方法827

半挥发性有机化合物

气相色谱/质谱法

sw-846不是分析培训手册。因此，方法程序的编写基于这样一个假设：这些程序将由受过至少化学分析基本原理和主题技术使用方面正式培训的分析员执行。

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| 化合物 | 化学文摘社登记号码一 | 3510 | 3520 | 3540/  3541 | 3550 | 3580 |
| 苊 | 83-32-9 | X | X | X | X | X |
| 苊 | 208-96-8 | X | X | X | X | X |
| 苯乙酮 | 98-86-2 | X | 钕 | 钕 | 钕 | X |
| 2-乙酰氨基芴 | 53-96-3 | X | 钕 | 钕 | 钕 | X |
| 1-乙酰基-2-硫脲 | 591-08-2 | LR | 钕 | 钕 | 钕 | LR |
| 奥尔德林 | 309-00-2 | X | X | X | X | X |
| 2-氨基蒽醌 | 117-79-3 | X | 钕 | 钕 | 钕 | X |
| 氨基偶氮苯 | 60-09-3 | X | 钕 | 钕 | 钕 | X |
| 4-氨基联苯 | 92-67-1 | X | 钕 | 钕 | 钕 | X |
| 3-氨基-9-乙基咔唑 | 132-32-1 | X | X | 钕 | 钕 | 钕 |
| 哌嗪 | 101-05-3 | X | 钕 | 钕 | 钕 | X |
| 苯胺 | 62-53-3 | X | X | 钕 | X | X |
| 邻茴香胺 | 90-04-0 | X | 钕 | 钕 | 钕 | X |
| 蒽 | 120-12-7 | X | X | X | X | X |
| 亚砷酸盐 | 140-57-8 | HS | 钕 | 钕 | 钕 | X |
| 阿罗克罗1016 | 12674-11-2 | X | X | X | X | X |
| 阿罗克罗1221 | 11104-28-2 | X | X | X | X | X |
| 阿罗克罗1232 | 11141-16-5 | X | X | X | X |  |
| 阿罗克罗1242 | 53469-21-9 | X | X | X | X |  |
| 阿罗克罗1248 | 12672-29-6 | X | X | X | X |  |
| 阿罗克罗1254 | 11097-69-1 | X | X | X | X |  |
| 阿罗克罗1260 | 11096-82-5 |  | X | X | X |  |

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此外，除了用于分析方法定义参数（MDP）的所需方法外，SW-846方法旨在作为指导方法，其中包含有关如何执行分析程序或技术的一般信息，实验室可将其用作生成自己详细标准操作的基本起点。技术指导程序（SOP），用于其自身的一般用途或特定的项目应用。本方法中包含的性能数据仅供参考，不得用作实验室认可的绝对质量控制（QC）验收标准。

1.0范围和应用

1.1本方法用于测定多种固体废物基质、土壤、空气取样介质和水样提取物中半挥发性有机化合物的浓度。样品的直接注射可用于有限的应用。以下资源保护和恢复法（RCRA）分析物已通过该方法确定：

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| 甲基偶氮苯甲磷 | 86-50-0 | HS |  |  |  |  |
| 巴班 | 101-27-9 | LR |  |  |  | LR |
| 联苯胺 | 92-87-5 | 人物配对关系 | 人物配对关系 | 人物配对关系 | 人物配对关系 | 人物配对关系 |
| 苯甲酸 | 65-85-0 | X | X | 钕 | X | X |
| 苯并（a）蒽 | 56-55-3 | X | X | X | X | X |
| 苯并（b）荧蒽 | 205-99-2 | X | X | X | X | X |
| 苯并（k）荧蒽 | 207-08-9 | X | X | X | X | X |
| 苯并（g，h，i）亚乙烯 | 191-24-2 | X | X | X | X | X |
| Benzo（A）芘 | 50-32-8 | X | X | X | X | X |
| 对苯醌 | 106-51-4 | OE | 钕 | 钕 | 钕 | X |
| 苄醇 | 100-51-6 | X | X | 钕 | X | X |
| α- BHC | 319-84-6 | X | X | X | X | X |
| β- BHC | 319-85-7 | X | X | X | X | X |
| δ- BHC | 319-86-8 | X | X | X | X | X |
| γ-BHC（林丹） | 58-89-9 | X | X | X | X | X |
| 双（2-氯乙氧基）甲烷 | 111-91-1 | X | X | X | X | X |
| 双（2-氯乙基）醚 | 111-44-4 | X | X | X | X | X |
| 双（2-氯-1-甲基乙基）醚 | 108-60-1 | X | X | X | X | X |
| 邻苯二甲酸二（2-乙基己基）酯 | 117-81-7 | X | X | X | X | X |
| 4-溴苯基苯醚 | 101-55-3 | X | X | X | X | X |
| 溴苯腈 | 1689-84-5 | X | 钕 | 钕 | 钕 | X |
| 邻苯二甲酸丁苄酯 | 85-68-7 | X | X | X | X | X |
| 卡塔福尔 | 2425-06-1 | HS | 钕 | 钕 | 钕 | X |
| 克坦坦 | 133-06-2 | HS | 钕 | 钕 | 钕 | X |
| 西维因 | 63-25-2 | X | 钕 | 钕 | 钕 | X |
| 呋喃丹 | 1563-66-2 | X | 钕 | 钕 | 钕 | X |
| 碳硫磷 | 786-19-6 | X | 钕 | 钕 | 钕 | X |
| 氯丹（NOS） | 57-74-9 | X | X | X | X | X |
| 杀虫磷 | 470-90-6 | X | 钕 | 钕 | 钕 | X |
| 4-氯苯胺 | 106-47-8 | X | 钕 | 钕 | 钕 | X |
| 氯苄酯 | 510-15-6 | X | 钕 | 钕 | 钕 | X |
| 5-氯-2-甲基苯胺 | 95-79-4 | X | 钕 | 钕 | 钕 | X |
| 4-氯-3-甲基苯酚  3-氯甲基吡啶 | 59-50-7 | X | X | X | X | X |
| 盐酸盐 | 6959-48-4 | X | 钕 | 钕 | 钕 | X |
| 1-氯萘 | 90-13-1 | X | X | X | X | X |
| 2-氯萘 | 91-58-7 | X | X | X | X | X |
| 2-氯酚 | 95-57-8 | X | X | X | X | X |
| 4-氯-1,2-苯二胺 | 95-83-0 | X | X | 钕 | 钕 | 钕 |
| 4-氯-1,3-苯二胺 | 5131-60-2 | X | X | 钕 | 钕 | 钕 |
| 4-氯苯基醚 | 7005-72-3 | X | X | X | X | X |
| 菊粉 | 218-01-9 | X | X | X | X | X |
| 敌螨磷 | 56-72-4 | X | 钕 | 钕 | 钕 | X |
| 吡克西丁 | 120-71-8 | X | 钕 | 钕 | 钕 | X |
| 克毒磷 | 7700-17-6 | X | 钕 | 钕 | 钕 | X |
| 2-环己基-4,6-二硝基苯酚 | 131-89-5 | X | 钕 | 钕 | 钕 | LR |
| 4，4’- DDD | 72-54-8 | X | X | X | X |  |
| 4，4’- DDE | 72-55-9 | X | X | X | X |  |
| 4，4′-滴滴涕 | 50-29-3 | X | X | X | X |  |
| 德米顿O | 298-03-3 | HS |  |  |  |  |
| 内吸磷 | 126-75-0 |  |  |  |  |  |

