot volumes of PCB Common Reference Stock Solution (Table 11), selected individual PCB standards (Table 6), and individual pesticide standards (Table 25) together as show in Table 12 and dilute to 100 mL in hexane.

**Table 12. Table of PCB Common Reference Standard working mixture.**

| Stock             | Stock conc., $\mu\text{g/mL}$ | Aliquot, mL | Final Vol., mL | Final Conc., ng/mL |
|-------------------|-------------------------------|-------------|----------------|--------------------|
| PCB CRS Stock     | 0.3                           | 2.5         | 100            | 7.5                |
| PCB 30            | 1.0                           | 0.8         | 100            | 8.0                |
| PCB 204           | 1.0                           | 0.6         | 100            | 6.0                |
| PCB 14            | 2.0                           | 1.0         | 100            | 20.0               |
| PCB 65            | 2.0                           | 0.25        | 100            | 5.0                |
| PCB 166           | 1.0                           | 0.5         | 100            | 5.0                |
| PCB 11            | 2.0                           | 1.0         | 100            | 20.0               |
| HCB               | 1.0                           | 2.0         | 100            | 20.0               |
| Octachlorostyrene | 2.0                           | 1.0         | 100            | 20.0               |
| Aldrin            | 1.0                           | 2.0         | 100            | 20.0               |
| <u>O,p'</u> -DDT  | 1.0                           | 2.0         | 100            | 20.0               |
| <u>p,p'</u> -DDT  | 1.0                           | 2.0         | 100            | 20.0               |
| <u>p,p'</u> -DDE  | 2.0                           | 1.0         | 100            | 20.0               |
| T-Nona            | 1.0                           | 2.0         | 100            | 20.0               |

**Table 13. Concentration of PCB congeners and selected pesticides in the PCB Common Reference Standard working mixture.**

| PCB / Pesticide | Conc., ng/mL | PCB / Pesticide   | Conc., ng/mL | PCB / Pesticide  | Conc., ng/mL |
|-----------------|--------------|-------------------|--------------|------------------|--------------|
| 4+10            | 15           | 44                | 7.5          | 135+144          | 15           |
| 7+9             | 15           | 37                | 7.5          | 123+149          | 15           |
| 6               | 7.5          | 42                | 7.5          | 118              | 7.5          |
| 8+5             | 15           | 41+71             | 15           | 114              | 7.5          |
| HCB             | 20           | 64                | 7.5          | 131              | 7.5          |
| 14              | 20           | 100               | 7.5          | <u>o,p'</u> -DDT | 20           |
| 19              | 7.5          | Octachlorostyrene | 20           | 132+153+105      | 22.5         |
| 30              | 8            | 74                | 7.5          | <u>p,p'</u> -DDT | 20           |
| 12              | 7.5          | 70+76             | 15           | 163+138          | 15           |
| 13              | 7.5          | 66                | 7.5          | 126              | 7.5          |
| 18              | 7.5          | 95                | 7.5          | 166              | 5.0          |
| 15+17           | 15           | 91                | 7.5          | 128              | 7.5          |
| 16              | 7.5          | 56+60             | 15           | 167              | 7.5          |
| 32              | 7.5          | 92+84             | 15           | 174              | 7.5          |
| 26              | 7.5          | 89                | 7.5          | 202+171          | 15           |
| 31              | 7.5          | 101               | 7.5          | 156              | 7.5          |
| 28              | 7.5          | 99                | 7.5          | 204              | 6            |
| 33              | 7.5          | t-Nona            | 20.0         | 172              | 7.5          |
| 53              | 7.5          | 119               | 7.5          | 180              | 7.5          |
| 22              | 7.5          | 83                | 7.5          | 199              | 7.5          |
| 45              | 7.5          | 97                | 7.5          | 169              | 7.5          |
| 52              | 7.5          | 81                | 7.5          | 170+190          | 15           |
| 49              | 7.5          | 87                | 7.5          | 201              | 7.5          |
| Aldrin          | 20.0         | 85                | 7.5          | 207              | 7.5          |
| 47              | 7.5          | <u>p,p'</u> -DDE  | 20.0         | 194              | 7.5          |
| 48              | 7.5          | 77                | 7.5          | 205              | 7.5          |
| 65              | 5.0          | 110               | 7.5          | 206              | 7.5          |

### PCB Matrix Spike Recovery Standard

Instructions: Dilute aliquot of stock mixture S-97607 (Table 5) to 100 mL in hexane.

Table 14. Table of PCB matrix spike recovery standard working mixture.

| Stock          | PCB   | PCB conc.,<br>µg/mL | Aliquot,<br>mL | Final Vol.,<br>mL | Final Conc.,<br>ng/mL |
|----------------|-------|---------------------|----------------|-------------------|-----------------------|
| <i>S-97607</i> |       | <i>90.5</i>         | <i>0.1</i>     | <i>100</i>        | <i>90.5</i>           |
|                | 4+10  | 3.0                 | 0.1            | 100               | 3.0                   |
|                | 7+9   | 3.0                 | 0.1            | 100               | 3.0                   |
|                | 6     | 1.5                 | 0.1            | 100               | 1.5                   |
|                | 5+8   | 3.0                 | 0.1            | 100               | 3.0                   |
|                | 19    | 1.0                 | 0.1            | 100               | 1.0                   |
|                | 11    | 1.5                 | 0.1            | 100               | 1.5                   |
|                | 12    | 1.5                 | 0.1            | 100               | 1.5                   |
|                | 13    | 1.5                 | 0.1            | 100               | 1.5                   |
|                | 18    | 1.0                 | 0.1            | 100               | 1.0                   |
|                | 17+15 | 2.5                 | 0.1            | 100               | 2.5                   |
|                | 16    | 1.0                 | 0.1            | 100               | 1.0                   |
|                | 32    | 1.0                 | 0.1            | 100               | 1.0                   |
|                | 26    | 1.0                 | 0.1            | 100               | 1.0                   |
|                | 31    | 1.0                 | 0.1            | 100               | 1.0                   |
|                | 28    | 1.0                 | 0.1            | 100               | 1.0                   |
|                | 33    | 1.0                 | 0.1            | 100               | 1.0                   |
|                | 53    | 1.0                 | 0.1            | 100               | 1.0                   |
|                | 22    | 1.0                 | 0.1            | 100               | 1.0                   |
|                | 45    | 1.0                 | 0.1            | 100               | 1.0                   |
|                | 52    | 1.0                 | 0.1            | 100               | 1.0                   |
|                | 49    | 1.0                 | 0.1            | 100               | 1.0                   |
|                | 47    | 1.0                 | 0.1            | 100               | 1.0                   |
|                | 48    | 1.0                 | 0.1            | 100               | 1.0                   |
|                | 44    | 1.0                 | 0.1            | 100               | 1.0                   |
|                | 37    | 1.0                 | 0.1            | 100               | 1.0                   |
|                | 42    | 1.0                 | 0.1            | 100               | 1.0                   |
|                | 41+71 | 2.0                 | 0.1            | 100               | 2.0                   |
|                | 64    | 1.0                 | 0.1            | 100               | 1.0                   |
|                | 100   | 1.0                 | 0.1            | 100               | 1.0                   |
|                | 74    | 1.0                 | 0.1            | 100               | 1.0                   |
|                | 70+76 | 2.0                 | 0.1            | 100               | 2.0                   |
|                | 66    | 1.0                 | 0.1            | 100               | 1.0                   |
|                | 95    | 1.0                 | 0.1            | 100               | 1.0                   |
|                | 91    | 1.0                 | 0.1            | 100               | 1.0                   |
|                | 56+60 | 2.0                 | 0.1            | 100               | 2.0                   |
|                | 84+92 | 2.0                 | 0.1            | 100               | 2.0                   |
|                | 89    | 1.0                 | 0.1            | 100               | 1.0                   |
|                | 101   | 1.0                 | 0.1            | 100               | 1.0                   |
|                | 99    | 1.0                 | 0.1            | 100               | 1.0                   |
|                | 119   | 1.0                 | 0.1            | 100               | 1.0                   |

|  |             |     |     |     |     |
|--|-------------|-----|-----|-----|-----|
|  | 83          | 1.0 | 0.1 | 100 | 1.0 |
|  | 97          | 1.0 | 0.1 | 100 | 1.0 |
|  | 81          | 1.0 | 0.1 | 100 | 1.0 |
|  | 87          | 1.0 | 0.1 | 100 | 1.0 |
|  | 85          | 1.0 | 0.1 | 100 | 1.0 |
|  | 77          | 1.0 | 0.1 | 100 | 1.0 |
|  | 110         | 1.0 | 0.1 | 100 | 1.0 |
|  | 135+144     | 2.0 | 0.1 | 100 | 2.0 |
|  | 123+149     | 2.0 | 0.1 | 100 | 2.0 |
|  | 118         | 1.0 | 0.1 | 100 | 1.0 |
|  | 114         | 1.0 | 0.1 | 100 | 1.0 |
|  | 131         | 1.0 | 0.1 | 100 | 1.0 |
|  | 105+132+153 | 3.0 | 0.1 | 100 | 3.0 |
|  | 163+138     | 2.0 | 0.1 | 100 | 2.0 |
|  | 126         | 1.0 | 0.1 | 100 | 1.0 |
|  | 128         | 1.0 | 0.1 | 100 | 1.0 |
|  | 167         | 1.0 | 0.1 | 100 | 1.0 |
|  | 174         | 1.0 | 0.1 | 100 | 1.0 |
|  | 202+171     | 2.0 | 0.1 | 100 | 2.0 |
|  | 156         | 1.0 | 0.1 | 100 | 1.0 |
|  | 172         | 1.0 | 0.1 | 100 | 1.0 |
|  | 180         | 1.0 | 0.1 | 100 | 1.0 |
|  | 199         | 1.0 | 0.1 | 100 | 1.0 |
|  | 169         | 1.0 | 0.1 | 100 | 1.0 |
|  | 170+190     | 2.0 | 0.1 | 100 | 2.0 |
|  | 201         | 1.0 | 0.1 | 100 | 1.0 |
|  | 207         | 1.0 | 0.1 | 100 | 1.0 |
|  | 194         | 1.0 | 0.1 | 100 | 1.0 |
|  | 205         | 1.0 | 0.1 | 100 | 1.0 |
|  | 206         | 1.0 | 0.1 | 100 | 1.0 |

## PCB Linearity Standard

### *Stock Mixture*

**Instructions:** Dilute 100 µL of S-75951 (Table 4) standard to 10 mL with hexane as shown in Table 15.

**Table 15. Table of PCB Linearity Stock mixture.**

| Stock   | Stock conc.,<br>µg/mL | Aliquot,<br>mL | Final Vol.,<br>mL | Final conc.,<br>ng/mL |
|---------|-----------------------|----------------|-------------------|-----------------------|
| S-75951 | 547.216               | 0.1            | 10                | 5,472.16              |

### *Working Mixture*

**Instructions:** Perform serial dilutions of S-75951 standard (Table 15) to 3.0 mL in hexane as shown in Table 16.

**Table 16. Table of PCB Linearity working mixtures**

| Level | Stock               | Stock conc.,<br>ng/mL | Aliquot, mL | Final Vol.,<br>mL | Final conc.,<br>ng/mL |
|-------|---------------------|-----------------------|-------------|-------------------|-----------------------|
| 01    | S-75951             | 5,472.16              |             |                   | 5,472.16              |
| 02    | S-75951             | 5,472.16              | 1.0         | 3.0               | 1,824.05              |
| 03    | PCB Linearity<br>02 | 1,824.05              | 1.0         | 3.0               | 608.02                |
| 04    | PCB Linearity<br>03 | 608.02                | 1.0         | 3.0               | 202.67                |
| 05    | PCB Linearity<br>04 | 202.67                | 1.0         | 3.0               | 67.56                 |
| 06    | PCB Linearity<br>05 | 67.56                 | 1.0         | 3.0               | 22.52                 |
| 07    | PCB Linearity<br>06 | 22.52                 | 1.0         | 3.0               | 7.51                  |
| 08    | PCB Linearity<br>07 | 7.51                  | 1.0         | 3.0               | 2.50                  |
| 09    | PCB Linearity<br>08 | 2.50                  | 1.0         | 3.0               | 0.83                  |
| 10    | PCB Linearity<br>09 | 0.83                  | 1.0         | 3.0               | 0.27                  |

**Table 17. Concentration of PCB congeners in PCB Linearity working mixtures.**

| PCB   | Concentration (ng/mL) per Linearity Level |       |       |       |      |      |      |       |       |       |
|-------|---|-------|-------|-------|------|------|------|-------|-------|-------|
|       | 01  | 02    | 03    | 04    | 05   | 06   | 07   | 08    | 09    | 10    |
| 4+10  | 137.0                                     | 45.7  | 15.22 | 5.07  | 1.69 | 0.56 | 0.19 | 0.063 | 0.021 | 0.007 |
| 7+9   | 48.1                                      | 16.0  | 5.34  | 1.78  | 0.59 | 0.20 | 0.07 | 0.022 | 0.007 | 0.002 |
| 6     | 76.2                                      | 25.4  | 8.46  | 2.82  | 0.94 | 0.31 | 0.10 | 0.035 | 0.012 | 0.004 |
| 5+8   | 561.4                                     | 187.1 | 62.38 | 20.79 | 6.93 | 2.31 | 0.77 | 0.257 | 0.086 | 0.029 |
| 19    | 11.2                                      | 3.7   | 1.24  | 0.41  | 0.14 | 0.05 | 0.02 | 0.005 | 0.002 | 0.001 |
| 11    | 202.0                                     | 67.3  | 22.44 | 7.48  | 2.49 | 0.83 | 0.28 | 0.092 | 0.031 | 0.010 |
| 12    | 68.1                                      | 22.7  | 7.57  | 2.52  | 0.84 | 0.28 | 0.09 | 0.031 | 0.010 | 0.003 |
| 13    | 3.9                                       | 1.3   | 0.44  | 0.15  | 0.05 | 0.02 | 0.01 | 0.002 | 0.001 | 0.000 |
| 18    | 149.0                                     | 49.7  | 16.56 | 5.52  | 1.84 | 0.61 | 0.20 | 0.068 | 0.023 | 0.008 |
| 17+15 | 149.5                                     | 49.8  | 16.61 | 5.54  | 1.85 | 0.62 | 0.21 | 0.068 | 0.023 | 0.008 |
| 16    | 80.2                                      | 26.7  | 8.91  | 2.97  | 0.99 | 0.33 | 0.11 | 0.037 | 0.012 | 0.004 |
| 32    | 76.2                                      | 25.4  | 8.46  | 2.82  | 0.94 | 0.31 | 0.10 | 0.035 | 0.012 | 0.004 |
| 26    | 28.2                                      | 9.4   | 3.13  | 1.04  | 0.35 | 0.12 | 0.04 | 0.013 | 0.004 | 0.001 |
| 31    | 188.2                                     | 62.7  | 20.91 | 6.97  | 2.32 | 0.77 | 0.26 | 0.086 | 0.029 | 0.010 |
| 28    | 188.2                                     | 62.7  | 20.91 | 6.97  | 2.32 | 0.77 | 0.26 | 0.086 | 0.029 | 0.010 |
| 33    | 132.1                                     | 44.0  | 14.68 | 4.89  | 1.63 | 0.54 | 0.18 | 0.060 | 0.020 | 0.007 |
| 53    | 26.0                                      | 8.7   | 2.89  | 0.96  | 0.32 | 0.11 | 0.04 | 0.012 | 0.004 | 0.001 |
| 22    | 116.0                                     | 38.7  | 12.89 | 4.30  | 1.43 | 0.48 | 0.16 | 0.053 | 0.018 | 0.006 |
| 45    | 36.0                                      | 12.0  | 4.00  | 1.33  | 0.44 | 0.15 | 0.05 | 0.016 | 0.005 | 0.002 |
| 52    | 181.1                                     | 60.4  | 20.12 | 6.71  | 2.24 | 0.75 | 0.25 | 0.083 | 0.028 | 0.009 |
| 49    | 92.9                                      | 31.0  | 10.32 | 3.44  | 1.15 | 0.38 | 0.13 | 0.042 | 0.014 | 0.005 |
| 47    | 40.0                                      | 13.3  | 4.45  | 1.48  | 0.49 | 0.16 | 0.05 | 0.018 | 0.006 | 0.002 |
| 48    | 40.2                                      | 13.4  | 4.47  | 1.49  | 0.50 | 0.17 | 0.06 | 0.018 | 0.006 | 0.002 |
| 44    | 172.9                                     | 57.6  | 19.21 | 6.40  | 2.13 | 0.71 | 0.24 | 0.079 | 0.026 | 0.009 |
| 37    | 48.4                                      | 16.1  | 5.38  | 1.79  | 0.60 | 0.20 | 0.07 | 0.022 | 0.007 | 0.002 |



|                 |       |      |       |      |      |      |      |       |       |       |
|-----------------|-------|------|-------|------|------|------|------|-------|-------|-------|
| 42              | 56.0  | 18.7 | 6.22  | 2.07 | 0.69 | 0.23 | 0.08 | 0.026 | 0.009 | 0.003 |
| 41+71           | 92.9  | 31.0 | 10.32 | 3.44 | 1.15 | 0.38 | 0.13 | 0.042 | 0.014 | 0.005 |
| 64              | 72.0  | 24.0 | 8.00  | 2.67 | 0.89 | 0.30 | 0.10 | 0.033 | 0.011 | 0.004 |
| 100             | 4.5   | 1.5  | 0.49  | 0.16 | 0.05 | 0.02 | 0.01 | 0.002 | 0.001 | 0.000 |
| 74              | 76.5  | 25.5 | 8.50  | 2.83 | 0.94 | 0.31 | 0.10 | 0.035 | 0.012 | 0.004 |
| 70+76           | 136.7 | 45.6 | 15.19 | 5.06 | 1.69 | 0.56 | 0.19 | 0.063 | 0.021 | 0.007 |
| 66              | 210.1 | 70.0 | 23.34 | 7.78 | 2.59 | 0.86 | 0.29 | 0.096 | 0.032 | 0.011 |
| 95              | 80.3  | 26.8 | 8.92  | 2.97 | 0.99 | 0.33 | 0.11 | 0.037 | 0.012 | 0.004 |
| 91              | 20.4  | 6.8  | 2.27  | 0.76 | 0.25 | 0.08 | 0.03 | 0.009 | 0.003 | 0.001 |
| 56+60           | 140.6 | 46.9 | 15.63 | 5.21 | 1.74 | 0.58 | 0.19 | 0.064 | 0.021 | 0.007 |
| 84+92           | 72.4  | 24.1 | 8.04  | 2.68 | 0.89 | 0.30 | 0.10 | 0.033 | 0.011 | 0.004 |
| 89              | 4.1   | 1.4  | 0.46  | 0.15 | 0.05 | 0.02 | 0.01 | 0.002 | 0.001 | 0.000 |
| 101             | 72.2  | 24.1 | 8.02  | 2.67 | 0.89 | 0.30 | 0.10 | 0.033 | 0.011 | 0.004 |
| 99              | 29.7  | 9.9  | 3.30  | 1.10 | 0.37 | 0.12 | 0.04 | 0.014 | 0.005 | 0.002 |
| 119             | 1.1   | 0.4  | 0.12  | 0.04 | 0.01 | 0.00 | 0.00 | 0.001 | 0.000 | 0.000 |
| 83              | 6.0   | 2.0  | 0.67  | 0.22 | 0.07 | 0.02 | 0.01 | 0.003 | 0.001 | 0.000 |
| 97              | 22.6  | 7.5  | 2.51  | 0.84 | 0.28 | 0.09 | 0.03 | 0.010 | 0.003 | 0.001 |
| 81              | 6.4   | 2.1  | 0.71  | 0.24 | 0.08 | 0.03 | 0.01 | 0.003 | 0.001 | 0.000 |
| 87              | 40.0  | 13.3 | 4.44  | 1.48 | 0.49 | 0.16 | 0.05 | 0.018 | 0.006 | 0.002 |
| 85              | 28.1  | 9.4  | 3.12  | 1.04 | 0.35 | 0.12 | 0.04 | 0.013 | 0.004 | 0.001 |
| 77              | 9.4   | 3.1  | 1.04  | 0.35 | 0.12 | 0.04 | 0.01 | 0.004 | 0.001 | 0.000 |
| 110             | 76.4  | 25.5 | 8.49  | 2.83 | 0.94 | 0.31 | 0.10 | 0.035 | 0.012 | 0.004 |
| 135+144         | 35.6  | 11.9 | 3.96  | 1.32 | 0.44 | 0.15 | 0.05 | 0.016 | 0.005 | 0.002 |
| 123+149         | 112.4 | 37.5 | 12.49 | 4.16 | 1.39 | 0.46 | 0.15 | 0.051 | 0.017 | 0.006 |
| 118             | 48.1  | 16.0 | 5.35  | 1.78 | 0.59 | 0.20 | 0.07 | 0.022 | 0.007 | 0.002 |
| 114             | 5.2   | 1.7  | 0.58  | 0.19 | 0.06 | 0.02 | 0.01 | 0.002 | 0.001 | 0.000 |
| 131             | 1.2   | 0.4  | 0.13  | 0.04 | 0.01 | 0.00 | 0.00 | 0.001 | 0.000 | 0.000 |
| 105+132+15<br>3 | 173.1 | 57.7 | 19.23 | 6.41 | 2.14 | 0.71 | 0.24 | 0.079 | 0.026 | 0.009 |
| 163+138         | 108.7 | 36.2 | 12.07 | 4.02 | 1.34 | 0.45 | 0.15 | 0.050 | 0.017 | 0.006 |
| 126             | 40.2  | 13.4 | 4.46  | 1.49 | 0.50 | 0.17 | 0.06 | 0.018 | 0.006 | 0.002 |
| 128             | 4.0   | 1.3  | 0.45  | 0.15 | 0.05 | 0.02 | 0.01 | 0.002 | 0.001 | 0.000 |
| 167             | 2.0   | 0.7  | 0.22  | 0.07 | 0.02 | 0.01 | 0.00 | 0.001 | 0.000 | 0.000 |
| 174             | 128.1 | 42.7 | 14.23 | 4.74 | 1.58 | 0.53 | 0.18 | 0.059 | 0.020 | 0.007 |
| 202+171         | 31.8  | 10.6 | 3.53  | 1.18 | 0.39 | 0.13 | 0.04 | 0.015 | 0.005 | 0.002 |
| 156             | 2.6   | 0.9  | 0.29  | 0.10 | 0.03 | 0.01 | 0.00 | 0.001 | 0.000 | 0.000 |
| 172             | 22.4  | 7.5  | 2.49  | 0.83 | 0.28 | 0.09 | 0.03 | 0.010 | 0.003 | 0.001 |
| 180             | 244.0 | 81.3 | 27.11 | 9.04 | 3.01 | 1.00 | 0.33 | 0.112 | 0.037 | 0.012 |
| 199             | 17.3  | 5.8  | 1.92  | 0.64 | 0.21 | 0.07 | 0.02 | 0.008 | 0.003 | 0.001 |
| 169             | 40.2  | 13.4 | 4.47  | 1.49 | 0.50 | 0.17 | 0.06 | 0.018 | 0.006 | 0.002 |
| 170+190         | 68.1  | 22.7 | 7.57  | 2.52 | 0.84 | 0.28 | 0.09 | 0.031 | 0.010 | 0.003 |
| 201             | 168.3 | 56.1 | 18.70 | 6.23 | 2.08 | 0.69 | 0.23 | 0.077 | 0.026 | 0.009 |
| 207             | 3.7   | 1.2  | 0.42  | 0.14 | 0.05 | 0.02 | 0.01 | 0.002 | 0.001 | 0.000 |
| 194             | 72.1  | 24.0 | 8.02  | 2.67 | 0.89 | 0.30 | 0.10 | 0.033 | 0.011 | 0.004 |
| 205             | 4.4   | 1.5  | 0.49  | 0.16 | 0.05 | 0.02 | 0.01 | 0.002 | 0.001 | 0.000 |
| 206             | 27.5  | 9.2  | 3.05  | 1.02 | 0.34 | 0.11 | 0.04 | 0.013 | 0.004 | 0.001 |