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| 拨号（顺式或反式） | 2303-16-4 | X |  |  |  |  |
| 2,4-二氨基甲苯 | 95-80-7 | 直流光电 |  |  |  |  |
| 二苯并（a，j）吖啶 | 224-42-0 | X | 钕 |  | 钕 |  |
| 二苯并（a，h）蒽 | 53-70-3 | X | X | X | X |  |
| 二苯并呋喃 | 132-64-9 | X | X | 钕 | X |  |
| 二苯并（a，e）芘 | 192-65-4 | 钕 | 钕 | 钕 | 钕 | X |
| 1,2-二溴-3-氯丙烷 | 96-12-8 | X | X | 钕 | 钕 | 钕 |
| 邻苯二甲酸二正丁酯 | 84-74-2 | X | X | X | X | X |
| 二氯酮 | 117-80-6 | OE | 钕 | 钕 | 钕 | X |
| 1,2-二氯苯 | 95-50-1 | X | X | X | X | X |
| 1,3-二氯苯 | 541-73-1 | X | X | X | X | X |
| 1,4-二氯苯 | 106-46-7 | X | X | X | X | X |
| 3,3’-二氯联苯胺 | 91-94-1 | X | X | X | X | X |
| 2,4-二氯苯酚 | 120-83-2 | X | X | X | X | X |
| 2,6-二氯苯酚 | 87-65-0 | X | 钕 | 钕 | 钕 | X |
| 敌敌畏 | 62-73-7 | X | 钕 | 钕 | 钕 | X |
| 双扑磷 | 141-66-2 | X | 钕 | 钕 | 钕 | X |
| 狄氏剂 | 60-57-1 | X | X | X | X | X |
| 邻苯二甲酸二乙酯 | 84-66-2 | X | X | X | X | X |
| 己烯雌酚 | 56-53-1 | 操作系统，操作系统 | 钕 | 钕 | 钕 | X |
| Diethyl硫酸盐 | 64-67-5 | LR | 钕 | 钕 | 钕 | LR |
| 乐果 | 60-51-5 | 他，HS | 钕 | 钕 | 钕 | X |
| 3,3’-二甲氧基联苯胺 | 119-90-4 | X | 钕 | 钕 | 钕 | LR |
| 二甲氨基偶氮苯 | 60-11-7 | X | 钕 | 钕 | 钕 | X |
| 7,12-二甲基苯（a）-蒽 | 57-97-6 | 人物配对关系 | 钕 | 钕 | 钕 | 人物配对关系 |
| 3,3’-二甲基联苯胺 | 119-93-7 | X | 钕 | 钕 | 钕 | X |
| α，α-二甲基苯乙胺 | 122-09-8 | 钕 | 钕 | 钕 | 钕 | X |
| 2,4-二甲基苯酚 | 105-67-9 | X | X | X | X | X |
| 邻苯二甲酸二甲酯 | 131-11-3 | X | X | X | X | X |
| 1,2-二硝基苯 | 528-29-0 | X | 钕 | 钕 | 钕 | X |
| 1,3-二硝基苯 | 99-65-0 | X | 钕 | 钕 | 钕 | X |
| 1,4-二硝基苯 | 100-25-4 | 他 | 钕 | 钕 | 钕 | X |
| 4,6-二硝基-2-甲基苯酚 | 534-52-1 | X | X | X | X | X |
| 2,4-二硝基苯酚 | 51-28-5 | X | X | X | X | X |
| 2,4-二硝基甲苯 | 121-14-2 | X | X | X | X | X |
| 2,6-二硝基甲苯 | 606-20-2 | X | X | X | X | X |
| 迪诺卡 | 39300-45-3 | CP | 钕 | 钕 | 钕 | 人物配对关系 |
| 地乐酚 | 88-85-7 | X | 钕 | 钕 | 钕 | X |
| 二苯胺 | 122-39-4 | X | X | X | X | X |
| 5,5-二苯基海因 | 57-41-0 | X | 钕 | 钕 | 钕 | X |
| 1,2-二苯基肼 | 122-66-7 | X | X | X | X | X |
| 邻苯二甲酸二正辛酯 | 117-84-0 | X | X | X | X | X |
| 双磺酸盐 | 298-04-4 | X | 钕 | 钕 | 钕 | X |
| Endosulfan一世 | 959-98-8 | X | X | X | X | X |
| 硫丹Ⅱ | 33213-65-9 | X | X | X | X | X |
| 硫丹硫酸盐 | 1031-07-8 | X | X | X | X | X |
| 恩德林 | 72-20-8 | X | X | X | X |  |
| 异狄氏剂醛 | 7421-93-4 | X | X | X | X |  |
| 异狄氏剂酮 | 53494-70-5 | X | X |  | X |  |
| EPN | 2104-64-5 | X |  |  |  |  |
| 乙硫磷 | 563-12-2 |  |  |  |  |  |