## PCB IDL Standard

### *Stock Mixture*

**Instructions:** Dilute 1.0 mL of CUS 937 custom PCB mixture to 100 mL in hexane as shown in Table 18.

**Table 18. Table of the PCB IDL Stock Solution mixture.**

| Stock   | Stock conc.,<br>$\mu\text{g/mL}$ | Aliquot, mL | Final Vol.,<br>mL | Final conc.,<br>ng/mL |
|---------|----------------------------------|-------------|-------------------|-----------------------|
| CUS 937 | 52.79                            | 1.0         | 100               | 527.9                 |

### *Working Mixture*

**Instructions:** Dilute 1.0 mL of PCB IDL Stock Standard (Table 18) to 100 mL in hexane.

**Table 19. Table of the PCB IDL working mixture.**

| Stock   | Stock conc.,<br>ng/mL | Aliquot, mL | Final Vol.,<br>mL | Final conc.,<br>ng/mL |
|---------|-----------------------|-------------|-------------------|-----------------------|
| PCB IDL | 527.9                 | 1.0         | 100               | 5.279                 |

**Table 20. Table of individual PCB congeners and their concentrations in the PCB IDL working solution.**

| PCB | Conc., ng/mL |
|-----|--------------|
| 1   | 0.400        |
| 6   | 0.473        |
| 29  | 0.210        |
| 49  | 0.195        |
| 101 | 0.164        |
| 141 | 0.073        |
| 180 | 0.080        |
| 194 | 0.056        |
| 206 | 0.068        |
| 209 | 0.045        |

## PESTICIDE STANDARDS

### Pesticide custom and individual stock mixtures

**Table 21. Table of Pesticide abbreviations**

| Full Name         | Abbreviation |
|-------------------|--------------|
| Dibutylchlorodate | DBC          |
| Octachlorostyrene | OCS          |
| trans-Nonachlor   | t-Nona       |
| Hexachlorobenzene | HCB          |

**Table 22. Table of Pesticide custom and individual stock standards and their concentrations.**

| Standard                      | Vendor       | Mixture Type | Catalog ID | Conc., $\mu\text{g/ml}$ |
|-------------------------------|--------------|--------------|------------|-------------------------|
| US112B                        | Agilent      | Mixture      | ppm-508b-1 | 1,000                   |
| S-8206A                       | AccuStandard | Mixture      | S-8206A    | 100                     |
| $\alpha$ -Chlordane           | Agilent      | Individual   | pp-470-1   | 100                     |
| $\gamma$ -Chlordane           | Agilent      | Individual   | pp-480-1   | 100                     |
| trans-Nonachlor (t-Nona)      | Agilent      | Individual   | pp-500-1   | 100                     |
| Oxychlordane                  | AccuStandard | Individual   | p-331s-h   | 100                     |
| Dibutylchlorodate (DBC)       | Agilent      | Individual   | sts-280n-1 | 2,000                   |
| <i>p,p'</i> -DDD              | Agilent      | Individual   | pst-210s   | 100                     |
| <i>p,p'</i> -DDT              | Agilent      | Individual   | pst-271s   | 100                     |
| <i>p,p'</i> -DDD              | Agilent      | Individual   | pp-160-1   | 100                     |
| <i>p,p'</i> -DDE              | Agilent      | Individual   | pp-170-1   | 100                     |
| <i>p,p'</i> -DDT              | Agilent      | Individual   | pp-180-1   | 100                     |
| $\alpha$ -HCH                 | Agilent      | Individual   | pp-110     | 100                     |
| $\beta$ -HCH                  | Agilent      | Individual   | EPA-1077   | 1,000                   |
| $\delta$ -HCH                 | Agilent      | Individual   | EPA-1078   | 100                     |
| $\epsilon$ -HCH               | Chem Service | Individual   | ps-2222    | 100                     |
| $\gamma$ -HCH                 | Agilent      | Individual   | pp-140     | 100                     |
| Aldrin                        | Agilent      | Individual   | pp-100     | 100                     |
| Endrin                        | AccuStandard | Individual   | pp-230-1   | 100                     |
| Dieldrin                      | Agilent      | Individual   | pp-190     | 100                     |
| Endosulfan I                  | Agilent      | Individual   | pp-200     | 100                     |
| Endosulfan II                 | Agilent      | Individual   | pp-210     | 100                     |
| Endosulfan sulfate            | Agilent      | Individual   | pp-220     | 100                     |
| Heptachlor Epoxide (isomer B) | Agilent      | Individual   | EPA-1164   | 1,000                   |
| Methoxychlor                  | AccuStandard | Individual   | app-9-127  | 100                     |
| Octachlorostyrene (OCS)       | Chem Service | Individual   | S-12710X1  | 100                     |
| Hexachlorobenzene (HCB)       | Agilent      | Individual   | ch-151-1   | 100                     |

**Table 23. Table of pesticides and their concentrations in US112B custom stock mixture.**

| Pesticide              | Conc., $\mu\text{g/mL}$ |
|------------------------|-------------------------|
| Aldrin                 | 1,000                   |
| $\alpha$ -HCH          | 1,000                   |
| $\beta$ -HCH           | 1,000                   |
| $\gamma$ -HCH          | 1,000                   |
| $\delta$ -HCH          | 1,000                   |
| <i>p,p'</i> -DDD       | 1,000                   |
| <i>p,p'</i> -DDE       | 1,000                   |
| <i>p,p'</i> -DDT       | 1,000                   |
| Dieldrin               | 1,000                   |
| Endosulfan I           | 1,000                   |
| Endosulfan II          | 1,000                   |
| Endosulfan Sulfate     | 1,000                   |
| Endrin                 | 1,000                   |
| Endrin Aldehyde        | 1,000                   |
| Heptachlor             | 1,000                   |
| Heptachlor Epoxide (B) | 1,000                   |
| Methoxychlor           | 1,000                   |

**Table 24. Table of pesticides and their concentrations in S-8206A custom stock mixture.**

| Pesticide               | Conc., $\mu\text{g/mL}$ |
|-------------------------|-------------------------|
| Hexachlorobenzene       | 100                     |
| $\alpha$ -HCH           | 100                     |
| $\beta$ -HCH            | 100                     |
| $\gamma$ -HCH           | 100                     |
| Heptachlor Epoxide (B)  | 100                     |
| $\alpha$ -Chlordane     | 100                     |
| $\gamma$ -Chlordane     | 100                     |
| <i>trans</i> -Nonachlor | 100                     |
| Methoxychlor            | 100                     |
| Endosulfan I            | 100                     |
| Endosulfan II           | 100                     |
| Endosulfan sulfate      | 100                     |
| <i>p,p'</i> -DDD        | 100                     |
| <i>p,p'</i> -DDE        | 100                     |
| <i>p,p'</i> -DDT        | 100                     |
| <i>o,p'</i> -DDT        | 100                     |
| Aldrin                  | 100                     |
| Endrin                  | 100                     |
| Dieldrin                | 100                     |

**Pesticide Individual Stock Solutions**

**Instructions:** Dilute with hexane indicated aliquot volumes of each custom and individual pesticide stock standard as shown in Table 25. Each solution is prepared separately.

**Table 25: Table of pesticide stock solutions.**

| Stock               | Stock conc., $\mu\text{g/mL}$ | Aliquot, mL | Final Vol., mL | Final conc., ng/mL |
|---------------------|-------------------------------|-------------|----------------|--------------------|
| US112B              | 1,000                         | 0.1         | 50             | 2,000              |
| $\alpha$ -Chlordane | 100                           | 1.0         | 100            | 1,000              |
| $\gamma$ -Chlordane | 100                           | 1.0         | 100            | 1,000              |
| t-Nona              | 100                           | 1.0         | 100            | 1,000              |
| Oxychlordane        | 100                           | 1.0         | 100            | 1,000              |
| DBC                 | 2,000                         | 0.1         | 100            | 2,000              |
| <i>o,p'</i> -DDD    | 100                           | 1.0         | 100            | 1,000              |
| <i>o,p'</i> -DDT    | 100                           | 1.0         | 100            | 1,000              |
| <i>p,p'</i> -DDD    | 100                           | 1.0         | 50             | 2,000              |
| <i>p,p'</i> -DDE    | 100                           | 1.0         | 50             | 2,000              |
| <i>p,p'</i> -DDT    | 100                           | 1.0         | 100            | 1,000              |
| $\alpha$ -HCH       | 100                           | 1.0         | 50             | 2,000              |
| $\beta$ -HCH        | 1,000                         | 1.0         | 100            | 10,000             |
| $\beta$ -HCH        | 10                            | 1.0         | 50             | 200                |
| $\delta$ -HCH       | 100                           | 1.0         | 50             | 2,000              |
| $\epsilon$ -HCH     | 100                           | 1.0         | 50             | 2,000              |
| $\gamma$ -HCH       | 100                           | 1.0         | 50             | 2,000              |
| Aldrin              | 100                           | 1.0         | 100            | 1,000              |
| Endrin              | 100                           | 1.0         | 50             | 2,000              |

|                        |       |     |     |        |
|------------------------|-------|-----|-----|--------|
| Dieldrin               | 100   | 1.0 | 50  | 2,000  |
| Endosulfan I           | 100   | 1.0 | 100 | 1,000  |
| Endosulfan II          | 100   | 1.0 | 100 | 1,000  |
| Endosulfan sulfate     | 100   | 1.0 | 100 | 1,000  |
| Heptachlor Epoxide (B) | 1,000 | 1.0 | 100 | 10,000 |
| Methoxychlor           | 100   | 1.0 | 50  | 2,000  |
| OCS                    | 100   | 2.0 | 100 | 2,000  |
| HCB                    | 100   | 1.0 | 100 | 1,000  |

### **Pesticide Internal Standard (IS)**

#### ***Working mixture***

**Instructions:** Mix aliquot volumes of PCB-65 and -155 stock standards (Table 6) as shown in Table 26 and dilute to 100 mL with hexane.

**Table 26. Table of working Pesticide Internal Standard mixture.**

| Compound | Stock conc.,<br>ng/mL | Aliquot, mL | Final Vol.,<br>mL | Final conc.,<br>ng/mL |
|----------|-----------------------|-------------|-------------------|-----------------------|
| PCB-65   | 2,000                 | 10          | 100               | 200                   |
| PCB-155  | 2,000                 | 10          | 100               | 200                   |

### **Pesticide Surrogate Recovery Standard (SS)**

**Instructions:** See Table 8 in the PCB section.

### **Pesticide Common Calibration Standard**

**Instructions:** Mix aliquot volumes of stock pesticide mixture US112B (Table 23), selected individual pesticides (Table 25), and selected individual PCB standards (Table 6) together as shown in Table 27 and dilute to 100 mL with hexane.

**Table 27. Table of Pesticide Common Calibration Standard.**

| Stock         | Compound         | Stock conc.,<br>ng/mL | Aliquot, mL | Final Vol.,<br>mL | Final conc.,<br>ng/mL |
|---------------|------------------|-----------------------|-------------|-------------------|-----------------------|
| <i>US112B</i> |                  |                       | <i>1.0</i>  | <i>100</i>        |                       |
|               | Aldrin           | 2,000                 | 1.0         | 100               | 20                    |
|               | $\alpha$ -HCH    | 2,000                 | 1.0         | 100               | 20                    |
|               | $\beta$ -HCH     | 2,000                 | 1.0         | 100               | 20                    |
|               | $\gamma$ -HCH    | 2,000                 | 1.0         | 100               | 20                    |
|               | $\delta$ -HCH    | 2,000                 | 1.0         | 100               | 20                    |
|               | <i>p,p'</i> -DDD | 2,000                 | 1.0         | 100               | 20                    |
|               | <i>p,p'</i> -DDE | 2,000                 | 1.0         | 100               | 20                    |
|               | <i>p,p'</i> -DDT | 2,000                 | 1.0         | 100               | 20                    |
|               | Dieldrin         | 2,000                 | 1.0         | 100               | 20                    |
|               | Endosulfan I     | 2,000                 | 1.0         | 100               | 20                    |

|                             |                     |       |     |     |    |
|-----------------------------|---------------------|-------|-----|-----|----|
|                             | Endosulfan II       | 2,000 | 1.0 | 100 | 20 |
|                             | Endosulfan Sulfate  | 2,000 | 1.0 | 100 | 20 |
|                             | Endrin              | 2,000 | 1.0 | 100 | 20 |
|                             | Endrin Aldehyde     | 2,000 | 1.0 | 100 | 20 |
|                             | Heptachlor          | 2,000 | 1.0 | 100 | 20 |
|                             | Heptachlor Epoxide  | 2,000 | 1.0 | 100 | 20 |
|                             | Methoxychlor        | 2,000 | 1.0 | 100 | 20 |
| <b>Individual Standards</b> |                     |       |     |     |    |
|                             | $\alpha$ -chlordane | 1,000 | 2.0 | 100 | 20 |
|                             | $\gamma$ -chlordane | 1,000 | 2.0 | 100 | 20 |
|                             | $\iota$ -Nona       | 1,000 | 2.0 | 100 | 20 |
|                             | <u>Oxychlordane</u> | 1,000 | 2.0 | 100 | 20 |
|                             | DBC                 | 2,000 | 1.0 | 100 | 20 |
|                             | PCB-65              | 2,000 | 1.0 | 100 | 20 |
|                             | PCB-155             | 2,000 | 1.0 | 100 | 20 |
|                             | $o,p'$ -DDD         | 1,000 | 2.0 | 100 | 20 |
|                             | $o,p'$ -DDT         | 1,000 | 2.0 | 100 | 20 |
|                             | HCB                 | 1,000 | 2.0 | 100 | 20 |
|                             | $\epsilon$ -HCH     | 2,000 | 1.0 | 100 | 20 |

**Table 28. Concentrations of individual compounds in the Pesticide Common Calibration Standard mixture.**

| <b>Compound</b>     | <b>Conc., ng/mL</b> |
|---------------------|---------------------|
| $\alpha$ -HCH       | 20                  |
| $\beta$ -HCH        | 20                  |
| $\gamma$ -HCH       | 20                  |
| $\delta$ -HCH       | 20                  |
| $\epsilon$ -HCH     | 20                  |
| Heptachlor          | 20                  |
| Heptachlor Epoxide  | 20                  |
| Aldrin              | 20                  |
| Dieldrin            | 20                  |
| Endrin              | 20                  |
| $p,p'$ -DDT         | 20                  |
| $p,p'$ -DDD         | 20                  |
| $p,p'$ -DDE         | 20                  |
| $o,p'$ -DDD         | 20                  |
| $o,p'$ -DDT         | 20                  |
| $\alpha$ -Chlordane | 20                  |
| $\gamma$ -Chlordane | 20                  |
| <u>Oxychlordane</u> | 20                  |
| $\iota$ -Nona       | 20                  |
| Methoxychlor        | 20                  |
| Endosulfan I        | 20                  |
| Endosulfan II       | 20                  |
| Endosulfan sulfate  | 20                  |
| HCB                 | 20                  |
| DBC                 | 20                  |
| PCB-155             | 20                  |
| PCB-65              | 20                  |

## Pesticide Common Reference Standard

### *Working Mixture*

**Instructions:** Mix indicated aliquot of custom pesticide mixture S-8206A (Table 24) and selected PCB standards (Table 6) together as shown in Table 29 and dilute to 100 mL with hexane.

**Table 29. Table of Pesticide Common Reference Standard mixture.**

| Stock                       | Compound           | Stock conc.,<br>µg/mL | Aliquot, mL  | Final Vol.,<br>mL | Final conc.,<br>ng/mL |
|-----------------------------|--------------------|-----------------------|--------------|-------------------|-----------------------|
| <i>S-8206A</i>              |                    |                       | <i>0.025</i> | <i>100</i>        |                       |
|                             | HCB                | 100                   | 0.025        | 100               | 25                    |
|                             | α-HCH              | 100                   | 0.025        | 100               | 25                    |
|                             | β-HCH              | 100                   | 0.025        | 100               | 25                    |
|                             | γ-HCH              | 100                   | 0.025        | 100               | 25                    |
|                             | Heptachlor Epoxide | 100                   | 0.025        | 100               | 25                    |
|                             | α-Chlordane        | 100                   | 0.025        | 100               | 25                    |
|                             | γ-Chlordane        | 100                   | 0.025        | 100               | 25                    |
|                             | t-Nona             | 100                   | 0.025        | 100               | 25                    |
|                             | Methoxychlor       | 100                   | 0.025        | 100               | 25                    |
|                             | Endosulfan I       | 100                   | 0.025        | 100               | 25                    |
|                             | Endosulfan II      | 100                   | 0.025        | 100               | 25                    |
|                             | Endosulfan sulfate | 100                   | 0.025        | 100               | 25                    |
|                             | <i>p,p'</i> -DDD   | 100                   | 0.025        | 100               | 25                    |
|                             | <i>p,p'</i> -DDE   | 100                   | 0.025        | 100               | 25                    |
|                             | <i>p,p'</i> -DDT   | 100                   | 0.025        | 100               | 25                    |
|                             | <i>o,p'</i> -DDT   | 100                   | 0.025        | 100               | 25                    |
|                             | Aldrin             | 100                   | 0.025        | 100               | 25                    |
|                             | Endrin             | 100                   | 0.025        | 100               | 25                    |
|                             | Dieldrin           | 100                   | 0.025        | 100               | 25                    |
| <i>Individual Standards</i> |                    |                       |              |                   |                       |
|                             | PCB-65             | 2.0                   | 1.0          | 100               | 20                    |
|                             | PCB-155            | 2.0                   | 1.0          | 100               | 20                    |
|                             | DBC                | 2.0                   | 1.0          | 100               | 20                    |
|                             | ε-HCH              | 2.0                   | 1.0          | 100               | 20                    |
|                             | δ-HCH              | 2.0                   | 1.0          | 100               | 20                    |

**Table 30. Table of pesticides and their concentrations in the Pesticide Common Reference Standard mixture.**

| Compound           | Conc.,<br>ng/mL |
|--------------------|-----------------|
| HCB                | 25              |
| α-HCH              | 25              |
| β-HCH              | 25              |
| γ-HCH              | 25              |
| heptachlor Epoxide | 25              |
| α-Chlordane        | 25              |
| γ-Chlordane        | 25              |
| t-Nona             | 25              |
| Methoxychlor       | 25              |
| Endosulfan I       | 25              |
| Endosulfan II      | 25              |

|                    |    |
|--------------------|----|
| Endosulfan sulfate | 25 |
| <i>p,p'</i> -DDD   | 25 |
| <i>p,p'</i> -DDE   | 25 |
| <i>p,p'</i> -DDT   | 25 |
| <i>o,p'</i> -DDT   | 25 |
| Aldrin             | 25 |
| Endrin             | 25 |
| Dieldrin           | 25 |
| PCB-65             | 20 |
| PCB-155            | 20 |
| DBC                | 20 |
| $\epsilon$ -HCH    | 20 |
| $\delta$ -HCH      | 20 |

### **Pesticide Matrix Spike Recovery Standard**

**Instructions:** Mix aliquot volumes of the custom pesticide mixture S-8206A (Table 24) and select individual pesticide compounds (Table 25) as shown in Table 31 and dilute to 100 mL with hexane.