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| 氨基甲酸乙酯 | 51-79-6 | 直流 |  |  |  |  |
| 甲基磺酸乙酯 | 62-50-0 | X |  |  |  |  |
| 法弗 | 52-85-7 | X | 钕 |  | 钕 |  |
| 芬硫磷 | 115-90-2 | X | 钕 | 钕 | 钕 |  |
| 倍硫磷 | 55-38-9 | X | 钕 | 钕 | 钕 |  |
| 氟氯灵 | 33245-39-5 | X | 钕 | 钕 | 钕 | X |
| 荧蒽 | 206-44-0 | X | X | X | X | X |
| 芴 | 86-73-7 | X | X | X | X | X |
| 2-氟联苯（SURR） | 321-60-8 | X | X | X | X | X |
| 2-氟苯酚（SURR） | 367-12-4 | X | X | X | X | X |
| 七氯 | 76-44-8 | X | X | X | X | X |
| 七氯环氧化物 | 1024-57-3 | X | X | X | X | X |
| 六氯苯 | 118-74-1 | X | X | X | X | X |
| 六氯丁二烯 | 87-68-3 | X | X | X | X | X |
| 六氯环戊二烯 | 77-47-4 | X | X | X | X | X |
| 六氯乙烷 | 67-72-1 | X | X | X | X | X |
| 六氯酚 | 70-30-4 | CP | 钕 | 钕 | 钕 | 人物配对关系 |
| 六氯丙烯 | 1888-71-7 | X | 钕 | 钕 | 钕 | X |
| 六甲基磷酰胺 | 680-31-9 | X | 钕 | 钕 | 钕 | X |
| 对苯二酚 | 123-31-9 | 钕 | 钕 | 钕 | 钕 | X |
| 因德诺（1,2,3-cd）芘 | 193-39-5 | X | X | X | X | X |
| 异狄氏剂 | 465-73-6 | X | 钕 | 钕 | 钕 | X |
| 异佛尔酮 | 78-59-1 | X | X | X | X | X |
| 异黄樟油素 | 120-58-1 | 直流 | 钕 | 钕 | 钕 | X |
| 凯普内 | 143-50-0 | X | 钕 | 钕 | 钕 | X |
| 来虫磷 | 21609-90-5 | X | 钕 | 钕 | 钕 | X |
| 马拉硫磷 | 121-75-5 | HS | 钕 | 钕 | 钕 | X |
| 顺丁烯二酸酐 | 108-31-6 | 他 | 钕 | 钕 | 钕 | X |
| 甲磺酰氯 | 72-33-3 | X | 钕 | 钕 | 钕 | X |
| 甲基吡喃 | 91-80-5 | X | 钕 | 钕 | 钕 | X |
| 甲氧滴滴涕 | 72-43-5 | X | 钕 | 钕 | 钕 | X |
| 3-甲基胆蒽 | 56-49-5 | X | 钕 | 钕 | 钕 | X |
| 4,4’-亚甲基双（2-氯苯胺）4,4’-亚甲基双（N，N-二甲基）- | 101-14-4 | 操作系统，操作系统 | 钕 | 钕 | 钕 | LR |
| 苯胺） | 101-61-1 | X | X | 钕 | 钕 | 钕 |
| 甲基磺酸盐 | 66-27-3 | X | 钕 | 钕 | 钕 | X |
| 2-甲基萘 | 91-57-6 | X | X | 钕 | X | X |
| 甲基对硫磷 | 298-00-0 | X | 钕 | 钕 | 钕 | X |
| 2-甲基苯酚 | 95-48-7 | X | 钕 | 钕 | 钕 | X |
| 3-甲基苯酚 | 108-39-4 | X | 钕 | 钕 | 钕 | X |
| 4-甲基苯酚 | 106-44-5 | X | 钕 | 钕 | 钕 | X |
| 美维磷 | 7786-34-7 | X | 钕 | 钕 | 钕 | X |
| 十六烷酸盐 | 315-18-4 | 他，HS | 钕 | 钕 | 钕 | X |
| 米雷克斯 | 2385-85-5 | X | 钕 | 钕 | 钕 | X |
| 久效磷 | 6923-22-4 | 他 | 钕 | 钕 | 钕 | X |
| 纳贝特 | 300-76-5 | X | 钕 | 钕 | 钕 | X |
| 萘 | 91-20-3 | X | X | X | X |  |
| 1,4-萘醌 | 130-15-4 | X | 钕 | 钕 | 钕 |  |
| 1-萘胺 | 134-32-7 | 操作系统 | 钕 |  | 钕 |  |
| 2-萘胺 | 91-59-8 | X |  |  |  |  |
| 尼古丁 | 54-11-5 | 直流 |  |  |  |  |

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| 5-硝基环烷 | 602-87-9 | X |  |  |  |  |
| 2-硝基苯胺 | 88-74-4 | X | X |  | X |  |
| 3-硝基苯胺 | 99-09-2 | X | X |  | X |  |
| 4-硝基苯胺 | 100-01-6 | X | X | 钕 | X |  |
| 5-硝基邻茴香胺 | 99-59-2 | X | 钕 | 钕 | 钕 |  |
| 硝基苯 | 98-95-3 | X | X | X | X | X |
| 4-硝基联苯 | 92-93-3 | X | 钕 | 钕 | 钕 | X |
| 除草醚 | 1836-75-5 | X | 钕 | 钕 | 钕 | X |
| 2-硝基苯酚 | 88-75-5 | X | X | X | X | X |
| 4-硝基苯酚 | 100-02-7 | X | X | X | X | X |
| 5-硝基邻甲苯胺 | 99-55-8 | X | X | 钕 | 钕 | X |
| 一氧化二氮喹啉 | 56-57-5 | X | 钕 | 钕 | 钕 | X |
| N-亚硝基正丁胺 | 924-16-3 | X | 钕 | 钕 | 钕 | X |
| N-亚硝基二乙胺 | 55-18-5 | X | 钕 | 钕 | 钕 | X |
| N-亚硝基二甲胺 | 62-75-9 | X | X | X | X | X |
| N-硝基二苯胺 | 86-30-6 | X | X | X | X | X |
| N-亚硝基-N-丙胺 | 621-64-7 | X | X | X | X | X |
| N-亚硝基甲基乙胺 | 10595-95-6 | X | 钕 | 钕 | 钕 | X |
| N-亚硝基吗啉 | 59-89-2 | 钕 | 钕 | 钕 | 钕 | X |
| N-亚硝基哌啶 | 100-75-4 | X | 钕 | 钕 | 钕 | X |
| n-亚硝基吡咯烷 | 930-55-2 | X | 钕 | 钕 | 钕 | X |
| 八甲基焦磷酰胺 | 152-16-9 | LR | 钕 | 钕 | 钕 | LR |
| 4,4’-氧二苯胺 | 101-80-4 | X | 钕 | 钕 | 钕 | X |
| 对硫磷 | 56-38-2 | X | X | 钕 | 钕 | X |
| 五氯苯 | 608-93-5 | X | 钕 | 钕 | 钕 | X |
| 五氯硝基苯 | 82-68-8 | X | 钕 | 钕 | 钕 | X |
| 五氯酚 | 87-86-5 | X | X | X | X | X |
| 非那西丁 | 62-44-2 | X | 钕 | 钕 | 钕 | X |
| 菲 | 85-01-8 | X | X | X | X | X |
| 苯巴比妥 | 50-06-6 | X | 钕 | 钕 | 钕 | X |
| 苯酚 | 108-95-2 | 直流 | X | X | X | X |
| 1,4-苯二胺 | 106-50-3 | X | 钕 | 钕 | 钕 | X |
| 甲拌磷 | 298-02-2 | X | 钕 | 钕 | 钕 | X |
| 硫磷 | 2310-17-0 | HS | 钕 | 钕 | 钕 | X |
| 硫磷 | 732-11-6 | HS | 钕 | 钕 | 钕 | X |
| 磷酰胺 | 13171-21-6 | 他 | 钕 | 钕 | 钕 | X |
| 邻苯二甲酸酐 | 85-44-9 | 他 | 钕 | 钕 | 钕 | 人物配对关系 |
| 2-吡啶（2-甲基吡啶） | 109-06-8 | X | X | 钕 | 钕 | 钕 |
| 胡椒基亚砜 | 120-62-7 | X | 钕 | 钕 | 钕 | X |
| 丙胺 | 23950-58-5 | X | 钕 | 钕 | 钕 | X |
| 丙硫脲嘧啶 | 51-52-5 | LR | 钕 | 钕 | 钕 | LR |
| 芘 | 129-00-0 | X | X | X | X | X |
| 间苯二酚 | 108-46-3 | 直流光电 | 钕 | 钕 | 钕 | X |
| 黄樟素 | 94-59-7 | X | 钕 | 钕 | 钕 | X |
| 士的宁 | 57-24-9 | 操作系统，操作系统 | 钕 | 钕 | 钕 | X |
| 硫柳汞 | 95-06-7 | X | 钕 | 钕 | 钕 | X |
| 特布福斯 | 13071-79-9 | X | 钕 | 钕 | 钕 | X |
| 1,2,4,5-四氯苯 | 95-94-3 | X | 钕 | 钕 | 钕 | X |
| 2,3,4,6-四氯苯酚 | 58-90-2 | X | 钕 |  | 钕 | X |
| 四氯乙烯 | 961-11-5 | X |  |  |  | X |
| 二硫代磷酸四乙酯 | 3689-24-5 | X | X |  |  | 钕 |

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|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| 焦磷酸四乙酯 | 107-49-3 | X | 钕 | 钕 | 钕 | X |
| 硫堇嗪 | 297-97-2 | X | 钕 | 钕 | 钕 | X |
| 硫酚（苯硫醇） | 108-98-5 | X | 钕 | 钕 | 钕 | X |
| 甲苯二异氰酸酯 | 584-84-9 | 他 | 钕 | 钕 | 钕 | X |
| 邻甲苯胺 | 95-53-4 | X | 钕 | 钕 | 钕 | X |
| 小精灵 | 8001-35-2 | X | X | X | X | X |
| 1,2,4-三氯苯 | 120-82-1 | X | X | X | X | X |
| 2,4,5-三氯苯酚 | 95-95-4 | X | X | 钕 | X | X |
| 2,4,6-三氯苯酚 | 88-06-2 | X | X | X | X | X |
| 氟乐灵 | 1582-09-8 | X | 钕 | 钕 | 钕 | X |
| 2,4,5-三甲基苯胺 | 137-17-7 | X | 钕 | 钕 | 钕 | X |
| 磷酸三甲酯 | 512-56-1 | 他 | 钕 | 钕 | 钕 | X |
| 1,3,5-三硝基苯 | 99-35-4 | X | 钕 | 钕 | 钕 | X |
| 磷酸三（2,3-二溴丙基）酯 | 126-72-7 | X | 钕 | 钕 | 钕 | LR |
| 三对甲苯磷酸酯 | 78-32-0 | X | 钕 | 钕 | 钕 | X |
| O，O，O-三乙基硫代磷酸酯 | 126-68-1 | X | 钕 | 钕 | 钕 | X |