**Table 31. Table of Pesticide Matrix Spike Recovery Standard mixture.**

| Stock                              | Compound            | Stock conc.,<br>$\mu\text{g/mL}$ | Aliquot, mL | Final Vol.,<br>mL | Final conc.,<br>ng/mL |
|------------------------------------|---------------------|----------------------------------|-------------|-------------------|-----------------------|
| <b><i>S-8206A</i></b>              |                     |                                  |             |                   |                       |
|                                    | HCB                 | 100                              | 0.1         | 100               | 100                   |
|                                    | $\alpha$ -HCH       | 100                              | 0.1         | 100               | 100                   |
|                                    | $\beta$ -HCH        | 100                              | 0.1         | 100               | 100                   |
|                                    | $\gamma$ -HCH       | 100                              | 0.1         | 100               | 100                   |
|                                    | Heptachlor Epoxide  | 100                              | 0.1         | 100               | 100                   |
|                                    | $\alpha$ -Chlordane | 100                              | 0.1         | 100               | 100                   |
|                                    | $\gamma$ -Chlordane | 100                              | 0.1         | 100               | 100                   |
|                                    | t-Nona              | 100                              | 0.1         | 100               | 100                   |
|                                    | Methoxychlor        | 100                              | 0.1         | 100               | 100                   |
|                                    | Endosulfan I        | 100                              | 0.1         | 100               | 100                   |
|                                    | Endosulfan II       | 100                              | 0.1         | 100               | 100                   |
|                                    | Endosulfan sulfate  | 100                              | 0.1         | 100               | 100                   |
|                                    | <i>p,p'</i> -DDD    | 100                              | 0.1         | 100               | 100                   |
|                                    | <i>p,p'</i> -DDE    | 100                              | 0.1         | 100               | 100                   |
|                                    | <i>p,p'</i> -DDT    | 100                              | 0.1         | 100               | 100                   |
|                                    | <i>o,p'</i> -DDT    | 100                              | 0.1         | 100               | 100                   |
|                                    | Aldrin              | 100                              | 0.1         | 100               | 100                   |
|                                    | Endrin              | 100                              | 0.1         | 100               | 100                   |
|                                    | Dieldrin            | 100                              | 0.1         | 100               | 100                   |
| <b><i>Individual Standards</i></b> |                     |                                  |             |                   |                       |
|                                    | OCS                 | 2.0                              | 5.0         | 100               | 100                   |
|                                    | Oxychlordane        | 1.0                              | 10.0        | 100               | 100                   |
|                                    | <i>o,p'</i> -DDD    | 1.0                              | 10.0        | 100               | 100                   |



**Table 32. Table of pesticides and their concentrations in the Pesticide Matrix Spike Recovery Standard mixture.**

| Compound                | Conc.,<br>ng/mL |
|-------------------------|-----------------|
| Hexachlorobenzene       | 100             |
| $\alpha$ -HCH           | 100             |
| $\beta$ -HCH            | 100             |
| $\gamma$ -HCH           | 100             |
| heptachlor epoxide      | 100             |
| $\alpha$ -Chlordane     | 100             |
| $\gamma$ -Chlordane     | 100             |
| <i>trans</i> -Nonachlor | 100             |
| Methoxychlor            | 100             |
| Endosulfan I            | 100             |
| Endosulfan II           | 100             |
| Endosulfan sulfate      | 100             |
| <i>p,p'</i> -DDD        | 100             |
| <i>p,p'</i> -DDE        | 100             |
| <i>p,p'</i> -DDT        | 100             |
| <i>o,p'</i> -DDT        | 100             |
| Aldrin                  | 100             |
| Endrin                  | 100             |
| Dieldrin                | 100             |
| Octachlorostyrene       | 100             |
| Oxychlordane            | 100             |
| <i>o,p'</i> -DDD        | 100             |

### **Pesticide Linearity Standards**

#### ***Stock Mixtures***

**Instructions:** Two separate standards are used to prepare pesticide linearity solutions.

**Pest Linearity Stock I:** Combine the indicated aliquot volumes of S-8206A custom pest mixture (Table 24) and selected individual pesticide standards (Table 25), then dilute to 50 mL with hexane as shown in Table 33.

**Pest Linearity Stock II:** Combine the indicated aliquot volumes of selected individual pesticide standards (Table 25), then dilute to 25 mL with hexane as shown in Table 34.

**Table 33. Table of pesticide linearity stock solution I.**

| Stock          | Compound            | Stock conc.,<br>$\mu\text{g/mL}$ | Aliquot, mL | Final Vol.,<br>mL | Final conc.,<br>ng/mL |
|----------------|---------------------|----------------------------------|-------------|-------------------|-----------------------|
| <i>S-8206A</i> |                     |                                  | <i>0.05</i> | <i>50</i>         |                       |
|                | HCB                 | 100                              | 0.05        | 50                | 100                   |
|                | $\alpha$ -HCH       | 100                              | 0.05        | 50                | 100                   |
|                | $\beta$ -HCH        | 100                              | 0.05        | 50                | 100                   |
|                | $\gamma$ -HCH       | 100                              | 0.05        | 50                | 100                   |
|                | Heptachlor Epoxide  | 100                              | 0.05        | 50                | 100                   |
|                | $\alpha$ -Chlordane | 100                              | 0.05        | 50                | 100                   |
|                | $\gamma$ -Chlordane | 100                              | 0.05        | 50                | 100                   |

|                             |                    |     |      |    |     |
|-----------------------------|--------------------|-----|------|----|-----|
|                             | t-Nona             | 100 | 0.05 | 50 | 100 |
|                             | Methoxychlor       | 100 | 0.05 | 50 | 100 |
|                             | Endosulfan I       | 100 | 0.05 | 50 | 100 |
|                             | Endosulfan II      | 100 | 0.05 | 50 | 100 |
|                             | Endosulfan sulfate | 100 | 0.05 | 50 | 100 |
|                             | <i>p,p'</i> -DDD   | 100 | 0.05 | 50 | 100 |
|                             | <i>p,p'</i> -DDE   | 100 | 0.05 | 50 | 100 |
|                             | <i>p,p'</i> -DDT   | 100 | 0.05 | 50 | 100 |
|                             | <i>o,p'</i> -DDT   | 100 | 0.05 | 50 | 100 |
|                             | Aldrin             | 100 | 0.05 | 50 | 100 |
|                             | Endrin             | 100 | 0.05 | 50 | 100 |
|                             | Dieldrin           | 100 | 0.05 | 50 | 100 |
| <i>Individual Standards</i> |                    |     |      |    |     |
|                             | Oxychlorane        | 1.0 | 5.0  | 50 | 100 |
|                             | <i>o,p'</i> -DDD   | 1.0 | 5.0  | 50 | 100 |

**Table 34. Table of pesticide linearity stock solution II.**

| Compound     | Stock conc., $\mu\text{g}/\text{mL}$ | Aliquot, mL | Final Vol., mL | Final conc., ng/mL |
|--------------|--------------------------------------|-------------|----------------|--------------------|
| Endosulfan I | 1.0                                  | 6.25        | 25             | 250                |
| Dieldrin     | 2.0                                  | 3.125       | 25             | 250                |
| HCB          | 1.0                                  | 6.25        | 25             | 250                |
| OCS          | 0.2                                  | 3.75        | 25             | 30                 |

### Working Mixtures

**Instructions:** Perform serial dilutions of Pesticide Linearity Stock Solutions I & II (Tables 35 & 36) with hexane as shown in Tables 35 & 36. Each dilution is done separately.

**Table 35. Table of Pesticide Linearity I working standards.**

| Level | Stock                   | Stock conc., ng/mL | Aliquot, mL | Final Vol., mL | Final Conc., ng/mL |
|-------|-------------------------|--------------------|-------------|----------------|--------------------|
| 01    | Pest Linearity Stock I  | 100                |             |                | 100                |
| 02    | Pest Linearity Stock I  | 100                | 2.0         | 4.0            | 50                 |
| 03    | Pest Linearity I Lvl 02 | 50                 | 2.0         | 4.0            | 25                 |
| 04    | Pest Linearity I Lvl 03 | 25                 | 1.0         | 2.5            | 10                 |
| 05    | Pest Linearity I Lvl 04 | 10                 | 0.5         | 2.5            | 2                  |
| 06    | Pest Linearity I Lvl 05 | 2                  | 0.5         | 2.5            | 0.4                |
| 07    | Pest Linearity I Lvl 06 | 0.4                | 0.5         | 2              | 0.1                |
| 08    | Pest Linearity I Lvl 07 | 0.1                | 1           | 2              | 0.05               |
| 09    | Pest Linearity I Lvl 08 | 0.05               | 0.5         | 2              | 0.01               |

**Table 36. Table of Pesticide Linearity II working standards.**

| Level | Stock                   | Stock conc., ng/mL | Aliquot, mL | Final Vol., mL | Final Conc., ng/mL |
|-------|-------------------------|--------------------|-------------|----------------|--------------------|
| 01    | Pest Linearity Stock II | 250 (30)           |             |                | 250 (30)           |
| 02    | Pest Linearity Stock II | 250 (30)           | 1.2         | 3.0            | 100 (12)           |

|    |                             |              |     |     |              |
|----|-----------------------------|--------------|-----|-----|--------------|
| 03 | Pest Linearity II Lvl<br>02 | 100 (12)     | 1.0 | 2.0 | 50 (6)       |
| 04 | Pest Linearity II Lvl<br>03 | 50 (6)       | 1.0 | 2.0 | 25 (3)       |
| 05 | Pest Linearity II Lvl<br>04 | 25 (3)       | 1.0 | 2.5 | 10 (1.2)     |
| 06 | Pest Linearity II Lvl<br>05 | 10 (1.2)     | 0.5 | 2.5 | 2 (0.24)     |
| 07 | Pest Linearity II Lvl<br>06 | 2 (0.24)     | 0.5 | 2.5 | 0.4 (0.048)  |
| 08 | Pest Linearity II Lvl<br>07 | 0.4 (0.048)  | 0.5 | 2.0 | 0.1 (0.012)  |
| 09 | Pest Linearity II Lvl<br>08 | 0.1 (0.012)  | 1.0 | 2.0 | 0.05 (0.006) |
| 10 | Pest Linearity II Lvl<br>09 | 0.05 (0.006) | 0.5 | 2.0 | 0.01 (0.001) |

**Table 37. Concentration of pesticide compounds in the Pesticide Linearity I working mixtures.**

| PCB                 | Concentration (ng/mL) per Linearity Level |    |    |    |     |     |     |      |      |
|---------------------|---|----|----|----|-----|-----|-----|------|------|
|                     | 01  | 02 | 03 | 04 | 05  | 06  | 07  | 08   | 09   |
| HCb                 | 100                                       | 50 | 25 | 10 | 2.0 | 0.4 | 0.1 | 0.05 | 0.01 |
| $\alpha$ -HCH       | 100                                       | 50 | 25 | 10 | 2.0 | 0.4 | 0.1 | 0.05 | 0.01 |
| $\beta$ -HCH        | 100                                       | 50 | 25 | 10 | 2.0 | 0.4 | 0.1 | 0.05 | 0.01 |
| $\gamma$ -HCH       | 100                                       | 50 | 25 | 10 | 2.0 | 0.4 | 0.1 | 0.05 | 0.01 |
| Heptachlor Epoxide  | 100                                       | 50 | 25 | 10 | 2.0 | 0.4 | 0.1 | 0.05 | 0.01 |
| $\alpha$ -Chlordane | 100                                       | 50 | 25 | 10 | 2.0 | 0.4 | 0.1 | 0.05 | 0.01 |
| $\gamma$ -Chlordane | 100                                       | 50 | 25 | 10 | 2.0 | 0.4 | 0.1 | 0.05 | 0.01 |
| t-Nona              | 100                                       | 50 | 25 | 10 | 2.0 | 0.4 | 0.1 | 0.05 | 0.01 |
| Methoxychlor        | 100                                       | 50 | 25 | 10 | 2.0 | 0.4 | 0.1 | 0.05 | 0.01 |
| Endosulfan I        | 100                                       | 50 | 25 | 10 | 2.0 | 0.4 | 0.1 | 0.05 | 0.01 |
| Endosulfan II       | 100                                       | 50 | 25 | 10 | 2.0 | 0.4 | 0.1 | 0.05 | 0.01 |
| Endosulfan sulfate  | 100                                       | 50 | 25 | 10 | 2.0 | 0.4 | 0.1 | 0.05 | 0.01 |
| <i>p,p'</i> -DDD    | 100                                       | 50 | 25 | 10 | 2.0 | 0.4 | 0.1 | 0.05 | 0.01 |
| <i>p,p'</i> -DDE    | 100                                       | 50 | 25 | 10 | 2.0 | 0.4 | 0.1 | 0.05 | 0.01 |
| <i>p,p'</i> -DDT    | 100                                       | 50 | 25 | 10 | 2.0 | 0.4 | 0.1 | 0.05 | 0.01 |
| <i>o,p'</i> -DDT    | 100                                       | 50 | 25 | 10 | 2.0 | 0.4 | 0.1 | 0.05 | 0.01 |
| Aldrin              | 100                                       | 50 | 25 | 10 | 2.0 | 0.4 | 0.1 | 0.05 | 0.01 |
| Endrin              | 100                                       | 50 | 25 | 10 | 2.0 | 0.4 | 0.1 | 0.05 | 0.01 |
| Dieldrin            | 100                                       | 50 | 25 | 10 | 2.0 | 0.4 | 0.1 | 0.05 | 0.01 |
| Oxychlordane        | 100                                       | 50 | 25 | 10 | 2.0 | 0.4 | 0.1 | 0.05 | 0.01 |
| <i>o,p'</i> -DDD    | 100                                       | 50 | 25 | 10 | 2.0 | 0.4 | 0.1 | 0.05 | 0.01 |

**Table 38. Concentration of pesticide compounds in the Pesticide Linearity II working mixtures.**

| PCB          | Concentration (ng/mL) per Linearity Level |     |    |    |     |      |       |       |       |       |
|--------------|---|-----|----|----|-----|------|-------|-------|-------|-------|
|              | 01  | 02  | 03 | 04 | 05  | 06   | 07    | 08    | 09    | 10    |
| Endosulfan I | 250                                       | 100 | 50 | 25 | 10  | 2.0  | 0.4   | 0.1   | 0.025 | 0.006 |
| Dieldrin     | 250                                       | 100 | 50 | 25 | 10  | 2.0  | 0.4   | 0.1   | 0.025 | 0.006 |
| HCb          | 250                                       | 100 | 50 | 25 | 10  | 2.0  | 0.4   | 0.1   | 0.025 | 0.006 |
| OCS          | 30  | 12  | 6  | 3  | 1.2 | 0.24 | 0.048 | 0.012 | 0.003 | 0.001 |

## **Pesticide IDL Standard**

### ***Stock Mixtures***

**Instructions:** Two separate standards are used to prepare pesticide IDL solutions.

**Stock IDL Standard I:** Mix the indicated aliquot volumes of selected pesticides stock solutions (Table 25) as shown in Table 39 and dilute to 50 mL in hexane.

Stock IDL Standard II: Mix the indicated aliquot volumes of selected pesticides stock solutions (Table 25) as shown in Table 40 and dilute to 50 mL in hexane.

**Table 39. Table of Pesticide Stock IDL Standard I mixture.**

| Stock               | Stock conc.,<br>ng/mL | Aliquot,<br>mL | Final Vol.,<br>mL | Final conc.,<br>ng/mL |
|---------------------|-----------------------|----------------|-------------------|-----------------------|
| $\alpha$ -HCH       | 2,000                 | 2.5            | 50                | 100                   |
| $\beta$ -HCH        | 2,000                 | 2.5            | 50                | 100                   |
| $\alpha$ -Chlordane | 1,000                 | 5.0            | 50                | 100                   |
| $\gamma$ -Chlordane | 1,000                 | 5.0            | 50                | 100                   |
| t-Nona              | 1,000                 | 5.0            | 50                | 100                   |
| Dieldrin            | 2,000                 | 2.5            | 50                | 100                   |
| <i>p,p'</i> -DDD    | 2,000                 | 2.5            | 50                | 100                   |
| <i>p,p'</i> -DDE    | 2,000                 | 2.5            | 50                | 100                   |
| <i>p,p'</i> -DDT    | 1,000                 | 5.0            | 50                | 100                   |
| HCB                 | 1,000                 | 5.0            | 50                | 100                   |

**Table 40. Table of Pesticide Stock IDL Standard II mixture.**

| Stock              | Stock conc.,<br>ng/mL | Aliquot,<br>mL | Final Vol.,<br>mL | Final conc.,<br>ng/mL |
|--------------------|-----------------------|----------------|-------------------|-----------------------|
| Endosulfan I       | 1,000                 | 5.0            | 50                | 100                   |
| Endosulfan II      | 1,000                 | 5.0            | 50                | 100                   |
| Endosulfan sulfate | 1,000                 | 5.0            | 50                | 100                   |
| Heptachlor Epoxide | 10,000                | 0.5            | 50                | 100                   |
| $\beta$ -HCH       | 10,000                | 0.5            | 50                | 100                   |
| Aldrin             | 1,000                 | 5.0            | 50                | 100                   |
| Oxychlordane       | 1,000                 | 5.0            | 50                | 100                   |
| Endrin             | 2,000                 | 2.5            | 50                | 100                   |
| <i>o,p'</i> -DDD   | 1,000                 | 5.0            | 50                | 100                   |
| <i>o,p'</i> -DDT   | 1,000                 | 5.0            | 50                | 100                   |
| OCS                | 2,000                 | 2.5            | 50                | 100                   |
| Methoxychlor       | 2,000                 | 2.5            | 50                | 100                   |

### Working Mixture

**Instructions:** Mix the indicated aliquot volumes of Pesticide Stock IDL Standards I & II (Tables 39 and 40) and PCB-155 and -65 standards (Table 6) as shown in Table 41 and dilute to 50 mL in hexane.

**Table 41. Table of the Pesticide IDL working standard mixture.**

| Stock             | Stock conc.,<br>ng/mL | Aliquot, $\mu$ L | Final Vol.,<br>mL | Final conc.,<br>ng/mL |
|-------------------|-----------------------|------------------|-------------------|-----------------------|
| Pest Stock IDL I  | 100                   | 250              | 50                | 0.50                  |
| Pest Stock IDL II | 100 (105 OCS)         | 250              | 50                | 0.50 (0.525 OSC)      |

|         |       |    |    |   |
|---------|-------|----|----|---|
| PCB-155 | 2,000 | 25 | 50 | 1 |
| PCB-65  | 2,000 | 25 | 50 | 1 |

**Table 42. Levels of individual Pesticide compounds in the Pest IDL working standard.**

| Compound            | Conc., ng/mL |
|---------------------|--------------|
| $\alpha$ -HCH       | 0.5          |
| $\beta$ -HCH        | 0.5          |
| $\alpha$ -Chlordane | 0.5          |
| $\gamma$ -Chlordane | 0.5          |
| t-Nona              | 0.5          |
| Dieldrin            | 0.5          |
| <i>p,p'</i> -DDD    | 0.5          |
| <i>p,p'</i> -DDE    | 0.5          |
| <i>p,p'</i> -DDT    | 0.5          |
| HCB                 | 0.5          |
| Endosulfan I        | 0.5          |
| Endosulfan II       | 0.5          |
| Endosulfan sulfate  | 0.5          |
| Heptachlor Epoxide  | 0.5          |
| $\beta$ -HCH        | 0.5          |
| Aldrin              | 0.5          |
| Oxychlordane        | 0.5          |
| Endrin              | 0.5          |
| <i>o,p'</i> -DDD    | 0.5          |
| <i>o,p'</i> -DDT    | 0.5          |
| OCS                 | 0.525        |
| Methoxychlor        | 0.5          |
| PCB-155             | 1.0          |
| PCB-65              | 1.0          |

### PAH Standards

PAH Custom and individual stock standards

**Table 43. PAH Custom and individual stock standards.**

| Stock                        | Vendor       | Mixture Type        | Catalog ID     | Conc., $\mu$ g/mL |
|------------------------------|--------------|---------------------|----------------|-------------------|
| S-8206B                      | AccuStandard | Mixture             | S-8206B        | 100               |
| PAH Mixture                  | Agilent      | Mixture             | PM-611-1       | 100               |
| Retene                       | AccuStandard | Individual Compound | H-250S         | 50                |
| Retene                       | Chem Service | Individual Compound | 13178U1-1 mL   | 100               |
| Coronene                     | Chem Service | Individual Compound | S-11505U1-1 mL | 100               |
| Benzo[e]pyrene               | Chem Service | Individual Compound | S-11166U1-1 mL | 100               |
| $d_{12}$ -Perylene           | Agilent      | Individual Compound | ATS-150-1      | 2,000             |
| $d_{12}$ - Benz[a]anthracene | Agilent      | Individual Compound | IST-120-1      | 1,000             |
| $d_{10}$ -Anthracene         | Agilent      | Individual Compound | IST-110-1      | 1,000             |

**Table 44. Table of PAHs and their concentrations in S-8206B and PAH Mixture custom stock solutions.**

| S-8206B      | PAH Mixture  | Conc., $\mu$ g/mL |
|--------------|--------------|-------------------|
| Fluorene     | Naphthalene  | 100               |
| Phenanthrene | Acenaphthene | 100               |

|                        |                        |     |
|------------------------|------------------------|-----|
| Anthracene             | Acenaphthylene         | 100 |
| Fluoranthene           | Fluorene               | 100 |
| Pyrene                 | Phenanthrene           | 100 |
| Benz[a]anthracene      | Anthracene             | 100 |
| Triphenylene           | Fluoranthene           | 100 |
| Chrysene               | Pyrene                 | 100 |
| Benzo[b]fluoranthene   | Benz[a]anthracene      | 100 |
| Benzo[k]fluoranthene   | Chrysene               | 100 |
| Benzo[e]pyrene         | Benzo[b]fluoranthene   | 100 |
| Benzo[a]pyrene         | Benzo[k]fluoranthene   | 100 |
| Indeno(1,2,3-cd)pyrene | Benzo[a]pyrene         | 100 |
| Dibenz[a,h]anthracene  | Indeno(1,2,3-cd)pyrene | 100 |
| Benzo[g,h,i]perylene   | Dibenz[a,h]anthracene  | 100 |
| Coronene               | Benzo[g,h,i]perylene   | 100 |

### **PAH Internal Standard**

#### *Stock Mixture*

**Instructions:** Mix indicated aliquot volumes of stock d12-Perylene, d12-Benz[a]anthracene, d10-Anthracene standards as shown in Table 45 and dilute to 50 mL in hexane.

**Table 45. Table of PAH Internal Standard Stock mixture.**

| Stock                              | Stock conc.,<br>µg/mL | Aliquot,<br>mL | Final Vol.,<br>mL | Final conc.,<br>µg/mL |
|------------------------------------|-----------------------|----------------|-------------------|-----------------------|
| d <sub>12</sub> -Perylene          | 2,000                 | 0.5            | 50                | 20                    |
| d <sub>12</sub> -Benz[a]anthracene | 1,000                 | 1              | 50                | 20                    |
| d <sub>10</sub> -Anthracene        | 1,000                 | 1              | 50                | 20                    |

#### *Working Mixture*

**Instructions:** Dilute 10 mL of stock PAH Internal Standard solution (Tables 44) in 50 mL of hexane as shown in Table 46.