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 一

化学文摘服务（CAS）注册号

B见秒。1.2其他可接受的制备方法。

C

2007年11月30日，综合风险信息系统（IRIS）将化学名称从双（2-氯异丙基）醚改为双（2-氯-1-甲基乙基）醚（通用名称）。这种化合物也称为2，2’-氧双（1-氯丙烷）（CAS索引名）。关于“修订历史”，请参见第七节的链接，关于该化学品的“同义词”，请参见第八节。网址：http://www.epa.gov/iris/subst/0407.htm

分析物清单的关键

aw=在提取和储存过程中对玻璃器皿壁的吸附

cp=不可重复的色谱性能

dc=不利分布系数

He=在酸性或碱性条件下加速提取过程中的水解

Hs=储存期间的水解

lr=低响应

nd=未确定

oe=萃取过程中的氧化，由基本条件加速

OS=储存期间氧化

X=历史上，通过这种技术可以获得足够的回收率。然而，实际回收率可能因萃取效率、同时分析的组分数量和分析仪器的不同而不同。

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1.2除了上述分析物列表中所列的样品制备方法外，方法3535还描述了一种固相萃取（SPE）程序，可用于从毒性特征浸出程序（TCLP）浸出液中提取半挥发物（性能数据见本方法表16和17）。方法3542描述了通过方法0010（关于替代性能数据，见本方法表10）取样的空气中半挥发性有机化合物的样品制备，方法3545描述了用于固体中半挥发性的自动溶剂萃取（ASE）装置（关于性能数据，见本方法表11），方法3561描述了一种替代物。用于从固体中提取多环芳烃（PAH）的Cal流体萃取（SFE）装置（见表12、13和14

性能数据的方法）和方法3546提供了一种提取程序，该程序使用市售微波设备提取半挥发物，同时使用的溶剂和时间比索格利特提取等程序少（适用性能数据见本方法表18至22）。表格数据仅供参考。

1.3该方法可用于定量大多数中性、酸性和碱性有机化合物，这些有机化合物可溶于二氯甲烷（或其他合适的溶剂，前提是可以生成所需的性能数据），并且能够在无衍生作用的情况下以气相色谱熔融石英毛细管柱的尖峰洗脱。涂上一层极性的硅树脂。这些化合物包括多环芳烃、氯化烃和农药、邻苯二甲酸酯、有机磷酸酯、亚硝胺、卤代醚、醛、醚、酮、苯胺、吡啶、喹啉、芳香族硝基化合物和酚，包括硝基酚。已评价的化合物及其特征离子清单见表1。

在大多数情况下，该方法不适用于多组分分析物（如芳香烃、毒杀芬、氯丹等）的定量，因为这些分析物的灵敏度有限。当用另一种技术鉴定这些分析物时，方法8270可能适用于在提取物浓度允许的情况下确认这些分析物的鉴定。关于多组分分析物（如芳香烃、毒杀芬和氯丹）的校准和定量指南，请参考方法8081和8082。

1.4当用这种方法测定时，下列化合物可能需要特殊处理：

1.4.1联苯胺在溶剂浓缩过程中可能发生氧化损失，其色谱行为较差。

1.4.2在从水基质中提取步骤的碱性条件下，α-bhc、γ-bhc、硫丹I和II以及endrin会分解。如果预计存在这些化合物，应进行中性萃取。

1.4.3六氯环戊二烯在气相色谱仪入口进行热分解，在丙酮溶液中进行化学反应，进行光化学分解。

1.4.4在所述色谱条件下，N-亚硝基二甲胺很难与溶剂分离。

1.4.5 N-硝基二苯胺在气相色谱入口分解，不能与二苯胺分离。因此，可以将这些化合物的n-硝基二苯基胺和二苯胺的组合结果作为组合浓度进行报告。

1.4.6 1,2-二苯基肼即使在室温下也不稳定，很容易转化为偶氮苯。考虑到稳定性问题，用偶氮苯校准1，2-二苯基肼是可以接受的。在这些不良的化合物分离情况下，这些化合物的结果应作为一个组合浓度报告。

1.4.7五氯酚、2,4-二硝基苯酚、4-硝基苯酚、苯甲酸、4,6-二硝基-2-甲基苯酚、4-氯-3-甲基苯酚、2-硝基苯胺、3-硝基苯胺、4-硝基苯胺和苯甲醇的色谱行为不稳定，特别是如果气相色谱仪（GC）系统被高沸点物质污染。

1.4.8吡啶在本方法所列的GC进样口温度下可能表现不佳。降低喷油口温度可能会减少降解量。但是，由于其他分析物的性能可能受到不利影响，分析人员在修改注入口温度时必须谨慎。因此，如果除其他目标分析物外还要测定吡啶，则可能需要进行单独分析。此外，在样品提取物的蒸发浓缩过程中，吡啶可能会丢失。因此，上面列出的许多提取方法可能会产生较低的回收率，除非在浓缩步骤中非常小心。因此，分析人员可能希望考虑使用萃取技术，例如加压流体萃取（方法3545）、微波萃取（方法3546）或超临界流体萃取，这涉及较小的萃取体积，从而减少或消除许多应用中蒸发浓缩技术的需要。

1.4.9甲苯二异氰酸酯在水中快速水解（半衰期小于30分钟）。因此，不应期望从水性基质中回收该化合物。此外，在固体基质中，甲苯二异氰酸酯经常与醇和胺反应生成氨基甲酸乙酯和尿素，因此通常不能在含有这些物质的溶液中共存。

1.4.10此外，当样品制备和/或色谱问题造成限制时，应标记上述列表中的分析物。

1.5测定单个化合物时，该方法的定量下限（LLOQ）约为土壤/沉积物样品660μg/kg（湿重），废物1-200 mg/kg（取决于基质和制备方法），地下水样品10μg/l（见表2）。对于需要稀释以避免检测器饱和的样品提取物，LLOQ将成比例地更高。表2中所列的定量下限仅供参考，并不总是可以实现的。

1.6在使用此方法之前，建议分析员参考总体分析中可能使用的每种类型程序的基本方法（例如，方法3500、3600、5000和8000），以获取有关质量控制程序、质量控制验收标准制定、计算和一般指南的更多信息。分析人员还应查阅手册前面的免责声明和第二章中的信息，以指导在选择方法、仪器、材料、试剂和供应品时的预期灵活性，以及分析人员证明所用技术适用于分析人员的责任。感兴趣的，在感兴趣的矩阵中，以及在关注的层面上。

此外，建议分析人员和数据用户，除非在法规中明确规定，否则使用sw-846方法不是强制性的，以响应联邦测试要求。本方法中包含的信息由环境保护局（EPA或机构）提供，作为分析人员和受管制社区在做出必要判断时使用的指南，以生成满足预期应用的数据质量目标（DQoS）的结果。

1.7本方法的使用仅限于由在使用GC/质谱仪（MS）方面具有适当经验和培训并熟练解释质谱的人员使用或在其监督下使用。每个分析员必须证明用这种方法产生可接受结果的能力。

2.0方法概述

2.1使用适当的样品制备（参考方法3500）和（如有必要）样品清理程序（参考方法3600），通过GC/MS制备样品进行分析。

2.2通过将样品提取物注入配有窄孔熔融石英毛细管柱的GC，将半挥发性化合物引入GC/MS。对GC柱进行温度编程，以分离分析物，然后用连接到GC的MS检测分析物。

2.3从毛细管柱洗脱的分析物通过喷射分离器或直接连接引入MS。目标分析物的鉴定是通过将其质谱与真实标准的电子碰撞（或类似电子碰撞）光谱进行比较来完成的。定量是通过比较主要（定量）离子相对于内标物的响应来完成的，使用适合预期应用的适当校准曲线。

2.4本方法包括替代方法8000中提供的一般建议的具体校准和质量控制步骤。

3个定义

有关与本程序相关的定义，请参阅第一章和制造商说明。

4.0干扰

4.1溶剂、试剂、玻璃器皿和其他样品处理硬件可能会对样品分析产生伪影和/或干扰。必须通过分析方法空白证明所有这些材料在分析条件下不受干扰。可能需要在所有玻璃系统中通过蒸馏对试剂和溶剂进行特殊选择。有关质量控制程序的具体指南，请参阅每种方法；有关玻璃器皿清洁的一般指南，请参阅第四章。关于干扰的讨论，也可参考方法8000。

4.2必须评估所有空白、样品和峰值的原始气相色谱/质谱数据是否存在干扰。确定干扰源是否在样品的制备和/或清理过程中，并采取纠正措施消除问题。

4.3如果按顺序分析高浓度和低浓度样品，则可能会出现残留污染。为了减少携带，样品注射器必须在样品注射之间用溶剂冲洗。当遇到异常浓缩的样品时，应在分析溶剂后检查交叉污染。

5安全

此方法不能解决与其使用相关的所有安全问题。实验室负责维护安全的工作环境和当前的意识文件。

职业安全与健康管理局（OSHA）关于本方法所列化学品安全处理的规定。材料安全数据表（MSDS）的参考文件应提供给参与这些分析的所有人员。

6.0设备和用品

本手册中提及的商品名称或商业产品仅用于说明目的，不构成EPA的认可或使用的独家建议。sw-846方法中引用的产品和仪器设置表示方法开发期间或随后由机构评估时使用的产品和设置。可使用本手册所列以外的玻璃器皿、试剂、供应品、设备和设置，前提是已证明并记录了适合预期应用的方法性能。

本节不列出常用的实验室玻璃器皿（例如烧杯和烧瓶）。

                            6.1气相色谱仪/质谱仪系统

6.1.1气相色谱-一种分析系统，配备一个温度可编程气相色谱，适用于无分流注射和所有所需附件，包括注射器、分析柱和气体。毛细管柱应直接与源头相连。

6.1.2柱-30 m x 0.25 mm ID（或0.32 mm ID）0.25、0.5或1μm膜厚硅酮涂层熔融石英毛细管柱（J&W Scientific DB-5或等效物）。本节中列出的列是开发方法时使用的列。在这个方法中列出这些列并不是为了排除可能开发的其他列的使用。实验室可使用这些柱或其他毛细管柱，前提是实验室记录了适合预期应用的方法性能数据（例如色谱分辨率、分析物分解和灵敏度）。

                                            6.1.3质谱仪

6.1.3.1能够在电子碰撞电离模式下，使用70伏（标称）电子能量，每1秒或更短时间扫描35至500安培。质谱仪必须能够产生十氟三苯基膦（DFTPP）的质谱，该质谱符合第2.1节所述标准。

11.3.1.