**Table 46. Table of PAH Internal Standard Working mixture.**

| Stock                              | Compound                           | Stock conc.,<br>µg/mL | Aliquot, mL | Final Vol.,<br>mL | Final conc.,<br>µg/mL |
|------------------------------------|------------------------------------|-----------------------|-------------|-------------------|-----------------------|
| <i>PAH Internal Standard Stock</i> |                                    |                       | <i>10</i>   | <i>50</i>         |                       |
|                                    | d <sub>12</sub> -Perylene          | 20                    | 10          | 50                | 4                     |
|                                    | d <sub>12</sub> -Benz[a]anthracene | 20                    | 10          | 50                | 4                     |
|                                    | d <sub>10</sub> -Anthracene        | 20                    | 10          | 50                | 4                     |

### **PAH Surrogate Recovery Standard (SS)**

#### *Stock Mixture*

**Instructions:** Mix indicated aliquot volumes of stock d10-Phenanthrene and d10-Pyrene as shown in Table 47 and dilute to 50 mL in hexane.

**Table 47. Table of PAH Surrogate Recovery Standard Stock mixture.**

| Stock                                | Stock conc.,<br>µg/mL | Aliquot, mL    | Final Vol.,<br>mL | Final conc.,<br>µg/mL |
|--------------------------------------|-----------------------|----------------|-------------------|-----------------------|
| d <sub>10</sub> -Phenanthrene        | 1,000                 | 1.0            | 50                | 20                    |
| d <sub>10</sub> -Pyrene (2 ampoules) | 500                   | 2.0 (1 mL/amp) | 50                | 20                    |

**Working Mixture**

**Instructions:** Dilute 10 mL of the stock PAH Surrogate Recovery Stock mixture (Table 46) in 50 mL of hexane as shown in Table 48.

**Table 48. Table of PAH Surrogate Recovery Standard working mixture.**

| Stock                               | Compound                      | Stock conc.,<br>µg/mL | Aliquot, mL | Final Vol.,<br>mL | Final conc.,<br>µg/mL |
|-------------------------------------|-------------------------------|-----------------------|-------------|-------------------|-----------------------|
| <i>PAH Surrogate Recovery Stock</i> |                               |                       | <i>10</i>   | <i>50</i>         |                       |
|                                     | d <sub>10</sub> -Phenanthrene | 20                    | 10          | 50                | 4                     |
|                                     | d <sub>10</sub> -Pyrene       | 20                    | 10          | 50                | 4                     |

**PAH Calibration Standard**

**Instructions:** Mix the indicated aliquot volumes of PAH matrix spike (Table 54), PAH IS Stock mix (Table 45), and PAH SS Stock mix (Tables 47) as shown in Table 49 and dilute to 50 mL in hexane.

**Table 49. Table of the PAH Calibration Standard**

| Stock                         | Compound                           | Stock conc.,<br>µg/mL | Aliquot, mL | Final Vol.,<br>mL | Final conc.,<br>ng/mL |
|-------------------------------|------------------------------------|-----------------------|-------------|-------------------|-----------------------|
| <i>PAH Matrix Spike Stock</i> |                                    |                       | <i>5.0</i>  | <i>50</i>         |                       |
|                               | Naphthalene                        | 2.0                   | 5.0         | 50                | 200                   |
|                               | Acenaphthene                       | 2.0                   | 5.0         | 50                | 200                   |
|                               | Acenaphthylene                     | 2.0                   | 5.0         | 50                | 200                   |
|                               | Fluorene                           | 2.0                   | 5.0         | 50                | 200                   |
|                               | Phenanthrene                       | 2.0                   | 5.0         | 50                | 200                   |
|                               | Anthracene                         | 2.0                   | 5.0         | 50                | 200                   |
|                               | Fluoranthene                       | 2.0                   | 5.0         | 50                | 200                   |
|                               | Pyrene                             | 2.0                   | 5.0         | 50                | 200                   |
|                               | Retene                             | 2.0                   | 5.0         | 50                | 200                   |
|                               | Benz[a]anthracene                  | 2.0                   | 5.0         | 50                | 200                   |
|                               | Chrysene                           | 2.0                   | 5.0         | 50                | 200                   |
|                               | Benzo[b]fluoranthene               | 2.0                   | 5.0         | 50                | 200                   |
|                               | Benzo[k]fluoranthene               | 2.0                   | 5.0         | 50                | 200                   |
|                               | Benzo[e]pyrene                     | 2.0                   | 5.0         | 50                | 200                   |
|                               | Benzo[a]pyrene                     | 2.0                   | 5.0         | 50                | 200                   |
|                               | Indeno(1,2,3-cd)pyrene             | 2.0                   | 5.0         | 50                | 200                   |
|                               | Dibenz[a,h]anthracene              | 2.0                   | 5.0         | 50                | 200                   |
|                               | Benzo[g,h,i]perylene               | 2.0                   | 5.0         | 50                | 200                   |
|                               | Coronene                           | 2.0                   | 5.0         | 50                | 200                   |
| <i>PAH IS Stock</i>           |                                    |                       | <i>0.5</i>  | <i>50</i>         |                       |
|                               | d <sub>12</sub> -Perylene          | 20                    | 0.5         | 50                | 200                   |
|                               | d <sub>12</sub> -Benz[a]anthracene | 20                    | 0.5         | 50                | 200                   |

|                     |                              |    |            |           |     |
|---------------------|------------------------------|----|------------|-----------|-----|
|                     | d <sub>10</sub> -Anthracene  | 20 | 0.5        | 50        | 200 |
| <b>PAH SS Stock</b> |                              |    | <b>0.5</b> | <b>50</b> |     |
|                     | d <sub>10</sub> -Phenathrene | 20 | 0.5        | 50        | 200 |
|                     | d <sub>10</sub> -Pyrene      | 20 | 0.5        | 50        | 200 |

### **PAH Common Reference Standard**

#### ***Stock Mixtures***

**Instructions:** Two separate standards are prepared for checking instrument PAH calibration.

**PAH CRS (QAQC) Stock:** Dilute 1.0 mL of stock S-8206B standard to 50 mL in hexane as shown in Table 50.

**Retene CRS Stock:** Dilute 1.0 mL of stock H-250S to 50 mL of hexane as shown in Table 51.

**Table 50. Table of PAH CRS (QAQC) Stock mixture.**

| Stock   | Stock conc.,<br>µg/mL | Aliquot, mL | Final Vol.,<br>mL | Final conc.,<br>µg/mL |
|---------|-----------------------|-------------|-------------------|-----------------------|
| S-8206B | 100                   | 1.0         | 50                | 2.0                   |

**Table 51. Table of Retene CRS Stock mixture.**

| Stock  | Stock conc.,<br>µg/mL | Aliquot, mL | Final Vol.,<br>mL | Final conc.,<br>µg/mL |
|--------|-----------------------|-------------|-------------------|-----------------------|
| H-250S | 50                    | 1.0         | 50                | 1.0                   |

#### ***Working Mixtures***

**Instructions:** Two separate standards are prepared.

**PAH Combined Reference Standard:** Mix the indicated aliquot volumes of PAH Common Reference Standard (QA/QC) (Table 50), Retene CRS Stock (Table 51), PAH IS Stock (Table 45), and PAH SS Stock (Table 47) and dilute to 50 mL in hexane as shown in Table 52.

**Retene CRS:** Mix the indicated aliquot volumes of Retene CRS Stock (Table 51), PAH IS Stock (Table 45), and PAH SS Stock (Table 47) and dilute to 50 mL with hexane as shown in Table 53.

**Table 52. Table of PAH Combined Common Reference Standard.**

| Stock                         | Compound          | Stock conc.,<br>µg/mL | Aliquot, mL | Final Vol.,<br>mL | Final conc.,<br>ng/mL |
|-------------------------------|-------------------|-----------------------|-------------|-------------------|-----------------------|
| <b>PAH CRS QAQC Stock Mix</b> |                   |                       | <b>5.0</b>  | <b>50</b>         |                       |
|                               | Fluorene          | 2.0                   | 5.0         | 50                | 200                   |
|                               | Phenanthrene      | 2.0                   | 5.0         | 50                | 200                   |
|                               | Anthracene        | 2.0                   | 5.0         | 50                | 200                   |
|                               | Fluoranthene      | 2.0                   | 5.0         | 50                | 200                   |
|                               | Pyrene            | 2.0                   | 5.0         | 50                | 200                   |
|                               | Benz[a]anthracene | 2.0                   | 5.0         | 50                | 200                   |
|                               | Triphenylene      | 2.0                   | 5.0         | 50                | 200                   |
|                               | Chrysene          | 2.0                   | 5.0         | 50                | 200                   |



|                         |                                    |     |             |           |     |
|-------------------------|------------------------------------|-----|-------------|-----------|-----|
|                         | Benzo[b]fluoranthene               | 2.0 | 5.0         | 50        | 200 |
|                         | Benzo[k]fluoranthene               | 2.0 | 5.0         | 50        | 200 |
|                         | Benzo[e]pyrene                     | 2.0 | 5.0         | 50        | 200 |
|                         | Benzo[a]pyrene                     | 2.0 | 5.0         | 50        | 200 |
|                         | Indeno(1,2,3-cd)pyrene             | 2.0 | 5.0         | 50        | 200 |
|                         | Dibenz[a,h]anthracene              | 2.0 | 5.0         | 50        | 200 |
|                         | Benzo[g,h,i]perylene               | 2.0 | 5.0         | 50        | 200 |
|                         | Coronene                           | 2.0 | 5.0         | 50        | 200 |
| <b>Retene CRS Stock</b> |                                    |     | <b>10.0</b> | <b>50</b> |     |
|                         | Retene                             | 1.0 | 10.0        | 50        | 200 |
| <b>PAH IS Stock</b>     |                                    |     | <b>0.5</b>  | <b>50</b> |     |
|                         | d <sub>12</sub> -Perylene          | 20  | 0.5         | 50        | 200 |
|                         | d <sub>12</sub> -Benz[a]anthracene | 20  | 0.5         | 50        | 200 |
|                         | d <sub>10</sub> -Anthracene        | 20  | 0.5         | 50        | 200 |
| <b>PAH SS Stock</b>     |                                    |     | <b>0.5</b>  | <b>50</b> |     |
|                         | d <sub>10</sub> -Phenathrene       | 20  | 0.5         | 50        | 200 |
|                         | d <sub>10</sub> -Pyrene            | 20  | 0.5         | 50        | 200 |

Table 53. Table of Retene Common Reference Standard.

| Stock                   | Compound                           | Stock conc.,<br>µg/mL | Aliquot, mL | Final Vol.,<br>mL | Final conc.,<br>ng/mL |
|-------------------------|------------------------------------|-----------------------|-------------|-------------------|-----------------------|
| <b>Retene CRS Stock</b> |                                    |                       | <b>10.0</b> | <b>50</b>         |                       |
|                         | Retene                             | 1.0                   | 10.0        | 50                | 200                   |
| <b>PAH IS Stock</b>     |                                    |                       | <b>0.5</b>  | <b>50</b>         |                       |
|                         | d <sub>12</sub> -Perylene          | 20                    | 0.5         | 50                | 200                   |
|                         | d <sub>12</sub> -Benz[a]anthracene | 20                    | 0.5         | 50                | 200                   |
|                         | d <sub>10</sub> -Anthracene        | 20                    | 0.5         | 50                | 200                   |
| <b>PAH SS Stock</b>     |                                    |                       | <b>0.5</b>  | <b>50</b>         |                       |
|                         | d <sub>10</sub> -Phenathrene       | 20                    | 0.5         | 50                | 200                   |
|                         | d <sub>10</sub> -Pyrene            | 20                    | 0.5         | 50                | 200                   |

## Matrix Spike

### Stock Mixture

**Instructions:** Mix indicated aliquot volumes of stock PAH mixture (Table 44) and selected individual PAHs (Table 43) as shown in Table 54 and dilute to 50 mL in hexane

Table 54. Table of the PAH Matrix Spike stock solution.

| Stock              | Compound       | Stock conc.,<br>µg/mL | Aliquot, mL | Final Vol.,<br>mL | Final conc.,<br>µg/mL |
|--------------------|----------------|-----------------------|-------------|-------------------|-----------------------|
| <b>PAH Mixture</b> |                |                       | <b>1.0</b>  | <b>50</b>         |                       |
|                    | Naphthalene    | 100                   | 1.0         | 50                | 2.0                   |
|                    | Acenaphthene   | 100                   | 1.0         | 50                | 2.0                   |
|                    | Acenaphthylene | 100                   | 1.0         | 50                | 2.0                   |
|                    | Fluorene       | 100                   | 1.0         | 50                | 2.0                   |
|                    | Phenanthrene   | 100                   | 1.0         | 50                | 2.0                   |

|                          |                        |     |             |           |     |
|--------------------------|------------------------|-----|-------------|-----------|-----|
|                          | Anthracene             | 100 | 1.0         | 50        | 2.0 |
|                          | Fluoranthene           | 100 | 1.0         | 50        | 2.0 |
|                          | Pyrene                 | 100 | 1.0         | 50        | 2.0 |
|                          | Benz[a]anthracene      | 100 | 1.0         | 50        | 2.0 |
|                          | Chrysene               | 100 | 1.0         | 50        | 2.0 |
|                          | Benzo[b]fluoranthene   | 100 | 1.0         | 50        | 2.0 |
|                          | Benzo[k]fluoranthene   | 100 | 1.0         | 50        | 2.0 |
|                          | Benzo[a]pyrene         | 100 | 1.0         | 50        | 2.0 |
|                          | Indeno(1,2,3-cd)pyrene | 100 | 1.0         | 50        | 2.0 |
|                          | Dibenz[a,h]anthracene  | 100 | 1.0         | 50        | 2.0 |
|                          | Benzo[g,h,i]perylene   | 100 | 1.0         | 50        | 2.0 |
| <b>Individual Stocks</b> |                        |     | <b>10.0</b> | <b>50</b> |     |
|                          | Retene                 | 100 | 10.0        | 50        | 2.0 |
|                          | Coronene               | 100 | 0.5         | 50        | 2.0 |
|                          | Benzo[e]pyrene         | 100 | 0.5         | 50        | 2.0 |

### PAH Linearity Standards

**Instructions:** Perform dilutions of the PAH Matrix Spike Recovery stock solution (Tables 54) as shown in Table 55. Each dilution is done separately.

**Table 55. Table of PAH Linearity Standards.**

| Level | Stock                  | Stock conc.,<br>ng/mL | Aliquot, mL | Final Vol.,<br>mL | Final Conc.,<br>ng/mL |
|-------|------------------------|-----------------------|-------------|-------------------|-----------------------|
| 01    | PAH Matrix Spike Stock | 2,000                 |             |                   | 2,000                 |
| 02    | PAH Matrix Spike Stock | 2,000                 | 1.0         | 4.0               | 1,000                 |
|       | PAH SS Working         | 4,000                 | 1.0         | 4.0               | 1,000                 |
| 03    | PAH LIN 02             | 1,000                 | 0.5         | 1.0               | 500                   |
| 04    | PAH LIN 02             | 1,000                 | 0.4         | 1.0               | 400                   |
| 05    | PAH LIN 02             | 1,000                 | 0.3         | 1.0               | 300                   |
| 06    | PAH LIN 02             | 1,000                 | 0.2         | 1.0               | 200                   |
| 07    | PAH LIN 02             | 1,000                 | 0.2         | 2.0               | 100                   |
| 08    | PAH LIN 02             | 1,000                 | 0.05        | 1.0               | 50                    |
| 09    | PAH LIN 07             | 100                   | 0.2         | 2.0               | 10                    |
| 10    | PAH LIN 09             | 10                    | 0.1         | 1.0               | 1.0                   |
| 11    | PAH LIN 09             | 10                    | 0.05        | 5.0               | 0.1                   |

**Table 56. Concentrations of PAH compounds from the IADN annual reporting list in the various Linearity levels.**

| Compound         | Concentration per level (ng/mL) |       |     |     |     |     |     |    |    |     |     |
|------------------|---------------------------------|-------|-----|-----|-----|-----|-----|----|----|-----|-----|
|                  | 01                              | 02    | 03  | 04  | 05  | 06  | 07  | 08 | 09 | 10  | 11  |
| Fluorene         | 2,000                           | 1,000 | 500 | 400 | 300 | 200 | 100 | 50 | 10 | 1.0 | 0.1 |
| Phenanthrene     | 2,000                           | 1,000 | 500 | 400 | 300 | 200 | 100 | 50 | 10 | 1.0 | 0.1 |
| d10-Phenanthrene |                                 | 1,000 | 500 | 400 | 300 | 200 | 100 | 50 | 10 | 1.0 | 0.1 |
| Anthracene       | 2,000                           | 1,000 | 500 | 400 | 300 | 200 | 100 | 50 | 10 | 1.0 | 0.1 |

|                        |       |       |     |     |     |     |     |    |    |     |     |
|------------------------|-------|-------|-----|-----|-----|-----|-----|----|----|-----|-----|
| Fluoranthene           | 2,000 | 1,000 | 500 | 400 | 300 | 200 | 100 | 50 | 10 | 1.0 | 0.1 |
| Pyrene                 | 2,000 | 1,000 | 500 | 400 | 300 | 200 | 100 | 50 | 10 | 1.0 | 0.1 |
| d10-Pyrene             |       | 1,000 | 500 | 400 | 300 | 200 | 100 | 50 | 10 | 1.0 | 0.1 |
| Retene                 | 2,000 | 1,000 | 500 | 400 | 300 | 200 | 100 | 50 | 10 | 1.0 | 0.1 |
| Benz[a]anthracene      | 2,000 | 1,000 | 500 | 400 | 300 | 200 | 100 | 50 | 10 | 1.0 | 0.1 |
| Chrysene               | 2,000 | 1,000 | 500 | 400 | 300 | 200 | 100 | 50 | 10 | 1.0 | 0.1 |
| Benzo[b]fluoranthene   | 2,000 | 1,000 | 500 | 400 | 300 | 200 | 100 | 50 | 10 | 1.0 | 0.1 |
| Benzo[k]fluoranthene   | 2,000 | 1,000 | 500 | 400 | 300 | 200 | 100 | 50 | 10 | 1.0 | 0.1 |
| Benzo[e]pyrene         | 2,000 | 1,000 | 500 | 400 | 300 | 200 | 100 | 50 | 10 | 1.0 | 0.1 |
| Benzo[a]pyrene         | 2,000 | 1,000 | 500 | 400 | 300 | 200 | 100 | 50 | 10 | 1.0 | 0.1 |
| Indeno(1,2,3-cd)pyrene | 2,000 | 1,000 | 500 | 400 | 300 | 200 | 100 | 50 | 10 | 1.0 | 0.1 |
| Dibenz[a,h]anthracene  | 2,000 | 1,000 | 500 | 400 | 300 | 200 | 100 | 50 | 10 | 1.0 | 0.1 |
| Benzo[g,h,i]perylene   | 2,000 | 1,000 | 500 | 400 | 300 | 200 | 100 | 50 | 10 | 1.0 | 0.1 |
| Coronene               | 2,000 | 1,000 | 500 | 400 | 300 | 200 | 100 | 50 | 10 | 1.0 | 0.1 |

### PAH IDL Standard

Instructions: Dilute PAH Matrix Spike Stock mixture (Table 54) and PAH IS Working (Table 46) as shown in Table 57.

Table 57. Table of the working PAH IDL Standard.

| Stock                         | Compound                           | Stock conc.,<br>µg/mL | Aliquot, mL | Final Vol.,<br>mL | Final conc.,<br>ng/mL |
|-------------------------------|------------------------------------|-----------------------|-------------|-------------------|-----------------------|
| <i>PAH Matrix Spike Stock</i> |                                    |                       | <b>0.25</b> | <b>100</b>        |                       |
|                               | Naphthalene                        | 2.0                   | 0.25        | 100               | 5.0                   |
|                               | Acenaphthene                       | 2.0                   | 0.25        | 100               | 5.0                   |
|                               | Acenaphthylene                     | 2.0                   | 0.25        | 100               | 5.0                   |
|                               | Fluorene                           | 2.0                   | 0.25        | 100               | 5.0                   |
|                               | Phenanthrene                       | 2.0                   | 0.25        | 100               | 5.0                   |
|                               | Anthracene                         | 2.0                   | 0.25        | 100               | 5.0                   |
|                               | Fluoranthene                       | 2.0                   | 0.25        | 100               | 5.0                   |
|                               | Pyrene                             | 2.0                   | 0.25        | 100               | 5.0                   |
|                               | Retene                             | 2.0                   | 0.25        | 100               | 5.0                   |
|                               | Benz[a]anthracene                  | 2.0                   | 0.25        | 100               | 5.0                   |
|                               | Chrysene                           | 2.0                   | 0.25        | 100               | 5.0                   |
|                               | Benzo[b]fluoranthene               | 2.0                   | 0.25        | 100               | 5.0                   |
|                               | Benzo[k]fluoranthene               | 2.0                   | 0.25        | 100               | 5.0                   |
|                               | Benzo[e]pyrene                     | 2.0                   | 0.25        | 100               | 5.0                   |
|                               | Benzo[a]pyrene                     | 2.0                   | 0.25        | 100               | 5.0                   |
|                               | Indeno(1,2,3-cd)pyrene             | 2.0                   | 0.25        | 100               | 5.0                   |
|                               | Dibenz[a,h]anthracene              | 2.0                   | 0.25        | 100               | 5.0                   |
|                               | Benzo[g,h,i]perylene               | 2.0                   | 0.25        | 100               | 5.0                   |
|                               | Coronene                           | 2.0                   | 0.25        | 100               | 5.0                   |
| <i>PAH IS Working</i>         |                                    |                       | <b>5.0</b>  | <b>100</b>        |                       |
|                               | d <sub>12</sub> -Perylene          | 4.0                   | 5.0         | 100               | 200                   |
|                               | d <sub>12</sub> -Benz[a]anthracene | 4.0                   | 5.0         | 100               | 200                   |
|                               | d <sub>10</sub> -Anthracene        | 4.0                   | 5.0         | 100               | 200                   |