6.1.3.2如果离子阱MS能够轴向调制以减少离子分子反应，并且能够产生与EPA/国家标准与技术研究所（NIST）库中的光谱相匹配的电子碰撞类光谱，则可以使用离子阱MS。MS必须能够为DFTPP生成一个质谱，该质谱符合第2.5节所述的标准。11 3.1

6.1.4 GC/MS接口-可以使用任何GC-MS接口，为每种感兴趣的化合物提供可接受的校准点，并达到可接受的调谐性能标准。对于窄口径毛细管柱，界面通常是毛细管直接进入质谱源。

6.1.5数据系统-计算机系统应与MS接口。系统必须允许在机器可读介质上连续采集和存储色谱程序期间获得的所有质谱。计算机应该有能够搜索任何气相色谱/质谱数据文件中特定质量离子的软件，并且能够绘制出此类离子丰度与时间或扫描数的关系。这种类型的图被定义为提取离子电流剖面（EICP）。还应提供软件，允许在指定时间或扫描次数限制之间集成任何EICP中的丰度。还应提供最新版本的EPA/国家标准与技术研究所（NIST）质谱库。

6.1.6保护柱（可选）-（J&W失活熔融二氧化硅，0.25 m m ID x 6 m，或等效物），位于注入口和分析柱之间，连接柱连接器（安捷伦目录号5062-3556或等效物）。

                            6.2注射器-10微升

                            6.3 A级容量瓶-配备磨砂玻璃塞的适当尺寸

                            6.4天平-分析型，重量为0.0001 g

6.5瓶-配备聚四氟乙烯（PTFE）衬里螺帽或压接顶部的玻璃

7.0试剂和标准

7.1所有试验必须使用试剂级化学品。除非另有说明，否则所有试剂均应符合美国化学学会（ACS）分析试剂委员会的规范（若有此类规范）。可以使用其他等级，前提是首先确定试剂的纯度足够高，可以在不降低测定准确度的情况下使用。试剂应储存在玻璃中，以防止污染物从塑料容器中浸出。

7.2无有机试剂水-本方法中提及的水均指无有机试剂水。

                            7.3标准溶液

以下各节介绍有关化合物的原料、中间体和工作标准的制备。本讨论作为一个例子提供，并且可根据预期用途使用其他方法和目标化合物的浓度。关于校准标准制备的更多信息，见方法8000。

7.4储备标准溶液（1000 mg/l）-标准溶液可由纯标准材料制备或作为认证溶液购买。

7.4.1准确称取约0.0100g纯物质制备储备标准溶液。将材料溶解在农药丙酮或其他合适的溶剂中，并在10毫升容量瓶中稀释至一定体积。在分析员方便的情况下，可以使用更大的容量。当化合物纯度被测定为96%或更高时，可不经校正而使用重量来计算储备标准品的浓度。如果由制造商或独立来源认证，则可在任何浓度下使用商业制备的库存标准。

7.4.2将储备标准溶液转移到装有聚四氟乙烯螺帽的瓶子中。储存，避光，温度≤6°C或按照标准制造商的建议。应经常检查储备标准溶液是否有降解或蒸发的迹象，尤其是在从中制备校准标准之前。

7.4.3库存标准溶液必须在一年后或更早更换，如果与质量控制检查样品比较发现问题。

7.4.4建议将亚硝胺化合物放在单独的校准混合物中，而不要与其他校准混合物混合。使用预混合认证标准时，请参考制造商的说明以获取更多指导。

7.4.5与盐酸盐的混合物可能含有盐酸，这可能导致分析困难。使用预混合认证标准时，请参考制造商的说明以获取更多指导。

                            7.5内部标准溶液-推荐的内部标准为：

1,4-二氯苯-d4、萘-d8、亚环烷-d10、菲-d10、亚丁烯-d12和亚丁烯-d12（见表5）。其他化合物可作为内部标准，只要符合SEC标准。113.2得到满足。

7.5.1用少量二硫化碳溶解每种化合物0.200 g。转移到50毫升容量瓶中，用二氯甲烷稀释至一定体积，使最终溶剂约为20%二硫化碳。大多数化合物也可溶于小体积的甲醇、丙酮或甲苯，但不溶于亚乙烯-D12。所得溶液将含有浓度为4000 ng/μl的每种标准品。每一份进行分析的1-ml样品提取物应加入10μl内标溶液，使每种内标物的浓度为40 ng/μl。不使用时，应远离任何光源储存在≤6°C的环境中（建议-10°C）。使用预混合认证溶液时，应根据制造商提供的保存时间和储存温度建议进行储存。

7.5.2如果采用更灵敏的MS以达到更低的定量水平，则可能需要更稀释的内标溶液。内标峰值的面积计数应在中点校准分析中目标分析物面积的50-200%之间。

7.6 GC/MS调谐标准-应制备含有50 ng/μl DFTPP的二氯甲烷溶液。本标准还应含有50 ng/μl的4,4’-ddt、五氯酚和联苯胺，以验证进样口惰性和GC柱性能。如果注入总量小于等于50ng，可使用替代浓度来补偿不同的注入量。不使用时，应远离任何光源存放在≤6°C的环境中。（-

10       建议使用摄氏度）。如果使用更灵敏的MS来达到更低的定量水平，则可能需要更稀释的调谐溶液。使用预混合认证溶液时，应根据制造商提供的保存时间和储存温度建议进行储存。

7.7校准标准-至少应在不同浓度下制备五个校准标准。至少一个校准标准应对应于达到或低于项目DQOS所需的样品浓度。其余标准应与实际样品中发现的浓度范围相对应，但不应超过GC/MS系统的工作范围。在给定浓度下制备的每个标准和/或系列校准标准应包含所有所需的项目特定目标分析物，该方法将报告定量和定量结果。

7.7.1 EPA的目的是将特定分析的所有目标分析物包括在校准标准中。这些目标分析物可能不包括整个分析物列表（第1.1）已经证明了该方法。但是，实验室不应报告校准标准中未包括的目标分析物的定量结果。

7.7.2分析前，每1毫升等分校准标准品应加入10微升内标溶液。所有标准品在不使用时应远离任何光源在≤6°C下储存（建议使用-10°C），并应每年新制备一次，或者如果检查标准表明存在问题，则应尽早制备。必要时，应编制校准验证标准，并将其储存在≤6°C的温度下。使用预混合认证溶液时，应根据制造商记录的保存时间和储存温度建议进行储存。