**PBDE Standards**

**Table 58. Table of PBDE/BFR stock mixture and individual standards.**

| Stock                                  | Vendor       | Mixture Type | Solvent   | Catalog ID | Purpose | Conc., µg/mL |
|--|--------------|--------------|-----------|------------|---------|--------------|
| BDE-MXE                                | Wellington   | Mixture      | Toluene   | BDE-MXE    | MS      | 1.0 – 5.0    |
| PBFR-PAH                               | Wellington   | Mixture      | Toluene   | BFR-PAR    | Cal Std | 0.2 – 4.0    |
| S-21223                                | Accustandard | Mixture      | isooctane | S-21223    | CRS1    | 1.5 – 3.7    |
| S-20375                                | Accustandard | Mixture      | isooctane | S-20375    | CRS1    | 3.0 – 5.0    |
| S-20371                                | Accustandard | Mixture      | isooctane | S-20371    | CRS2    | 5.0 – 10.0   |
| anti-DP                                | Wellington   | Individual   | Toluene   | anti-DP    | Cal, MS | 50           |
| syn-DP                                 | Wellington   | Individual   | Toluene   | syn-DP     | Cal, MS | 50           |
| α-HBCD                                 | Accustandard | Individual   | Toluene   | HXBCD-01   | Cal, MS | 100          |
| EHTBB                                  | Wellington   | Individual   | Toluene   | EHTBB      | Cal, MS | 50           |
| BEHTBP                                 | Wellington   | Individual   | Toluene   | BEHTBP     | Cal, MS | 50           |
| pTBX                                   | Wellington   | Individual   | Toluene   | pTBX       | Cal, MS | 50           |
| PBBZ                                   | Wellington   | Individual   | Toluene   | PBBZ       | Cal, MS | 50           |
| BDE-118                                | Accustandard | Individual   | isooctane | BDE-118S   | IS      | 50           |
| BDE-181                                | Wellington   | Individual   | nonane    | BDE-181S   | IS      | 50           |
| BDE-209                                | Wellington   | Individual   |           |            | CRS2    | 50           |
| DBDPE                                  | Wellington   | Individual   |           |            | CRS2    | 25           |
| BDE-77                                 | Accustandard | Individual   | isooctane | BDE-077S   | SS      | 50           |
| BDE-166                                | Accustandard | Individual   | isooctane | BDE-166S   | SS      | 50           |
| <sup>13</sup> C <sub>12</sub> -BDE-209 | Wellington   | Individual   | Toluene   | MBDE-209   | SS      | 25           |

**Table 59. Table of PBDE/BFR compounds and their concentrations in stock mixtures BDE-MXE, BFR-PAH, S-21223, S-20375, and S20371 (µg/mL).**

| BDE-MXE |     | BFR-PAH |           |         |     | S-21223 |           |
|---------|-----|---------|-----------|---------|-----|---------|-----------|
| BDE-3   | 1.0 | BDE-1   | 0.2       | BDE-140 | 0.4 | BDE-1   | 1.50<br>3 |
| BDE-7   | 1.0 | BDE-2   | 0.2       | BDE-153 | 0.4 | BDE-2   | 1.48<br>5 |
| BDE-15  | 1.0 | BDE-3   | 0.2       | BDE-154 | 0.4 | BDE-3   | 1.48<br>7 |
| BDE-17  | 1.0 | BDE-7   | 0.2       | BDE-156 | 0.4 | BDE-7   | 1.49<br>6 |
| BDE-28  | 1.0 | BDE-10  | 0.2       | BDE-169 | 0.4 | BDE-8   | 1.51<br>5 |
| BDE-47  | 1.0 | BDE-15  | 0.2       | BB-153  | 0.4 | BDE-10  | 1.52<br>4 |
| BDE-49  | 1.0 | BDE-17  | 0.19<br>2 | BTBPE   | 0.4 | BDE-11  | 1.48<br>5 |
| BDE-66  | 1.0 | BDE-28  | 0.2       | BDE-171 | 0.8 | BDE-12  | 1.50<br>1 |
| BDE-71  | 1.0 | BDE-30  | 0.2       | BDE-180 | 0.8 | BDE-13  | 1.51<br>4 |
| BDE-77  | 1.0 | HBB     | 0.2       | BDE-183 | 0.8 | BDE-15  | 1.50<br>1 |

|         |     |
|---------|-----|
| BDE-85  | 1.0 |
| BDE-99  | 1.0 |
| BDE-100 | 1.0 |
| BDE-119 | 1.0 |
| BDE-126 | 1.0 |
| BDE-138 | 2.0 |
| BDE-153 | 2.0 |
| BDE-154 | 2.0 |
| BDE-156 | 2.0 |
| BDE-183 | 2.0 |
| BDE-184 | 2.0 |
| BDE-191 | 2.0 |
| BDE-196 | 2.0 |
| BDE-197 | 2.0 |
| BDE-206 | 5.0 |
| BDE-207 | 5.0 |
| BDE-209 | 5.0 |

|         |     |         |     |
|---------|-----|---------|-----|
| PBEB    | 0.2 | BDE-184 | 0.8 |
| BDE-47  | 0.4 | BDE-191 | 0.8 |
| BDE-49  | 0.4 | BDE-196 | 0.8 |
| BDE-66  | 0.4 | BDE-197 | 0.8 |
| BDE-71  | 0.4 | BDE-201 | 0.8 |
| BDE-77  | 0.4 | BDE-203 | 0.8 |
| BDE-85  | 0.4 | BDE-204 | 0.8 |
| BDE-99  | 0.4 | BDE-205 | 0.8 |
| BDE-100 | 0.4 | BDE-206 | 2.0 |
| BDE-119 | 0.4 | BDE-207 | 2.0 |
| BDE-126 | 0.4 | BDE-208 | 2.0 |
| BDE-138 | 0.4 | BDE-209 | 2.0 |
| BDE-139 | 0.4 | DBDPE   | 4.0 |

| S-20375 |       | S-20371 |      |
|---------|-------|---------|------|
| BDE-140 | 3.003 | BDE-209 | 5.0  |
| BDE-197 | 3.940 | DBDPE   | 10.0 |
| BDE-202 | 4.024 |         |      |
| BDE-206 | 4.975 |         |      |

|         |       |
|---------|-------|
| BDE-181 | 3.696 |
| BDE-183 | 3.789 |
| BDE-190 | 3.702 |

|         |       |
|---------|-------|
| BDE-17  | 1.528 |
| BDE-25  | 1.509 |
| BDE-28  | 1.518 |
| BDE-30  | 1.489 |
| BDE-32  | 1.516 |
| BDE-33  | 1.499 |
| BDE-35  | 1.508 |
| BDE-37  | 1.504 |
| BDE-47  | 1.504 |
| BDE-49  | 1.506 |
| BDE-66  | 1.531 |
| BDE-71  | 1.514 |
| BDE-75  | 1.503 |
| BDE-77  | 1.503 |
| BDE-85  | 2.237 |
| BDE-99  | 2.251 |
| BDE-100 | 2.264 |
| BDE-116 | 2.274 |
| BDE-119 | 2.292 |
| BDE-126 | 2.240 |
| BDE-138 | 3.033 |
| BDE-153 | 3.019 |
| BDE-154 | 3.012 |
| BDE-155 | 3.022 |
| BDE-166 | 2.958 |

### Non-BDE Stock Standard

**Instructions:** Mix indicated aliquot volumes of individual standards (Table 58) as shown in Table 60 and dilute to 50 mL in hexane.

**Table 60. Table of Non-BDE Stock Solution**

| Stock            | Stock conc.,<br><u>µg/mL</u> | Aliquot, mL | Final Vol.,<br>mL | Final conc.,<br>ng/mL |
|------------------|------------------------------|-------------|-------------------|-----------------------|
| <i>anti</i> -DP  | 50                           | 0.5         | 50                | 500                   |
| <i>syn</i> -DP   | 50                           | 0.5         | 50                | 500                   |
| $\alpha$ -HBCD   | 100                          | 0.5         | 50                | 1,000                 |
| EHTBB            | 50                           | 0.5         | 50                | 500                   |
| BEHTBP           | 50                           | 0.5         | 50                | 500                   |
| <del>p</del> TBX | 50                           | 0.25        | 50                | 250                   |
| PBBZ             | 50                           | 0.25        | 50                | 250                   |

### PBDE Internal Standard

#### *Stock Mixture*

**Instructions:** Mix indicated aliquot volumes of stock BDE-118 and BDE-181 standards as shown in Table 61 and dilute to 50 mL in hexane.

**Table 61. Table of PBDE Internal Standard Stock mixture.**

| Stock   | Stock conc.,<br><u>µg/mL</u> | Aliquot, mL | Final Vol.,<br>mL | Final conc.,<br>ng/mL |
|---------|------------------------------|-------------|-------------------|-----------------------|
| BDE-118 | 50                           | 0.5         | 50                | 500                   |
| BDE-181 | 50                           | 1.0         | 50                | 1,000                 |

#### *Working Mixture*

**Instructions:** Dilute 10 mL of stock PBDE Internal Standard Stock mixture (Tables 61) to 50 mL with hexane as shown in Table 62.

**Table 62. Table of PBDE Internal Standard Working mixture.**

| Stock                | Compound | Stock conc.,<br>ng/mL | Aliquot, mL | Final Vol.,<br>mL | Final conc.,<br>ng/mL |
|----------------------|----------|-----------------------|-------------|-------------------|-----------------------|
| <i>PBDE IS Stock</i> |          |                       | <i>10</i>   | <i>50</i>         |                       |
|                      | BDE-118  | 500                   | 10          | 50                | 100                   |
|                      | BDE-181  | 1,000                 | 10          | 50                | 200                   |

### PBDE Surrogate Recovery Standard

#### *Stock Mixture*

**Instructions:** Mix indicated aliquot volumes of stock BDE-77, BDE-166 and <sup>13</sup>C<sub>12</sub>-BDE-209 as shown in Table 63 and dilute to 50 mL with hexane.

**Table 63. Table of PBDE Surrogate Recovery Standard Stock mixture.**

| Stock                                  | Stock conc.,<br><u>µg/mL</u> | Aliquot, mL | Final Vol.,<br>mL | Final conc.,<br>ng/mL |
|--|------------------------------|-------------|-------------------|-----------------------|
| BDE-77                                 | 50                           | 0.3         | 50                | 300                   |
| BDE-166                                | 50                           | 0.5         | 50                | 500                   |
| <sup>13</sup> C <sub>12</sub> -BDE-209 | 25                           | 0.8         | 50                | 400                   |

**Working Mixture**

**Instructions:** Dilute 10 mL of the PBDE Surrogate Recovery Standard Stock mixture (Table 63) in 50 mL of hexane as shown in Table 64.

**Table 64. Table of PBDE Surrogate Recovery Standard Working mixture.**

| Stock                | Compound                               | Stock conc.,<br>ng/mL | Aliquot, mL | Final Vol.,<br>mL | Final conc.,<br>ng/mL |
|----------------------|--|-----------------------|-------------|-------------------|-----------------------|
| <i>PBDE SS Stock</i> |  |                       | <i>10</i>   | <i>50</i>         |                       |
|                      | BDE-77                                 | 300                   | 10          | 50                | 60                    |
|                      | BDE-166                                | 500                   | 10          | 50                | 100                   |
|                      | <sup>13</sup> C <sub>12</sub> -BDE-209 | 400                   | 10          | 50                | 80                    |

**PBDE/BFR Calibration Standard**

**Working Mixture**

**Instructions:** Mix the indicated aliquot volumes of Non-BDE stock (Table 60), BFR-PAR (Table 59), PBDE IS Stock (Table 61), and PBDE SS Stock (Tables 63) as shown in Table 65 and dilute to 100 mL with hexane.

**Table 65. Table of working PBDE Calibration Standard mixture.**

| Stock                | Compound        | Stock conc.,<br>ng/mL | Aliquot, mL | Final Vol.,<br>mL | Final conc.,<br>ng/mL |
|----------------------|-----------------|-----------------------|-------------|-------------------|-----------------------|
| <i>Non-BDE Stock</i> |                 |                       | <i>1.0</i>  | <i>100</i>        |                       |
|                      | <i>anti</i> -DP | 500                   | 1.0         | 100               | 5.0                   |
|                      | <i>syn</i> -DP  | 500                   | 1.0         | 100               | 5.0                   |
|                      | <i>α</i> -HBCD  | 1,000                 | 1.0         | 100               | 10.0                  |
|                      | EHTBB           | 500                   | 1.0         | 100               | 5.0                   |
|                      | BEHTBP          | 500                   | 1.0         | 100               | 5.0                   |
|                      | <u>pTBX</u>     | 250                   | 1.0         | 100               | 2.5                   |
|                      | PBBZ            | 250                   | 1.0         | 100               | 2.5                   |
| <i>BFR-PAR</i>       |                 |                       | <i>1.0</i>  | <i>100</i>        |                       |
|                      | BDE-1           | 200                   | 1.0         | 100               | 2.0                   |
|                      | BDE-2           | 200                   | 1.0         | 100               | 2.0                   |
|                      | BDE-3           | 200                   | 1.0         | 100               | 2.0                   |
|                      | BDE-7           | 200                   | 1.0         | 100               | 2.0                   |
|                      | BDE-10          | 200                   | 1.0         | 100               | 2.0                   |
|                      | BDE-15          | 200                   | 1.0         | 100               | 2.0                   |
|                      | BDE-17          | 192                   | 1.0         | 100               | 1.92                  |
|                      | BDE-28          | 200                   | 1.0         | 100               | 2.0                   |

|  |  |       |                   |                   |      |
|--|--|-------|-------------------|-------------------|------|
|  | BDE-30                                 | 200   | 1.0               | 100               | 2.0  |
|  | HBB                                    | 200   | 1.0               | 100               | 2.0  |
|  | PBEB                                   | 200   | 1.0               | 100               | 2.0  |
|  | BDE-47                                 | 400   | 1.0               | 100               | 4.0  |
|  | BDE-49                                 | 400   | 1.0               | 100               | 4.0  |
|  | BDE-66                                 | 400   | 1.0               | 100               | 4.0  |
|  | BDE-71                                 | 400   | 1.0               | 100               | 4.0  |
|  | BDE-77                                 | 400   | 1.0               | 100               | 4.0  |
|  | BDE-85                                 | 400   | 1.0               | 100               | 4.0  |
|  | BDE-99                                 | 400   | 1.0               | 100               | 4.0  |
|  | BDE-100                                | 400   | 1.0               | 100               | 4.0  |
|  | BDE-119                                | 400   | 1.0               | 100               | 4.0  |
|  | BDE-126                                | 400   | 1.0               | 100               | 4.0  |
|  | BDE-138                                | 400   | 1.0               | 100               | 4.0  |
|  | BDE-139                                | 400   | 1.0               | 100               | 4.0  |
|  | BDE-140                                | 400   | 1.0               | 100               | 4.0  |
|  | BDE-153                                | 400   | 1.0               | 100               | 4.0  |
|  | BDE-154                                | 400   | 1.0               | 100               | 4.0  |
|  | BDE-156                                | 400   | 1.0               | 100               | 4.0  |
|  | BDE-169                                | 400   | 1.0               | 100               | 4.0  |
|  | BB-153                                 | 400   | 1.0               | 100               | 4.0  |
|  | BTBPE                                  | 400   | 1.0               | 100               | 4.0  |
|  | BDE-171                                | 800   | 1.0               | 100               | 8.0  |
|  | BDE-180                                | 800   | 1.0               | 100               | 8.0  |
|  | BDE-183                                | 800   | 1.0               | 100               | 8.0  |
|  | BDE-184                                | 800   | 1.0               | 100               | 8.0  |
|  | BDE-191                                | 800   | 1.0               | 100               | 8.0  |
|  | BDE-196                                | 800   | 1.0               | 100               | 8.0  |
|  | BDE-197                                | 800   | 1.0               | 100               | 8.0  |
|  | BDE-201                                | 800   | 1.0               | 100               | 8.0  |
|  | BDE-203                                | 800   | 1.0               | 100               | 8.0  |
|  | BDE-204                                | 800   | 1.0               | 100               | 8.0  |
|  | BDE-205                                | 800   | 1.0               | 100               | 8.0  |
|  | BDE-206                                | 2,000 | 1.0               | 100               | 20.0 |
|  | BDE-207                                | 2,000 | 1.0               | 100               | 20.0 |
|  | BDE-208                                | 2,000 | 1.0               | 100               | 20.0 |
|  | BDE-209                                | 2,000 | 1.0               | 100               | 20.0 |
|  | DBDPE                                  | 4,000 | 1.0               | 100               | 40.0 |
|  | <b><i>PBDE IS Stock</i></b>            |       | <b><i>1.0</i></b> | <b><i>100</i></b> |      |
|  | BDE-118                                | 500   | 1.0               | 100               | 5.0  |
|  | BDE-181                                | 1,000 | 1.0               | 100               | 10.0 |
|  | <b><i>PBDE SS Stock</i></b>            |       | <b><i>1.0</i></b> | <b><i>100</i></b> |      |
|  | BDE-77                                 | 300   | 1.0               | 100               | 3.0  |
|  | BDE-166                                | 500   | 1.0               | 100               | 5.0  |
|  | <sup>13</sup> C <sub>12</sub> -BDE-209 | 400   | 1.0               | 100               | 4.0  |



**Table 66. PBDE concentrations for compounds of the IADN annual reporting list in the PBDE Calibration Standard.**

| Compound       | Conc., ng/mL | Compound                               | Conc., ng/mL |
|----------------|--------------|--|--------------|
| BDE-15         | 2.0          | BDE-208                                | 20.0         |
| BDE-17         | 1.92         | BDE-209                                | 20.0         |
| BDE-28         | 2.0          | pTBX                                   | 2.5          |
| BDE-47         | 4.0          | PBBZ                                   | 2.5          |
| BDE-49         | 4.0          | PBEB                                   | 2.0          |
| BDE-66         | 4.0          | HBB                                    | 2.0          |
| BDE-85         | 4.0          | EHTBB                                  | 5.0          |
| BDE-99         | 4.0          | HBCD                                   | 10.0         |
| BDE-100        | 4.0          | TBE                                    | 4.0          |
| BDE-139        | 4.0          | BEHTBP                                 | 5.0          |
| BDE-140        | 4.0          | syn-DP                                 | 5.0          |
| BDE-153        | 4.0          | anti-DP                                | 5.0          |
| BDE-154+BB-153 | 8.0          | DBDPE                                  | 40.0         |
| BDE-183        | 8.0          |  |              |
| BDE-197        | 8.0          | BDE-77                                 | 7.0          |
| BDE-201        | 8.0          | BDE-166                                | 5.0          |
| BDE-203        | 8.0          | <sup>13</sup> C <sub>12</sub> -BDE-209 | 4.0          |
| BDE-206        | 20.0         | BDE-118                                | 5.0          |
| BDE-207        | 20.0         | BDE-181                                | 10.0         |

### **PBDE Common Reference Standards**

#### *Stock Mixtures*

**Instructions:** Two separate stocks are prepared to check the PBDE calibration.

**PBDE CRS1 Stock:** Mix 1.0 mL aliquots of S-21223 and S-20375 (Table 59) as shown in Table 67 and dilute to 10 mL with hexane.

**PBDE CRS2 Stock:** Combine aliquots of PBDE IS Stock (Table 61), PBDE SS Stock (Table 63), and selected individual PBDE compounds (Table 58) as shown in Table 68 and dilute to 25 mL with hexane.

**Table 67. Table of PBDE CRS1 Stock.**

| Stock                 | Compound | Stock conc., ng/mL | Aliquot, mL       | Final Vol., mL     | Final conc., ng/mL |
|-----------------------|----------|--------------------|-------------------|--------------------|--------------------|
| <b><i>S-20375</i></b> |          |                    | <b><i>1.0</i></b> | <b><i>10.0</i></b> |                    |
|                       | BDE-140  | 3,003              | 1.0               | 10.0               | 300                |
|                       | BDE-197  | 3,940              | 1.0               | 10.0               | 394                |
|                       | BDE-202  | 4,024              | 1.0               | 10.0               | 402                |
|                       | BDE-206  | 4,975              | 1.0               | 10.0               | 498                |
| <b><i>S-21223</i></b> |          |                    | <b><i>1.0</i></b> | <b><i>10.0</i></b> |                    |
|                       | BDE-1    | 1,503              | 1.0               | 10.0               | 150                |
|                       | BDE-2    | 1,485              | 1.0               | 10.0               | 149                |
|                       | BDE-3    | 1,487              | 1.0               | 10.0               | 149                |
|                       | BDE-7    | 1,496              | 1.0               | 10.0               | 150                |
|                       | BDE-8    | 1,515              | 1.0               | 10.0               | 152                |

|  |         |       |     |      |     |
|--|---------|-------|-----|------|-----|
|  | BDE-10  | 1,524 | 1.0 | 10.0 | 152 |
|  | BDE-11  | 1,485 | 1.0 | 10.0 | 149 |
|  | BDE-12  | 1,501 | 1.0 | 10.0 | 150 |
|  | BDE-13  | 1,514 | 1.0 | 10.0 | 151 |
|  | BDE-15  | 1,501 | 1.0 | 10.0 | 150 |
|  | BDE-17  | 1,528 | 1.0 | 10.0 | 153 |
|  | BDE-25  | 1,509 | 1.0 | 10.0 | 151 |
|  | BDE-28  | 1,518 | 1.0 | 10.0 | 152 |
|  | BDE-30  | 1,489 | 1.0 | 10.0 | 149 |
|  | BDE-32  | 1,516 | 1.0 | 10.0 | 152 |
|  | BDE-33  | 1,499 | 1.0 | 10.0 | 150 |
|  | BDE-35  | 1,508 | 1.0 | 10.0 | 151 |
|  | BDE-37  | 1,504 | 1.0 | 10.0 | 150 |
|  | BDE-47  | 1,504 | 1.0 | 10.0 | 150 |
|  | BDE-49  | 1,506 | 1.0 | 10.0 | 151 |
|  | BDE-66  | 1,531 | 1.0 | 10.0 | 153 |
|  | BDE-71  | 1,514 | 1.0 | 10.0 | 151 |
|  | BDE-75  | 1,503 | 1.0 | 10.0 | 150 |
|  | BDE-77  | 1,503 | 1.0 | 10.0 | 150 |
|  | BDE-85  | 2,237 | 1.0 | 10.0 | 224 |
|  | BDE-99  | 2,251 | 1.0 | 10.0 | 225 |
|  | BDE-100 | 2,264 | 1.0 | 10.0 | 226 |
|  | BDE-116 | 2,274 | 1.0 | 10.0 | 227 |
|  | BDE-119 | 2,292 | 1.0 | 10.0 | 229 |
|  | BDE-126 | 2,240 | 1.0 | 10.0 | 224 |
|  | BDE-138 | 3,033 | 1.0 | 10.0 | 303 |
|  | BDE-153 | 3,019 | 1.0 | 10.0 | 302 |
|  | BDE-154 | 3,012 | 1.0 | 10.0 | 301 |
|  | BDE-155 | 3,022 | 1.0 | 10.0 | 302 |
|  | BDE-166 | 2,958 | 1.0 | 10.0 | 296 |
|  | BDE-181 | 3,696 | 1.0 | 10.0 | 370 |
|  | BDE-183 | 3,789 | 1.0 | 10.0 | 379 |
|  | BDE-190 | 3,702 | 1.0 | 10.0 | 370 |

**Table 68. Table of PBDE CRS2 Stock mixture.**

| Stock                    | Compound                               | Stock conc.,<br>ng/mL | Aliquot, mL | Final Vol.,<br>mL | Final conc.,<br>ng/mL |
|--------------------------|--|-----------------------|-------------|-------------------|-----------------------|
| <i>PBDE IS Stock</i>     |  |                       | <b>2.5</b>  | <b>25</b>         |                       |
|                          | BDE-118                                | 500                   | 2.5         | 25                | 50                    |
|                          | BDE-181                                | 1,000                 | 2.5         | 25                | 100                   |
| <i>PBDE SS Stock</i>     |  |                       | <b>2.5</b>  | <b>25</b>         |                       |
|                          | BDE-77                                 | 300                   | 2.5         | 25                | 30                    |
|                          | BDE-166                                | 500                   | 2.5         | 25                | 50                    |
|                          | <sup>13</sup> C <sub>12</sub> -BDE-209 | 400                   | 2.5         | 25                | 40                    |
| <i>Individual Stocks</i> |  |                       |             |                   |                       |
|                          | BDE-209                                | 50,000                | 0.1         | 25                | 200                   |
|                          | DBDPE                                  | 25,000                | 0.4         | 25                | 400                   |

## Working Mixtures

**Instructions:** Two working mixtures are prepared from the two PBDE CRS Stocks.

PBDE CRS1 Working: Combine aliquots of the PBDE CRS1 Stock (Table 67), Non-BDE Stock (Table 60), PBDE IS Stock (Table 61), and PBDE SS Stock (Table 63) as shown in Table 69 and dilute to 100 mL with hexane.

PBDE CRS2 Working: Dilute the PBDE CRS2 Stock ten-fold as shown in Table 70.

Table 69. Table of PBDE CRS1 working mixture.

| Stock                         | Compound                               | Stock conc.,<br>ng/mL | Aliquot, mL       | Final Vol.,<br>mL | Final conc.,<br>ng/mL |
|-------------------------------|--|-----------------------|-------------------|-------------------|-----------------------|
| <b><i>PBDE IS Stock</i></b>   |  |                       | <b><i>1.0</i></b> | <b><i>100</i></b> |                       |
|                               | BDE-118                                | 500                   | 1.0               | 100               | 5.0                   |
|                               | BDE-181                                | 1,000                 | 1.0               | 100               | 10.0                  |
| <b><i>PBDE SS Stock</i></b>   |  |                       | <b><i>1.0</i></b> | <b><i>100</i></b> |                       |
|                               | BDE-77                                 | 300                   | 1.0               | 100               | 3.0                   |
|                               | BDE-166                                | 500                   | 1.0               | 100               | 5.0                   |
|                               | <sup>13</sup> C <sub>12</sub> -BDE-209 | 400                   | 1.0               | 100               | 4.0                   |
| <b><i>Non-BDE Stock</i></b>   |  |                       | <b><i>1.0</i></b> | <b><i>100</i></b> |                       |
|                               | <i>anti</i> -DP                        | 500                   | 1.0               | 100               | 5.0                   |
|                               | <i>syn</i> -DP                         | 500                   | 1.0               | 100               | 5.0                   |
|                               | $\alpha$ -HBCD                         | 1,000                 | 1.0               | 100               | 10.0                  |
|                               | EHTBB                                  | 500                   | 1.0               | 100               | 5.0                   |
|                               | BEHTBP                                 | 500                   | 1.0               | 100               | 5.0                   |
|                               | <u>pTBX</u>                            | 250                   | 1.0               | 100               | 2.5                   |
|                               | PBBZ                                   | 250                   | 1.0               | 100               | 2.5                   |
| <b><i>PBDE CRS1 Stock</i></b> |  |                       | <b><i>1.0</i></b> | <b><i>100</i></b> |                       |
|                               | BDE-1                                  | 150                   | 1.0               | 100               | 1.5                   |
|                               | BDE-2                                  | 149                   | 1.0               | 100               | 1.5                   |
|                               | BDE-3                                  | 149                   | 1.0               | 100               | 1.5                   |
|                               | BDE-7                                  | 150                   | 1.0               | 100               | 1.5                   |
|                               | BDE-8                                  | 152                   | 1.0               | 100               | 1.5                   |
|                               | BDE-10                                 | 152                   | 1.0               | 100               | 1.5                   |
|                               | BDE-11                                 | 149                   | 1.0               | 100               | 1.5                   |
|                               | BDE-12                                 | 150                   | 1.0               | 100               | 1.5                   |
|                               | BDE-13                                 | 151                   | 1.0               | 100               | 1.5                   |
|                               | BDE-15                                 | 150                   | 1.0               | 100               | 1.5                   |
|                               | BDE-17                                 | 153                   | 1.0               | 100               | 1.5                   |
|                               | BDE-25                                 | 151                   | 1.0               | 100               | 1.5                   |
|                               | BDE-28                                 | 152                   | 1.0               | 100               | 1.5                   |
|                               | BDE-30                                 | 149                   | 1.0               | 100               | 1.5                   |
|                               | BDE-32                                 | 152                   | 1.0               | 100               | 1.5                   |
|                               | BDE-33                                 | 150                   | 1.0               | 100               | 1.5                   |
|                               | BDE-35                                 | 151                   | 1.0               | 100               | 1.5                   |
|                               | BDE-37                                 | 150                   | 1.0               | 100               | 1.5                   |
|                               | BDE-47                                 | 150                   | 1.0               | 100               | 1.5                   |
|                               | BDE-49                                 | 151                   | 1.0               | 100               | 1.5                   |
|                               | BDE-66                                 | 153                   | 1.0               | 100               | 1.5                   |
|                               | BDE-71                                 | 151                   | 1.0               | 100               | 1.5                   |
|                               | BDE-75                                 | 150                   | 1.0               | 100               | 1.5                   |
|                               | BDE-77                                 | 150                   | 1.0               | 100               | 1.5                   |
|                               | BDE-85                                 | 224                   | 1.0               | 100               | 2.2                   |
|                               | BDE-99                                 | 225                   | 1.0               | 100               | 2.3                   |

|  |         |     |     |     |     |
|--|---------|-----|-----|-----|-----|
|  | BDE-100 | 226 | 1.0 | 100 | 2.3 |
|  | BDE-116 | 227 | 1.0 | 100 | 2.3 |
|  | BDE-119 | 229 | 1.0 | 100 | 2.3 |
|  | BDE-126 | 224 | 1.0 | 100 | 2.2 |
|  | BDE-138 | 303 | 1.0 | 100 | 3.0 |
|  | BDE-140 | 300 | 1.0 | 100 | 3.0 |
|  | BDE-153 | 302 | 1.0 | 100 | 3.0 |
|  | BDE-154 | 301 | 1.0 | 100 | 3.0 |
|  | BDE-155 | 302 | 1.0 | 100 | 3.0 |
|  | BDE-166 | 296 | 1.0 | 100 | 3.0 |
|  | BDE-181 | 370 | 1.0 | 100 | 3.7 |
|  | BDE-183 | 379 | 1.0 | 100 | 3.8 |
|  | BDE-190 | 370 | 1.0 | 100 | 3.7 |
|  | BDE-197 | 394 | 1.0 | 100 | 3.9 |
|  | BDE-202 | 402 | 1.0 | 100 | 4.0 |
|  | BDE-206 | 498 | 1.0 | 100 | 5.0 |

Table 70. Table of the PBDE CRS2 Working mixture.

| Stock                  | Compound                               | Stock conc.,<br>ng/mL | Aliquot, mL | Final Vol.,<br>mL | Final conc.,<br>ng/mL |
|------------------------|--|-----------------------|-------------|-------------------|-----------------------|
| <i>PBDE CRS2 Stock</i> |  |                       | <i>1.0</i>  | <i>10</i>         |                       |
|                        | BDE-118                                | 50                    | 1.0         | 10                | 5.0                   |
|                        | BDE-181                                | 100                   | 1.0         | 10                | 10.0                  |
|                        | BDE-77                                 | 30                    | 1.0         | 10                | 3.0                   |
|                        | BDE-166                                | 50                    | 1.0         | 10                | 5.0                   |
|                        | <sup>13</sup> C <sub>12</sub> -BDE-209 | 40                    | 1.0         | 10                | 4.0                   |
|                        | BDE-209                                | 200                   | 1.0         | 10                | 20.0                  |
|                        | DBDPE                                  | 400                   | 1.0         | 10                | 40.0                  |

Table 71. Table of individual BFRs and their concentrations (ng/mL) from the annual IADN reporting list in the PBDE CRS1 and CRS2 working mixtures.

| PBDE CRS1 Working |     |         |      | PBDE CRS2 Working                      |      |
|-------------------|-----|---------|------|--|------|
| BDE-15            | 1.5 | BDE-85  | 2.2  | BDE-77                                 | 3.0  |
| pTBX              | 2.5 | BDE-154 | 3.0  | BDE-118                                | 5.0  |
| PBBZ              | 2.5 | BDE-153 | 3.0  | BDE-166                                | 5.0  |
| BDE-17            | 3.0 | HBCD    | 10.0 | BDE-181                                | 10.0 |
| BDE-28            | 3.0 | BDE-140 | 3.0  | <sup>13</sup> C <sub>12</sub> -BDE-209 | 4.0  |
| BDE-49            | 1.5 | BDE-166 | 8.0  | BDE-209                                | 20.0 |
| BDE-47            | 1.5 | BDE-183 | 3.8  | DBDPE                                  | 40.0 |
| BDE-66            | 1.5 | BDE-181 | 13.7 |  |      |
| BDE-77            | 4.5 | BEHTBP  | 5.0  |  |      |
| BDE-100           | 2.3 | syn-DP  | 5.0  |  |      |
| BDE-99            | 2.3 | BDE-197 | 3.9  |  |      |
| EHTBB             | 5.0 | anti-DP | 5.0  |  |      |
| BDE-118           | 5.0 | BDE-206 | 5.0  |  |      |

Note: BDE-17 and BDE-28 have concentrations greater than their actual stock concentrations due to co-elutions with non-target BDE compounds.

## **PBDE Matrix Spike**

### ***Working Mixtures***

**Instructions:** Two PBDE matrix spike mixtures are prepared.

PBDE MS Mix A: Combine indicated aliquots of the non-BDE stock (Table 60) and BDE-MXE (Table 59) mixtures as shown in Table 72 then dilute to 25 mL with hexane.

PBDE MS Mix B: Combine indicated aliquots of selected individual PBDE standards (Table 58) as shown in Table 73, then dilute to 25 mL.

**Table 72. Table of the PBDE Matrix Spike Mix A working mixture.**

| Stock                       | Compound        | Stock conc.,<br>ng/mL | Aliquot, mL | Final Vol.,<br>mL | Final conc.,<br>ng/mL |
|-----------------------------|-----------------|-----------------------|-------------|-------------------|-----------------------|
| <b><i>Non-BDE Stock</i></b> |                 |                       | <b>2.5</b>  | <b>25</b>         |                       |
|                             | <i>anti</i> -DP | 500                   | 2.5         | 25                | 50                    |
|                             | <i>syn</i> -DP  | 500                   | 2.5         | 25                | 50                    |
|                             | $\alpha$ -HBCD  | 1,000                 | 2.5         | 25                | 100                   |
|                             | EHTBB           | 500                   | 2.5         | 25                | 50                    |
|                             | BEHTBP          | 500                   | 2.5         | 25                | 50                    |
|                             | <u>p</u> TBX    | 250                   | 2.5         | 25                | 25                    |
|                             | PBBZ            | 250                   | 2.5         | 25                | 25                    |
| <b><i>BDE-MXE</i></b>       |                 |                       | <b>1.0</b>  | <b>100</b>        |                       |
|                             | BDE-3           | 1,000                 | 1.0         | 25                | 40                    |
|                             | BDE-7           | 1,000                 | 1.0         | 25                | 40                    |
|                             | BDE-15          | 1,000                 | 1.0         | 25                | 40                    |
|                             | BDE-17          | 1,000                 | 1.0         | 25                | 40                    |
|                             | BDE-28          | 1,000                 | 1.0         | 25                | 40                    |
|                             | BDE-47          | 1,000                 | 1.0         | 25                | 40                    |
|                             | BDE-49          | 1,000                 | 1.0         | 25                | 40                    |
|                             | BDE-66          | 1,000                 | 1.0         | 25                | 40                    |
|                             | BDE-71          | 1,000                 | 1.0         | 25                | 40                    |
|                             | BDE-77          | 1,000                 | 1.0         | 25                | 40                    |
|                             | BDE-85          | 1,000                 | 1.0         | 25                | 40                    |
|                             | BDE-99          | 1,000                 | 1.0         | 25                | 40                    |
|                             | BDE-100         | 1,000                 | 1.0         | 25                | 40                    |
|                             | BDE-119         | 1,000                 | 1.0         | 25                | 40                    |
|                             | BDE-126         | 1,000                 | 1.0         | 25                | 40                    |
|                             | BDE-138         | 2,000                 | 1.0         | 25                | 80                    |
|                             | BDE-153         | 2,000                 | 1.0         | 25                | 80                    |
|                             | BDE-154         | 2,000                 | 1.0         | 25                | 80                    |
|                             | BDE-156         | 2,000                 | 1.0         | 25                | 80                    |
|                             | BDE-183         | 2,000                 | 1.0         | 25                | 80                    |

|  |         |       |     |    |     |
|--|---------|-------|-----|----|-----|
|  | BDE-184 | 2,000 | 1.0 | 25 | 80  |
|  | BDE-191 | 2,000 | 1.0 | 25 | 80  |
|  | BDE-196 | 2,000 | 1.0 | 25 | 80  |
|  | BDE-197 | 2,000 | 1.0 | 25 | 80  |
|  | BDE-206 | 5,000 | 1.0 | 25 | 200 |
|  | BDE-207 | 5,000 | 1.0 | 25 | 200 |
|  | BDE-209 | 5,000 | 1.0 | 25 | 200 |

**Table 73. Table of the PBDE Matrix Spike Mix B working mixture.**

| Compound | Stock conc.,<br>ng/mL | Aliquot, mL | Final Vol.,<br>mL | Final conc.,<br>ng/mL |
|----------|-----------------------|-------------|-------------------|-----------------------|
| PBEB     | 5,000                 | 0.25        | 25                | 50                    |
| HBB      | 5,000                 | 0.25        | 25                | 50                    |
| TBE      | 5,000                 | 0.5         | 25                | 100                   |
| DBDPE    | 5,000                 | 2.0         | 25                | 400                   |

### PBDE Linearity Standard

#### PBDE Linearity High Stock

**Instructions:** Mix indicated aliquot volumes of Non-BDE stock (Tables 60), BFR-PAR (Tables 59), and the PBDE SS Stock mix (Table 63) as shown in Table 74 and dilute to 4 mL in hexane.

**Table 74. Table of the PBDE Linearity High Stock mixture.**

| Stock                | Compound        | Stock conc.,<br>ng/mL | Aliquot, mL | Final Vol.,<br>mL | Final conc.,<br>ng/mL |
|----------------------|-----------------|-----------------------|-------------|-------------------|-----------------------|
| <i>Non-BDE Stock</i> |                 |                       | <b>0.8</b>  | <b>4.0</b>        |                       |
|                      | <i>anti</i> -DP | 500                   | 0.8         | 4.0               | 100                   |
|                      | <i>syn</i> -DP  | 500                   | 0.8         | 4.0               | 100                   |
|                      | $\alpha$ -HBCD  | 1,000                 | 0.8         | 4.0               | 200                   |
|                      | EHTBB           | 500                   | 0.8         | 4.0               | 100                   |
|                      | BEHTBP          | 500                   | 0.8         | 4.0               | 100                   |
|                      | <u>pTBX</u>     | 250                   | 0.8         | 4.0               | 50                    |
|                      | PBBZ            | 250                   | 0.8         | 4.0               | 50                    |
| <i>BFR-PAR</i>       |                 |                       | <b>1.0</b>  | <b>4.0</b>        |                       |
|                      | BDE-1           | 200                   | 1.0         | 4.0               | 50                    |
|                      | BDE-2           | 200                   | 1.0         | 4.0               | 50                    |
|                      | BDE-3           | 200                   | 1.0         | 4.0               | 50                    |
|                      | BDE-7           | 200                   | 1.0         | 4.0               | 50                    |
|                      | BDE-10          | 200                   | 1.0         | 4.0               | 50                    |
|                      | BDE-15          | 200                   | 1.0         | 4.0               | 50                    |
|                      | BDE-17          | 192                   | 1.0         | 4.0               | 48                    |
|                      | BDE-28          | 200                   | 1.0         | 4.0               | 50                    |
|                      | BDE-30          | 200                   | 1.0         | 4.0               | 50                    |
|                      | HBB             | 200                   | 1.0         | 4.0               | 50                    |
|                      | PBEB            | 200                   | 1.0         | 4.0               | 50                    |
|                      | BDE-47          | 400                   | 1.0         | 4.0               | 100                   |
|                      | BDE-49          | 400                   | 1.0         | 4.0               | 100                   |

|  |  |       |                    |                   |       |
|--|--|-------|--------------------|-------------------|-------|
|  | BDE-66                                 | 400   | 1.0                | 4.0               | 100   |
|  | BDE-71                                 | 400   | 1.0                | 4.0               | 100   |
|  | BDE-77                                 | 400   | 1.0                | 4.0               | 100   |
|  | BDE-85                                 | 400   | 1.0                | 4.0               | 100   |
|  | BDE-99                                 | 400   | 1.0                | 4.0               | 100   |
|  | BDE-100                                | 400   | 1.0                | 4.0               | 100   |
|  | BDE-119                                | 400   | 1.0                | 4.0               | 100   |
|  | BDE-126                                | 400   | 1.0                | 4.0               | 100   |
|  | BDE-138                                | 400   | 1.0                | 4.0               | 100   |
|  | BDE-139                                | 400   | 1.0                | 4.0               | 100   |
|  | BDE-140                                | 400   | 1.0                | 4.0               | 100   |
|  | BDE-153                                | 400   | 1.0                | 4.0               | 100   |
|  | BDE-154                                | 400   | 1.0                | 4.0               | 100   |
|  | BDE-156                                | 400   | 1.0                | 4.0               | 100   |
|  | BDE-169                                | 400   | 1.0                | 4.0               | 100   |
|  | BB-153                                 | 400   | 1.0                | 4.0               | 100   |
|  | BTBPE                                  | 400   | 1.0                | 4.0               | 100   |
|  | BDE-171                                | 800   | 1.0                | 4.0               | 200   |
|  | BDE-180                                | 800   | 1.0                | 4.0               | 200   |
|  | BDE-183                                | 800   | 1.0                | 4.0               | 200   |
|  | BDE-184                                | 800   | 1.0                | 4.0               | 200   |
|  | BDE-191                                | 800   | 1.0                | 4.0               | 200   |
|  | BDE-196                                | 800   | 1.0                | 4.0               | 200   |
|  | BDE-197                                | 800   | 1.0                | 4.0               | 200   |
|  | BDE-201                                | 800   | 1.0                | 4.0               | 200   |
|  | BDE-203                                | 800   | 1.0                | 4.0               | 200   |
|  | BDE-204                                | 800   | 1.0                | 4.0               | 200   |
|  | BDE-205                                | 800   | 1.0                | 4.0               | 200   |
|  | BDE-206                                | 2,000 | 1.0                | 4.0               | 500   |
|  | BDE-207                                | 2,000 | 1.0                | 4.0               | 500   |
|  | BDE-208                                | 2,000 | 1.0                | 4.0               | 500   |
|  | BDE-209                                | 2,000 | 1.0                | 4.0               | 500   |
|  | DBDPE                                  | 4,000 | 1.0                | 4.0               | 1,000 |
|  | <b><i>PBDE SS Stock</i></b>            |       | <b><i>0.25</i></b> | <b><i>4.0</i></b> |       |
|  | BDE-77                                 | 300   | 0.25               | 4.0               | 18.75 |
|  | BDE-166                                | 500   | 0.25               | 4.0               | 31.25 |
|  | <sup>13</sup> C <sub>12</sub> -BDE-209 | 400   | 0.25               | 4.0               | 25.0  |

### Working Mixtures

**Instructions:** Perform dilution of the PBDE Linearity High Stock (Table 74) as shown in Table 75. Each dilution is done separately.

**Table 75. Table of the various PBDE linearity levels.**

| Level | Stock         | Stock conc.,<br>ng/mL | Aliquot, mL | Final Vol.,<br>mL | Final Conc.,<br>ng/mL |
|-------|---------------|-----------------------|-------------|-------------------|-----------------------|
| 01    | PBDE Lin High | 25-1000               |             |                   | 25-1000               |
| 02    | PBDE Lin High | 25-1000               | 0.8         | 1.0               | 20-800                |
| 03    | PBDE Lin High | 25-1000               | 0.6         | 1.0               | 15-600                |
| 04    | PBDE Lin High | 25-1000               | 0.4         | 1.0               | 10-400                |

|    |               |          |     |     |          |
|----|---------------|----------|-----|-----|----------|
| 05 | PBDE Lin High | 25-1000  | 0.2 | 1.0 | 5-200    |
| 06 | PBDE Lin High | 25-1000  | 0.1 | 1.0 | 2.5-100  |
| 07 | PBDE Lin 06   | 2.5-100  | 0.5 | 1.0 | 1.25-50  |
| 08 | PBDE Lin 07   | 1.25-50  | 0.5 | 1.0 | 0.6-25   |
| 09 | PBDE Lin 08   | 0.6-25   | 0.5 | 1.0 | 0.3-12.5 |
| 10 | PBDE Lin 09   | 0.3-12.5 | 0.5 | 1.0 | 0.2-6.25 |
| 11 | PBDE Lin 10   | 0.2-6.25 | 0.5 | 1.0 | 0.1-3.1  |
| 12 | PBDE Lin 11   | 0.1-3.1  | 0.5 | 1.0 | 0.04-1.6 |
| 13 | PBDE Lin 12   | 0.04-1.6 | 0.5 | 1.0 | 0.02-0.8 |
| 14 | PBDE Lin 13   | 0.02-0.8 | 0.5 | 1.0 | 0.01-0.4 |

**Table 76. Table of individual compound concentrations in the various PBDE Linearity levels.**

| BFRs    | Concentration (ng/mL) per Level |     |     |    |    |      |      |      |      |          |           |           |           |           |
|---------|---------------------------------|-----|-----|----|----|------|------|------|------|----------|-----------|-----------|-----------|-----------|
|         | 01                              | 02  | 03  | 04 | 05 | 06   | 07   | 08   | 09   | 10       | 11        | 12        | 13        | 14        |
| BDE-15  | 50                              | 40  | 30  | 20 | 10 | 5.0  | 2.5  | 1.25 | 0.63 | 0.3<br>1 | 0.15<br>6 | 0.07<br>8 | 0.03<br>9 | 0.02<br>0 |
| pTBX    | 50                              | 40  | 30  | 20 | 10 | 5.0  | 2.5  | 1.25 | 0.63 | 0.3<br>1 | 0.15<br>6 | 0.07<br>8 | 0.03<br>9 | 0.02<br>0 |
| PBBZ    | 50                              | 40  | 30  | 20 | 10 | 5.0  | 2.5  | 1.25 | 0.63 | 0.3<br>1 | 0.15<br>6 | 0.07<br>8 | 0.03<br>9 | 0.02<br>0 |
| BDE-17  | 48                              | 38  | 29  | 19 | 10 | 4.8  | 2.4  | 1.20 | 0.60 | 0.3<br>0 | 0.15<br>0 | 0.07<br>5 | 0.03<br>8 | 0.01<br>9 |
| BDE-28  | 50                              | 40  | 30  | 20 | 10 | 5.0  | 2.5  | 1.25 | 0.63 | 0.3<br>1 | 0.15<br>6 | 0.07<br>8 | 0.03<br>9 | 0.02<br>0 |
| PBEB    | 50                              | 40  | 30  | 20 | 10 | 5.0  | 2.5  | 1.25 | 0.63 | 0.3<br>1 | 0.15<br>6 | 0.07<br>8 | 0.03<br>9 | 0.02<br>0 |
| HBB     | 50                              | 40  | 30  | 20 | 10 | 5.0  | 2.5  | 1.25 | 0.63 | 0.3<br>1 | 0.15<br>6 | 0.07<br>8 | 0.03<br>9 | 0.02<br>0 |
| BDE-49  | 100                             | 80  | 60  | 40 | 20 | 10.0 | 5.0  | 2.50 | 1.25 | 0.6<br>3 | 0.31<br>3 | 0.15<br>6 | 0.07<br>8 | 0.03<br>9 |
| BDE-47  | 100                             | 80  | 60  | 40 | 20 | 10.0 | 5.0  | 2.50 | 1.25 | 0.6<br>3 | 0.31<br>3 | 0.15<br>6 | 0.07<br>8 | 0.03<br>9 |
| BDE-66  | 100                             | 80  | 60  | 40 | 20 | 10.0 | 5.0  | 2.50 | 1.25 | 0.6<br>3 | 0.31<br>3 | 0.15<br>6 | 0.07<br>8 | 0.03<br>9 |
| BDE-77  | 119                             | 95  | 71  | 48 | 24 | 11.9 | 5.9  | 2.97 | 1.48 | 0.7<br>4 | 0.37<br>1 | 0.18<br>6 | 0.09<br>3 | 0.04<br>6 |
| BDE-100 | 100                             | 80  | 60  | 40 | 20 | 10.0 | 5.0  | 2.50 | 1.25 | 0.6<br>3 | 0.31<br>3 | 0.15<br>6 | 0.07<br>8 | 0.03<br>9 |
| BDE-99  | 100                             | 80  | 60  | 40 | 20 | 10.0 | 5.0  | 2.50 | 1.25 | 0.6<br>3 | 0.31<br>3 | 0.15<br>6 | 0.07<br>8 | 0.03<br>9 |
| EHTBB   | 100                             | 80  | 60  | 40 | 20 | 10.0 | 5.0  | 2.50 | 1.25 | 0.6<br>3 | 0.31<br>3 | 0.15<br>6 | 0.07<br>8 | 0.03<br>9 |
| BDE-85  | 100                             | 80  | 60  | 40 | 20 | 10.0 | 5.0  | 2.50 | 1.25 | 0.6<br>3 | 0.31<br>3 | 0.15<br>6 | 0.07<br>8 | 0.03<br>9 |
| BDE-154 | 200                             | 160 | 120 | 80 | 40 | 20.0 | 10.0 | 5.00 | 2.50 | 1.2<br>5 | 0.62<br>5 | 0.31<br>3 | 0.15<br>6 | 0.07<br>8 |
| BDE-153 | 100                             | 80  | 60  | 40 | 20 | 10.0 | 5.0  | 2.50 | 1.25 | 0.6<br>3 | 0.31<br>3 | 0.15<br>6 | 0.07<br>8 | 0.03<br>9 |



|               |      |     |     |     |     |       |      |      |      |      |       |       |       |       |
|---------------|------|-----|-----|-----|-----|-------|------|------|------|------|-------|-------|-------|-------|
| BDE-139       | 100  | 80  | 60  | 40  | 20  | 10.0  | 5.0  | 2.50 | 1.25 | 0.63 | 0.313 | 0.156 | 0.078 | 0.039 |
| HBCD          | 200  | 160 | 120 | 80  | 40  | 20.0  | 10.0 | 5.00 | 2.50 | 1.25 | 0.625 | 0.313 | 0.156 | 0.078 |
| BDE-140       | 100  | 80  | 60  | 40  | 20  | 10.0  | 5.0  | 2.50 | 1.25 | 0.63 | 0.313 | 0.156 | 0.078 | 0.039 |
| BDE-166       | 31   | 25  | 19  | 13  | 6   | 3.1   | 1.6  | 0.78 | 0.39 | 0.20 | 0.098 | 0.049 | 0.024 | 0.012 |
| BDE-183       | 200  | 160 | 120 | 80  | 40  | 20.0  | 10.0 | 5.00 | 2.50 | 1.25 | 0.625 | 0.313 | 0.156 | 0.078 |
| TBE           | 100  | 80  | 60  | 40  | 20  | 10.0  | 5.0  | 2.50 | 1.25 | 0.63 | 0.313 | 0.156 | 0.078 | 0.039 |
| BEHTBP        | 100  | 80  | 60  | 40  | 20  | 10.0  | 5.0  | 2.50 | 1.25 | 0.63 | 0.313 | 0.156 | 0.078 | 0.039 |
| syn-DP        | 100  | 80  | 60  | 40  | 20  | 10.0  | 5.0  | 2.50 | 1.25 | 0.63 | 0.313 | 0.156 | 0.078 | 0.039 |
| BDE-201       | 200  | 160 | 120 | 80  | 40  | 20.0  | 10.0 | 5.00 | 2.50 | 1.25 | 0.625 | 0.313 | 0.156 | 0.078 |
| BDE-197       | 200  | 160 | 120 | 80  | 40  | 20.0  | 10.0 | 5.00 | 2.50 | 1.25 | 0.625 | 0.313 | 0.156 | 0.078 |
| anti-DP       | 100  | 80  | 60  | 40  | 20  | 10.0  | 5.0  | 2.50 | 1.25 | 0.63 | 0.313 | 0.156 | 0.078 | 0.039 |
| BDE-203       | 200  | 160 | 120 | 80  | 40  | 20.0  | 10.0 | 5.00 | 2.50 | 1.25 | 0.625 | 0.313 | 0.156 | 0.078 |
| BDE-208       | 500  | 400 | 300 | 200 | 100 | 50.0  | 25.0 | 12.5 | 6.25 | 3.13 | 1.563 | 0.781 | 0.391 | 0.195 |
| BDE-207       | 500  | 400 | 300 | 200 | 100 | 50.0  | 25.0 | 12.5 | 6.25 | 3.13 | 1.563 | 0.781 | 0.391 | 0.195 |
| BDE-206       | 500  | 400 | 300 | 200 | 100 | 50.0  | 25.0 | 12.5 | 6.25 | 3.13 | 1.563 | 0.781 | 0.391 | 0.195 |
| 13C12-BDE-209 | 25   | 20  | 15  | 10  | 5   | 2.5   | 1.3  | 0.63 | 0.31 | 0.16 | 0.078 | 0.039 | 0.020 | 0.010 |
| BDE-209       | 500  | 400 | 300 | 200 | 100 | 50.0  | 25.0 | 12.5 | 6.25 | 3.13 | 1.563 | 0.781 | 0.391 | 0.195 |
| DBDPE         | 1000 | 800 | 600 | 400 | 200 | 100.0 | 50.0 | 25.0 | 12.5 | 6.25 | 3.125 | 1.563 | 0.781 | 0.391 |

### **PBDE Instrument Detection Limit**

#### *Stock Mixture*

**Instructions:** Dilute 50 µL of the PBDE High Linearity stock (Table 74) to 4.0 mL with hexane as shown in Table 77.

**Table 77. Table of the PBDE IDL Stock Mixture.**

| Stock                            | Compound       | Stock conc., ng/mL | Aliquot, mL | Final Vol., mL | Final conc., ng/mL |
|----------------------------------|----------------|--------------------|-------------|----------------|--------------------|
| <i>PBDE High Linearity Stock</i> |                |                    | <i>0.05</i> | <i>4.0</i>     |                    |
|                                  | <i>anti-DP</i> | 100                | 0.05        | 4.0            | 1.25               |
|                                  | <i>syn-DP</i>  | 100                | 0.05        | 4.0            | 1.25               |
|                                  | <i>α-HBCD</i>  | 200                | 0.05        | 4.0            | 2.50               |

|  |                 |     |      |     |      |
|--|-----------------|-----|------|-----|------|
|  | EHTBB           | 100 | 0.05 | 4.0 | 1.25 |
|  | BEHTBP          | 100 | 0.05 | 4.0 | 1.25 |
|  | <del>pTBX</del> | 50  | 0.05 | 4.0 | 0.63 |
|  | PBBZ            | 50  | 0.05 | 4.0 | 0.63 |
|  | BDE-1           | 50  | 0.05 | 4.0 | 0.63 |
|  | BDE-2           | 50  | 0.05 | 4.0 | 0.63 |
|  | BDE-3           | 50  | 0.05 | 4.0 | 0.63 |
|  | BDE-7           | 50  | 0.05 | 4.0 | 0.63 |
|  | BDE-10          | 50  | 0.05 | 4.0 | 0.63 |
|  | BDE-15          | 50  | 0.05 | 4.0 | 0.63 |
|  | BDE-17          | 48  | 0.05 | 4.0 | 0.60 |
|  | BDE-28          | 50  | 0.05 | 4.0 | 0.63 |
|  | BDE-30          | 50  | 0.05 | 4.0 | 0.63 |
|  | HBB             | 50  | 0.05 | 4.0 | 0.63 |
|  | PBEB            | 50  | 0.05 | 4.0 | 0.63 |
|  | BDE-47          | 100 | 0.05 | 4.0 | 1.25 |
|  | BDE-49          | 100 | 0.05 | 4.0 | 1.25 |
|  | BDE-66          | 100 | 0.05 | 4.0 | 1.25 |
|  | BDE-71          | 100 | 0.05 | 4.0 | 1.25 |
|  | BDE-85          | 100 | 0.05 | 4.0 | 1.25 |
|  | BDE-99          | 100 | 0.05 | 4.0 | 1.25 |
|  | BDE-100         | 100 | 0.05 | 4.0 | 1.25 |
|  | BDE-119         | 100 | 0.05 | 4.0 | 1.25 |
|  | BDE-126         | 100 | 0.05 | 4.0 | 1.25 |
|  | BDE-138         | 100 | 0.05 | 4.0 | 1.25 |
|  | BDE-139         | 100 | 0.05 | 4.0 | 1.25 |
|  | BDE-140         | 100 | 0.05 | 4.0 | 1.25 |
|  | BDE-153         | 100 | 0.05 | 4.0 | 1.25 |
|  | BDE-154         | 100 | 0.05 | 4.0 | 1.25 |
|  | BDE-156         | 100 | 0.05 | 4.0 | 1.25 |
|  | BDE-169         | 100 | 0.05 | 4.0 | 1.25 |
|  | BB-153          | 100 | 0.05 | 4.0 | 1.25 |
|  | BTBPE           | 100 | 0.05 | 4.0 | 1.25 |
|  | BDE-171         | 200 | 0.05 | 4.0 | 2.50 |
|  | BDE-180         | 200 | 0.05 | 4.0 | 2.50 |
|  | BDE-183         | 200 | 0.05 | 4.0 | 2.50 |
|  | BDE-184         | 200 | 0.05 | 4.0 | 2.50 |
|  | BDE-191         | 200 | 0.05 | 4.0 | 2.50 |
|  | BDE-196         | 200 | 0.05 | 4.0 | 2.50 |
|  | BDE-197         | 200 | 0.05 | 4.0 | 2.50 |
|  | BDE-201         | 200 | 0.05 | 4.0 | 2.50 |
|  | BDE-203         | 200 | 0.05 | 4.0 | 2.50 |
|  | BDE-204         | 200 | 0.05 | 4.0 | 2.50 |
|  | BDE-205         | 200 | 0.05 | 4.0 | 2.50 |
|  | BDE-206         | 500 | 0.05 | 4.0 | 6.25 |
|  | BDE-207         | 500 | 0.05 | 4.0 | 6.25 |
|  | BDE-208         | 500 | 0.05 | 4.0 | 6.25 |
|  | BDE-209         | 500 | 0.05 | 4.0 | 6.25 |

|  |  |       |      |     |       |
|--|--|-------|------|-----|-------|
|  | DBDPE                                  | 1,000 | 0.05 | 4.0 | 12.50 |
|  | BDE-77                                 | 119   | 0.05 | 4.0 | 1.49  |
|  | BDE-166                                | 31.25 | 0.05 | 4.0 | 0.39  |
|  | <sup>13</sup> C <sub>12</sub> -BDE-209 | 25.0  | 0.05 | 4.0 | 0.31  |

### Working Mixture

**Instructions:** Combine aliquots of PBDE IDL Stock mixture (Table 77) and PBDE IS working mixtures as shown in Table 78.

**Table 78. Table of the PBDE IDL working mixture.**

| Stock          | Stock conc.,<br>ng/mL | Aliquot, mL | Final Vol.,<br>mL | Final Conc.,<br>ng/mL |
|----------------|-----------------------|-------------|-------------------|-----------------------|
| PBDE IDL Stock | 0.31 – 12.5           | 0.2         | 1.0               | 0.06 - 2.5            |
| PBDE Lin High  | 100 – 200             | 0.05        | 1.0               | 5.0 – 10.0            |

*Note: Occasionally multiple levels will need to be prepared to more accurately assess particular compounds given that this mixture has compounds over a range of concentrations.*

**Table 79. Table of individual compound concentrations (ng/mL) from the IADN annual PBDE reporting list in the PBDE IDL working mixture.**

| Compound        | Conc. | Compound                               | Conc. |
|-----------------|-------|--|-------|
| BDE-15          | 0.125 | HBCD                                   | 0.500 |
| <del>pTBX</del> | 0.125 | BDE-140                                | 0.250 |
| PBBZ            | 0.125 | BDE-166                                | 0.078 |
| BDE-17          | 0.125 | BDE-183                                | 0.500 |
| BDE-28          | 0.125 | TBE                                    | 0.250 |
| PBEB            | 0.125 | BDE-181                                | 10.0  |
| HBB             | 0.125 | BEHTBP                                 | 0.250 |
| BDE-49          | 0.250 | syn-DP                                 | 0.250 |
| BDE-47          | 0.250 | BDE-201                                | 0.500 |
| BDE-66          | 0.250 | BDE-197                                | 0.500 |
| BDE-77          | 0.297 | anti-DP                                | 0.250 |
| BDE-100         | 0.250 | BDE-203                                | 0.500 |
| BDE-99          | 0.250 | BDE-208                                | 1.250 |
| EHTBB           | 0.250 | BDE-207                                | 1.250 |
| BDE-118         | 5.0   | BDE-206                                | 1.250 |
| BDE-85          | 0.250 | <sup>13</sup> C <sub>12</sub> -BDE-209 | 0.063 |
| BDE-154         | 0.500 | BDE-209                                | 1.250 |
| BDE-153         | 0.250 | DBDPE                                  | 2.500 |
| BDE-139         | 0.250 |  |       |

### MATRIX SPIKE VIALS

**Instructions:** Mix indicated volumes of the working Matrix Spike Recovery standards for PCBs (see Tables 15), Pesticides (see Tables 32), PAHs (see Tables 51), PBDEs (see Tables 76), OPEs, and β-HCH (see Tables 22) in a 4 mL amber vial as shown in Table 82. The MS vials are used for matrix spike experiments.

**Table 80. Table of MS vials.**

| Working Standard               | Conc., ng/mL | Aliquot, mL | Final Mass, ng |
|--------------------------------|--------------|-------------|----------------|
| PCB MS Recovery Standard       | 90.5         | 1.0         | 90.5           |
| Pesticide MS Recovery Standard | 100          | 0.2         | 20             |
| PAH MS Recovery Standard       | 2,000        | 0.2         | 400            |
| PBDE MS Recovery Standard A    | 25-200       | 0.1         | 2.5-20         |
| PBDE MS Recovery Standard B    | 50-400       | 0.05        | 2.5-20         |
| OPE MS Recovery Standard       | 2-5          | 0.2         | 0.4-1          |
| $\beta$ -HCH Standard          | 200          | 0.1         | 20             |

### PFAS STANDARDS

The following sections list PFAS standards, including the stock solutions and working solutions for calibration, common reference, surrogate, internal, matrix spike, linearity and instrument detection limit (IDL) standards used in the IADN Project. **Due to possible sorption of PFAS to glassware, all the PFAS solutions should be prepared and stored in polypropylene plasticware.**

#### *Stock Solutions*

**Table 81. Table of PFAS individual stock standards and their concentrations.**

| Group        | Abbr.   | Vendor     | Conc., $\mu\text{g/mL}$ |
|--------------|---------|------------|-------------------------|
| <b>PFCAs</b> |         |            |                         |
|              | PFBA    | Wellington | 50                      |
|              | PFPeA   | Wellington | 50                      |
|              | PFHxA   | Wellington | 50                      |
|              | PFHpA   | Wellington | 50                      |
|              | PFOA    | Wellington | 50                      |
|              | PFNA    | Wellington | 50                      |
|              | PFDA    | Wellington | 50                      |
|              | PFUnDA  | Wellington | 50                      |
|              | PFDoDA  | Wellington | 50                      |
|              | PFTTrDA | Wellington | 50                      |
|              | PFTeDA  | Wellington | 50                      |
|              | PFHxDA  | Wellington | 50                      |
| <b>PFSAs</b> |         |            |                         |
|              | PFPrS   | Wellington | 50                      |
|              | PFBS    | Wellington | 50                      |
|              | PFPeS   | Wellington | 50                      |

|                                     |                |            |     |
|-------------------------------------|----------------|------------|-----|
|                                     | <u>PFHxS</u>   | Wellington | 50  |
|                                     | <u>PFHpS</u>   | Wellington | 50  |
|                                     | PFOS           | Wellington | 50  |
|                                     | PFNS           | Wellington | 50  |
|                                     | PFDS           | Wellington | 50  |
| <b>FASAs</b>                        |                |            |     |
|                                     | FBSA           | Wellington | 50  |
| <b>FTSAs</b>                        |                |            |     |
|                                     | 4:2 FTSA       | Wellington | 50  |
|                                     | 6:2 FTSA       | Wellington | 50  |
|                                     | 8:2 FTSA       | Wellington | 50  |
| <b>Internal Standards</b>           |                |            |     |
|                                     | MPFBA          | Wellington | 50  |
|                                     | M8PFOA         | Wellington | 50  |
|                                     | M7PFUnDA       | Wellington | 50  |
|                                     | M3PFHxS        | Wellington | 50  |
|                                     | M8PFOS         | Wellington | 50  |
| <b>Surrogate Recovery Standards</b> |                |            |     |
|                                     | M3PFBA         | Wellington | 50  |
|                                     | <u>MPFHxA</u>  | Wellington | 50  |
|                                     | MPFOA          | Wellington | 50  |
|                                     | <u>MPFUnDA</u> | Wellington | 50  |
|                                     | M2PFTeDA       | Wellington | 50  |
|                                     | M3PFBS         | Wellington | 50  |
|                                     | <u>MPFHxS</u>  | Wellington | 50  |
|                                     | MPFOS          | Wellington | 50  |
|                                     | M2-8:2<br>FTSA | Wellington | 50  |
| <b>PFAS CRS Mixture</b>             |                |            |     |
|                                     | PFCA30PAR      | Wellington | 1.0 |

### **PFAS Intermediate Stock Solutions**

**Instructions:** Dilute the indicated volume of selected individual PFAS standard(s) to 10 mL in LC/MS methanol in a polypropylene volumetric flask as shown in Tables 82-88.

**Table 82. Table of ionic PFAS MIX A intermediate stock solution.**

| <b>Stock</b> | <b>Stock conc.,<br/>µg/mL</b> | <b>Aliquot, mL</b> | <b>Final Vol.,<br/>mL</b> | <b>Final Conc.,<br/>µg/mL</b> |
|--------------|-------------------------------|--------------------|---------------------------|-------------------------------|
| PFBA         | 50                            | 1.0                | 10                        | 5.0                           |
| <u>PFPeA</u> | 50                            | 1.0                | 10                        | 5.0                           |
| <u>PFHxA</u> | 50                            | 1.0                | 10                        | 5.0                           |
| <u>PFHpA</u> | 50                            | 1.0                | 10                        | 5.0                           |
| PFOA         | 50                            | 1.0                | 10                        | 5.0                           |
| PFNA         | 50                            | 1.0                | 10                        | 5.0                           |

Table 83. Table of ionic PFAS MIX B intermediate stock solution.

| Stock          | Stock conc.,<br>µg/mL | Aliquot, mL | Final Vol.,<br>mL | Final Conc.,<br>µg/mL |
|----------------|-----------------------|-------------|-------------------|-----------------------|
| PFDA           | 50                    | 1.0         | 10                | 5.0                   |
| <u>PFUnDA</u>  | 50                    | 1.0         | 10                | 5.0                   |
| <u>PFDoDA</u>  | 50                    | 1.0         | 10                | 5.0                   |
| <u>PFTriDA</u> | 50                    | 1.0         | 10                | 5.0                   |
| <u>PFTeDA</u>  | 50                    | 1.0         | 10                | 5.0                   |
| <u>PFHxDA</u>  | 50                    | 1.0         | 10                | 5.0                   |

Table 84. Table of ionic PFAS MIX C intermediate stock solution.

| Stock        | Stock conc.,<br>µg/mL | Aliquot, mL | Final Vol.,<br>mL | Final Conc.,<br>µg/mL |
|--------------|-----------------------|-------------|-------------------|-----------------------|
| <u>PFPrS</u> | 50                    | 1.0         | 10                | 5.0                   |
| PFBS         | 50                    | 1.0         | 10                | 5.0                   |
| <u>PFPeS</u> | 50                    | 1.0         | 10                | 5.0                   |
| <u>PFHxS</u> | 50                    | 1.0         | 10                | 5.0                   |
| <u>PFHpS</u> | 50                    | 1.0         | 10                | 5.0                   |
| PFOS         | 50                    | 1.0         | 10                | 5.0                   |
| PFNS         | 50                    | 1.0         | 10                | 5.0                   |
| PFDS         | 50                    | 1.0         | 10                | 5.0                   |

Table 85. Table of ionic PFAS MIX D intermediate stock solution.

| Stock    | Stock conc.,<br>µg/mL | Aliquot, mL | Final Vol.,<br>mL | Final Conc.,<br>µg/mL |
|----------|-----------------------|-------------|-------------------|-----------------------|
| 4:2 FTSA | 50                    | 1.0         | 10                | 5.0                   |
| 6:2 FTSA | 50                    | 1.0         | 10                | 5.0                   |
| 8:2 FTSA | 50                    | 1.0         | 10                | 5.0                   |
| FBSA     | 50                    | 1.0         | 10                | 5.0                   |

Table 86. Table of ionic PFAS LC SS Mix 1 intermediate stock solution.

| Stock         | Stock conc.,<br>µg/mL | Aliquot, mL | Final Vol.,<br>mL | Final Conc.,<br>µg/mL |
|---------------|-----------------------|-------------|-------------------|-----------------------|
| M3PFBA        | 50                    | 1.0         | 25                | 2.0                   |
| <u>MPFHxA</u> | 50                    | 1.0         | 25                | 2.0                   |
| MPFOA         | 50                    | 1.0         | 25                | 2.0                   |
| <u>MPFUDa</u> | 50                    | 1.0         | 25                | 2.0                   |
| M2PFTeDA      | 50                    | 1.0         | 25                | 2.0                   |
| M3PFBS        | 50                    | 1.0         | 25                | 2.0                   |
| <u>MPFHxS</u> | 50                    | 1.0         | 25                | 2.0                   |
| MPFOS         | 50                    | 1.0         | 25                | 2.0                   |
| M2-8:2 FTSA   | 50                    | 1.0         | 25                | 2.0                   |

**Table 87. Table of ionic PFAS LC IS Mix intermediate stock solution.**

| Stock   | Stock conc.,<br>µg/mL | Aliquot, mL | Final Vol.,<br>mL | Final Conc.,<br>µg/mL |
|---------|-----------------------|-------------|-------------------|-----------------------|
| MPFBA   | 50                    | 1.0         | 50                | 1.0                   |
| M8PFOA  | 50                    | 1.0         | 50                | 1.0                   |
| M7PFUdA | 50                    | 1.0         | 50                | 1.0                   |
| M3PFHxS | 50                    | 1.0         | 50                | 1.0                   |
| M8PFOS  | 50                    | 1.0         | 50                | 1.0                   |

**Table 88. Table of ionic PFAS CRS Stock solution.**

| Stock     | Stock conc.,<br>µg/mL | Aliquot, mL | Final Vol.,<br>mL | Final Conc.,<br>µg/mL |
|-----------|-----------------------|-------------|-------------------|-----------------------|
| PFCA30PAR | 1.0                   | 1.0         | 10                | 0.1                   |

**PFAS Mixture Solutions.**

**Instructions:** Mix/Dilute the indicated volume of selected individual PFAS standard(s) to 10 mL in LC/MS methanol in a polypropylene volumetric flask as shown in Table 89.

**Table 89. Table of the IADN iPFAS LC Mix 1 Stock.**

| Stock       | Stock conc.,<br>µg/mL | Aliquot, mL | Final Vol.,<br>mL | Final Conc.,<br>µg/mL |
|-------------|-----------------------|-------------|-------------------|-----------------------|
| iPFAS Mix A | 5.0                   | 1.0         | 10                | 0.5                   |
| iPFAS Mix B | 5.0                   | 1.0         | 10                | 0.5                   |
| iPFAS Mix C | 5.0                   | 1.0         | 10                | 0.5                   |
| iPFAS Mix D | 5.0                   | 1.0         | 10                | 0.5                   |

**PFAS Internal Standard**

**Instructions:** Mix/Dilute the indicated volume of selected individual PFAS standard(s) to 10 mL in LC/MS methanol in a polypropylene volumetric flask as shown in Table 90.

**Table 90. Table of the working PFAS internal standard mixture.**

| Stock           | Stock conc.,<br>µg/mL | Aliquot, mL | Final Vol.,<br>mL | Final Conc.,<br>µg/mL |
|-----------------|-----------------------|-------------|-------------------|-----------------------|
| iPFAS LC IS Mix | 1.0                   | 1.0         | 10                | 0.1                   |

**Table 91. Concentration for individual PFAS in the working PFAS internal standard mixture.**

| Compound | Stock conc.,<br>ng/mL | Spike Vol., µL | Spike Mass,<br>ng |
|----------|-----------------------|----------------|-------------------|
| MPFBA    | 100                   | 50             | 5.0               |
| M8PFOA   | 100                   | 50             | 5.0               |
| M7PFUnDA | 100                   | 50             | 5.0               |
| M3PFHxS  | 100                   | 50             | 5.0               |
| M8PFOS   | 100                   | 50             | 5.0               |

**PFAS Surrogate Recovery Standards**

**Instructions:** Mix/Dilute the indicated volume of selected individual PFAS standard(s) to 10 mL in LC/MS methanol in a polypropylene volumetric flask as shown in Table 92.

**Table 92. Table of the working PFAS surrogate standard mixture.**

| Stock       | Stock conc.,<br>µg/mL | Aliquot, mL | Final Vol.,<br>mL | Final Conc.,<br>ng/mL |
|-------------|-----------------------|-------------|-------------------|-----------------------|
| iPFAS LC SS | 2.0                   | 0.2         | 10                | 40                    |

**Table 93. Concentrations for individual PFAS in working iPFAS SS mixture.**

| Compound             | Stock conc.,<br>ng/mL | Spike Vol., µL | Spike Mass,<br>ng |
|----------------------|-----------------------|----------------|-------------------|
| M3PFBA               | 40                    | 50             | 2.0               |
| MPFH <sub>x</sub> A  | 40                    | 50             | 2.0               |
| MPFOA                | 40                    | 50             | 2.0               |
| MPFU <sub>n</sub> DA | 40                    | 50             | 2.0               |
| M2PFTeDA             | 40                    | 50             | 2.0               |
| M3PFBS               | 40                    | 50             | 2.0               |
| MPFH <sub>x</sub> S  | 40                    | 50             | 2.0               |
| MPFOS                | 40                    | 50             | 2.0               |
| M2-8:2FTSA           | 40                    | 50             | 2.0               |

### **PFAS Matrix Spike Recovery Standard**

**Instructions:** Mix/Dilute the indicated volume of selected individual PFAS standard(s) to 10mL in LC/MS grade methanol in a polypropylene volumetric flask as shown in Table 94.

**Table 94. Table of the working PFAS matrix spike recovery standard.**

| Stock          | Stock conc.,<br>µg/mL | Aliquot, mL | Final Vol.,<br>mL | Final Conc.,<br>ng/mL |
|----------------|-----------------------|-------------|-------------------|-----------------------|
| iPFAS LC Mix 1 | 0.5                   | 2.0         | 10                | 100                   |

**Table 95. Concentration for individual PFAS in the working matrix spike recovery standard.**

| Compound            | Stock conc.,<br>ng/mL | Spike Vol., µL | Spike Mass,<br>ng |
|---------------------|-----------------------|----------------|-------------------|
| PFBA                | 100                   | 50             | 5.0               |
| PFPeA               | 100                   | 50             | 5.0               |
| PFH <sub>x</sub> A  | 100                   | 50             | 5.0               |
| PFH <sub>p</sub> A  | 100                   | 50             | 5.0               |
| PFOA                | 100                   | 50             | 5.0               |
| PFNA                | 100                   | 50             | 5.0               |
| PFDA                | 100                   | 50             | 5.0               |
| PFU <sub>n</sub> DA | 100                   | 50             | 5.0               |
| PFDoDA              | 100                   | 50             | 5.0               |
| PFT <sub>r</sub> DA | 100                   | 50             | 5.0               |
| PFTeDA              | 100                   | 50             | 5.0               |
| PFH <sub>x</sub> DA | 100                   | 50             | 5.0               |



|              |     |    |     |
|--------------|-----|----|-----|
| <u>PFPrS</u> | 100 | 50 | 5.0 |
| PFBS         | 100 | 50 | 5.0 |
| <u>PFPeS</u> | 100 | 50 | 5.0 |
| <u>PFHxS</u> | 100 | 50 | 5.0 |
| <u>PFHpS</u> | 100 | 50 | 5.0 |
| PFOS         | 100 | 50 | 5.0 |
| PFNS         | 100 | 50 | 5.0 |
| PFDS         | 100 | 50 | 5.0 |
| FBSA         | 100 | 50 | 5.0 |
| 4:2 FTSA     | 100 | 50 | 5.0 |
| 6:2 FTSA     | 100 | 50 | 5.0 |
| 8:2 FTSA     | 100 | 50 | 5.0 |

**PFAS Common Calibration Standard**

**Instructions:** First, mix the indicated aliquot volumes of selected PFAS mixture solutions together and dilute with LC/MS methanol in 25 mL volumetric flask as shown in Table 96 to make the solution for the preparation of low level (1-4) calibration. Mix indicated aliquot volumes of selected PFAS mixture solutions (Tables 89, 90 and 92) together and dilute with LC/MS methanol in 10 mL volumetric flask as shown in Table 97. Concentrations of individual PFAS compounds in each calibration standard solution are listed in Table 98.

**Table 96. Table of mixture solution for the preparation of low level (1-4) calibration.**

| Stock                   | Stock conc.,<br>µg/mL | Aliquot, mL | Final Vol.,<br>mL | Final Conc.,<br>ng/mL |
|-------------------------|-----------------------|-------------|-------------------|-----------------------|
| <u>iPFAS LC Mix 1</u>   | 0.50                  | 0.125       | 25                | 250                   |
| <u>iPFAS LC Work SS</u> | 0.04                  | 0.250       | 25                | 0.4                   |

**Table 97. Table of PFAS common calibration standard levels.**

| Stock solution                           | Spike Amounts (mL) per Calibration Level |     |     |     |      |     |     |     |
|--|--|-----|-----|-----|------|-----|-----|-----|
|  | 01                                       | 02  | 03  | 04  | 05   | 06  | 07  | 08  |
| <u>iPFAS Low Cal Mix</u>                 | 0.4                                      | 1.0 | 2.0 | 4.0 |      |     |     |     |
| <u>iPFAS LC IS Work (100 ng/mL)</u>      | 0.5                                      | 0.5 | 0.5 | 0.5 | 0.5  | 0.5 | 0.5 | 0.5 |
| <u>iPFA LC Mix 1 &amp; 2 (500 ng/mL)</u> |  |     |     |     | 0.05 | 0.1 | 0.2 | 0.5 |
| <u>iPFAS LC SS Work (40 ng/mL)</u>       |  |     |     |     | 0.1  | 0.2 | 0.5 | 1.0 |
| Total Volume                             | 10                                       | 10  | 10  | 10  | 10   | 10  | 10  | 10  |

**Table 98. Concentration (ng/mL) for individual PFAS compounds in iPFAS IADN common calibration standards.**

| <u>iPFAS Compound</u> | Compound Concentration (ng/mL) per Calibration Level |      |     |     |     |    |    |    |
|-----------------------|--|------|-----|-----|-----|----|----|----|
|                       | 01   | 02   | 03  | 04  | 05  | 06 | 07 | 08 |
| PFBA                  | 0.1  | 0.25 | 0.5 | 1.0 | 2.5 | 5  | 10 | 25 |

|                     |       |      |      |      |     |     |    |    |
|---------------------|-------|------|------|------|-----|-----|----|----|
| <u>PFPeA</u>        | 0.1   | 0.25 | 0.5  | 1.0  | 2.5 | 5   | 10 | 25 |
| <u>PFHxA</u>        | 0.1   | 0.25 | 0.5  | 1.0  | 2.5 | 5   | 10 | 25 |
| <u>PFHpA</u>        | 0.1   | 0.25 | 0.5  | 1.0  | 2.5 | 5   | 10 | 25 |
| PFOA                | 0.1   | 0.25 | 0.5  | 1.0  | 2.5 | 5   | 10 | 25 |
| PFNA                | 0.1   | 0.25 | 0.5  | 1.0  | 2.5 | 5   | 10 | 25 |
| PFDA                | 0.1   | 0.25 | 0.5  | 1.0  | 2.5 | 5   | 10 | 25 |
| <u>PFUnDA</u>       | 0.1   | 0.25 | 0.5  | 1.0  | 2.5 | 5   | 10 | 25 |
| <u>PFDoDA</u>       | 0.1   | 0.25 | 0.5  | 1.0  | 2.5 | 5   | 10 | 25 |
| <u>PFTeDA</u>       | 0.1   | 0.25 | 0.5  | 1.0  | 2.5 | 5   | 10 | 25 |
| <u>PFTeDA</u>       | 0.1   | 0.25 | 0.5  | 1.0  | 2.5 | 5   | 10 | 25 |
| <u>PFHxDA</u>       | 0.1   | 0.25 | 0.5  | 1.0  | 2.5 | 5   | 10 | 25 |
| <u>PFPeS</u>        | 0.1   | 0.25 | 0.5  | 1.0  | 2.5 | 5   | 10 | 25 |
| PFBS                | 0.1   | 0.25 | 0.5  | 1.0  | 2.5 | 5   | 10 | 25 |
| <u>PFPeS</u>        | 0.1   | 0.25 | 0.5  | 1.0  | 2.5 | 5   | 10 | 25 |
| <u>PFHxS</u>        | 0.1   | 0.25 | 0.5  | 1.0  | 2.5 | 5   | 10 | 25 |
| <u>PFHpS</u>        | 0.1   | 0.25 | 0.5  | 1.0  | 2.5 | 5   | 10 | 25 |
| PFOS                | 0.1   | 0.25 | 0.5  | 1.0  | 2.5 | 5   | 10 | 25 |
| PFNS                | 0.1   | 0.25 | 0.5  | 1.0  | 2.5 | 5   | 10 | 25 |
| PFDS                | 0.1   | 0.25 | 0.5  | 1.0  | 2.5 | 5   | 10 | 25 |
| FBSA                | 0.1   | 0.25 | 0.5  | 1.0  | 2.5 | 5   | 10 | 25 |
| 4:2 FTSA            | 0.1   | 0.25 | 0.5  | 1.0  | 2.5 | 5   | 10 | 25 |
| 6:2 FTSA            | 0.1   | 0.25 | 0.5  | 1.0  | 2.5 | 5   | 10 | 25 |
| 8:2 FTSA            | 0.1   | 0.25 | 0.5  | 1.0  | 2.5 | 5   | 10 | 25 |
| M3PFBA (SS)         | 0.016 | 0.04 | 0.08 | 0.16 | 0.4 | 0.8 | 2  | 4  |
| <u>MPFHxA (SS)</u>  | 0.016 | 0.04 | 0.08 | 0.16 | 0.4 | 0.8 | 2  | 4  |
| <u>MPFOA (SS)</u>   | 0.016 | 0.04 | 0.08 | 0.16 | 0.4 | 0.8 | 2  | 4  |
| <u>MPFUnDA (SS)</u> | 0.016 | 0.04 | 0.08 | 0.16 | 0.4 | 0.8 | 2  | 4  |
| M2PFTeDA (SS)       | 0.016 | 0.04 | 0.08 | 0.16 | 0.4 | 0.8 | 2  | 4  |
| M3PFBS (SS)         | 0.016 | 0.04 | 0.08 | 0.16 | 0.4 | 0.8 | 2  | 4  |
| <u>MPFHxS (SS)</u>  | 0.016 | 0.04 | 0.08 | 0.16 | 0.4 | 0.8 | 2  | 4  |
| <u>MPFOS (SS)</u>   | 0.016 | 0.04 | 0.08 | 0.16 | 0.4 | 0.8 | 2  | 4  |
| M2-8:2 FTSA (SS)    | 0.016 | 0.04 | 0.08 | 0.16 | 0.4 | 0.8 | 2  | 4  |
| MPFBA (IS)          | 5     | 5    | 5    | 5    | 5   | 5   | 5  | 5  |
| M8PFOA (IS)         | 5     | 5    | 5    | 5    | 5   | 5   | 5  | 5  |
| M7PFUnDA (IS)       | 5     | 5    | 5    | 5    | 5   | 5   | 5  | 5  |
| M3PFHxS (IS)        | 5     | 5    | 5    | 5    | 5   | 5   | 5  | 5  |
| M8PFOS (IS)         | 5     | 5    | 5    | 5    | 5   | 5   | 5  | 5  |

### **PFAS Common Reference Standard (CRS)**

**Instructions:** Mix indicated aliquot volumes of selected PFAS mixture solutions (see Tables 88, 90 and 92) together and dilute with LC/MS methanol in 10 mL volumetric flask as shown in Tables 99 & 100. Concentrations of individual PFAS compounds in each calibration standard solution are listed in Table 101.

**Table 99. Table of iPFAS High CRS.**

| Stock                   | Stock conc.,<br>µg/mL | Aliquot, mL | Final Vol.,<br>mL | Final Conc.,<br>ng/mL |
|-------------------------|-----------------------|-------------|-------------------|-----------------------|
| <u>iPFAS CRS Stock</u>  | 0.1                   | 0.5         | 10                | 5.0                   |
| <u>iPFAS LC SS Work</u> | 0.04                  | 1.0         | 10                | 4.0                   |
| <u>iPFAS LC IS Work</u> | 0.1                   | 0.5         | 10                | 5.0                   |

**Table 100. Table of iPFAS Low CRS.**

| Stock            | Stock conc.,<br>µg/mL | Aliquot, mL | Final Vol.,<br>mL | Final Conc.,<br>ng/mL |
|------------------|-----------------------|-------------|-------------------|-----------------------|
| iPFAS CRS Stock  | 0.1                   | 0.05        | 10                | 0.5                   |
| iPFAS LC SS Work | 0.04                  | 0.1         | 10                | 0.4                   |
| iPFAS LC IS Work | 0.1                   | 0.5         | 10                | 5.0                   |

**Table 101. Concentrations (ng/mL) for individual PFAS compounds in two iPFAS IADN common reference standards (iPFAS low CRS and iPFAS high CRS).**

| Compound            | iPFAS CRS<br>Low (ng/mL) | iPFAS CRS<br>High (ng/mL) |
|---------------------|--------------------------|---------------------------|
| PFBA                | 0.5                      | 5.0                       |
| PFPeA               | 0.5                      | 5.0                       |
| PFHxA               | 0.5                      | 5.0                       |
| PFHpA               | 0.5                      | 5.0                       |
| PFOA                | 0.5                      | 5.0                       |
| PFNA                | 0.5                      | 5.0                       |
| PFDA                | 0.5                      | 5.0                       |
| PFUnDA              | 0.5                      | 5.0                       |
| PFDoDA              | 0.5                      | 5.0                       |
| PFTeDA              | 0.5                      | 5.0                       |
| PFTeDA              | 0.5                      | 5.0                       |
| PFBS                | 0.5                      | 5.0                       |
| PFPeS               | 0.5                      | 5.0                       |
| PFHxS               | 0.5                      | 5.0                       |
| PFHpS               | 0.5                      | 5.0                       |
| PFOS                | 0.5                      | 5.0                       |
| PFNS                | 0.5                      | 5.0                       |
| PFDS                | 0.5                      | 5.0                       |
| FBSA                | 0.5                      | 5.0                       |
| 4:2 FTSA            | 0.5                      | 5.0                       |
| 6:2 FTSA            | 0.5                      | 5.0                       |
| 8:2 FTSA            | 0.5                      | 5.0                       |
| M3PFBA (SS)         | 0.4                      | 4.0                       |
| MPPHxA (SS)         | 0.4                      | 4.0                       |
| MPFOA (SS)          | 0.4                      | 4.0                       |
| MPFUnDA (SS)        | 0.4                      | 4.0                       |
| M2PFTeDA (SS)       | 0.4                      | 4.0                       |
| M3PFBS (SS)         | 0.4                      | 4.0                       |
| MPPHxS (SS)         | 0.4                      | 4.0                       |
| MPFOS (SS)          | 0.4                      | 4.0                       |
| M2-8:2 FTSA<br>(SS) | 0.4                      | 4.0                       |
| MPFBA (IS)          | 5.0                      | 5.0                       |
| M8PFOA (IS)         | 5.0                      | 5.0                       |
| M7PFUnDA (IS)       | 5.0                      | 5.0                       |
| M3PFHxS (IS)        | 5.0                      | 5.0                       |
| M8PFOS (IS)         | 5.0                      | 5.0                       |

**PFAS Linearity Standards.**

**Instructions:** The linearity for instrumental responses of PFAS can be evaluated using the common calibration standards. The user runs four concentration levels and applies the option "to force" origin".

**PFAS IDL Standard.**

**Instructions:** The IDL can be estimated using the lowest common calibration standard solution, iPFAS IADN Cal Lvl 1, as well as the equation below:

$$IDL = \frac{3 \text{ conc}_{cali}}{SNR_{cali}}$$

where  $\text{conc}_{cali}$  represents the concentration of a PFAS in the lowest common calibration standard solution,  $SNR_{cali}$  represents the corresponding signal-to-noise ratio and the constant "3" indicates that the levels generating peaks with a signal-to-noise ratio greater than 3 are above the IDL, and hence considered detected.