7.8替代标准-推荐的替代标准为：苯酚-d6、2-氟苯酚、2,4,6-三溴酚、硝基苯-d5、2-氟联苯和对三苯基-d14。关于准备替代溶液的说明，见方法3500。

注释：在含有余氯的样品中，已知苯酚-d6会发生反应，形成未被检测为原始加标替代物的氯化酚化合物。当已知存在残余氯时，应采用第四章中概述的样品保存预防措施，以尽量减少氘化酚或任何其他敏感目标分析物的降解。

7.8-1替代标准检查-确定所有提取、清理和浓缩步骤后的空白提取物应具有什么浓度。将该浓度注入GC/MS，以确定替代标准的回收率。建议在准备新的代替品加液时进行该检查。

注释：方法3561（SFE提取多环芳烃）建议使用溴苯和对四萜苯，以更好地覆盖方法中列出的多环芳烃范围。

7.8.2如果采用更灵敏的MS以达到更低的定量水平，则可能需要更稀释的替代溶液。

7.9基质峰值和实验室控制标准（LCSS）-关于基质峰值标准的制备说明，见方法3500。LCS可采用相同的标准，加标液应与初始校准标准使用相同的来源，以限制标准准确度对制备和分析回收率测定的影响。

7.9-1矩阵穗检查-确定什么浓度应在空白提取物应在所有提取，清理和浓缩步骤。将该浓度注入GC/MS以测定回收率。建议在制备新的基质添加溶液时进行此检查。

7.9.2如果采用更灵敏的MS以达到更低的定量水平，则可能需要更稀释的基质和LCS加标溶液。

7.9.3一些项目可能需要对特定的感兴趣化合物进行加插，因为方法3500中列出的加插化合物不能代表项目所需的感兴趣化合物。当发生这种情况时，基质和LCS加标标准应在甲醇中制备，每种化合物的浓度应适合项目。

7.10溶剂-可使用丙酮、己烷、二氯甲烷、异辛烷、二硫化碳、甲苯和其他适当溶剂。所有溶剂应为杀虫剂质量或同等产品。如有必要，可在使用前对溶剂进行脱气。

8.0   样品收集、保存和储存

8.1   见第四章“有机分析物”的介绍材料。

8.2   将样品萃取物储存在温度小于等于6°C的密封小瓶（例如螺旋盖小瓶或卷边盖小瓶）中，并配备未穿孔的聚四氟乙烯衬里隔膜。

9.0   质量控制

9.1   有关质量保证（QA）和质量控制协议的指南，请参阅第一章。当质量控制指南之间存在不一致时，方法特定的质量控制标准优先于技术特定标准和第一章中给出的标准，技术特定的质量控制标准优先于第一章中的标准。任何涉及分析数据收集的工作都应包括制定结构化和系统化的计划文件，如质量保证项目计划（QAPP）或抽样和分析计划（SAP），将项目目标和规范转化为实施项目和评估玷污。每个实验室应保持一个正式的质量保证计划。实验室还应保存记录，以记录所生成数据的质量。应保存所有数据表和质量控制数据，以供参考或检查。

9.2   具体的测定方法质量控制程序见方法8000。关于质量控制程序，请参考方法3500或5000，以确保各种样品制备技术的正确操作。如果执行提取清理程序，请参考方法3600了解适当的质量控制程序。本方法中提供的任何更具体的质量控制程序将取代方法8000、5000、3500或3600中所述的程序。

9.3   评估GC系统操作所需的质量控制程序见方法8000，包括保留时间窗口评估、校准验证和样品色谱分析。此外，关于下列仪器质量控制要求的讨论可在本方法的参考章节中找到：

                            9.3.1必须调整GC/MS，以满足推荐的DFTPP标准。

在初始校准之前，每12小时进行一次分析。见SECS。11.3.1和11.4.1了解更多详情。

9.3.2必须进行GC/MS系统的初始校准，如第2.3节所述。11.3。此外，在使用第二种源标准（使用不同于校准标准的标准制备）进行标准分析后，应立即验证初始校准曲线。初始校准验证分析的建议验收限值为70-130%。根据所需的特定于项目的DQOS，替代验收限制可能是适当的。对于未通过第二源标准初始校准验证的分析物，不应进行定量样品分析。然而，对于那些不符合标准的分析物，如果理解这些结果，则可以继续进行分析，这些结果可用于筛选目的，并被视为估计值。

9.3.3 GC/MS系统必须满足第2.3节中的校准验证验收标准。11.4。

9.3.4样品组分的相对保留时间（RRT）必须在第2.3节提供的标准组分的RRT窗口内。11.

                            9.4初步熟练程度证明（IDP）

在实施一种方法之前，每个实验室必须进行一次IDP，包括至少四个重复的参考样品，加入到整个样品制备和分析过程中获得的干净基质中。每当仪器或程序发生重大变化时，实验室必须证明通过改变条件仍能获得可接受的精度和偏差。每当新员工接受培训时，必须执行分析师IDP。有关如何完成IDP的更多信息，请参见方法8000。

9.4.1新分析员熟练程度的证明-每个实验室都应该有一个培训计划，证明新分析员能够执行分析员负责的方法或方法的一部分。该证明应证明新分析员能够成功遵守实验室制定的标准操作规程。

9.5      Initially, before processing any samples, the analyst should demonstrate that all parts of the equipment in contact with the sample and reagents are interference-free. This is accomplished through the analysis of a method blank. As a continuing check, each time samples are extracted, cleaned up, and analyzed, a method blank must be prepared and analyzed for the compounds of interest as a safeguard against chronic laboratory

contamination. If a peak is observed within the retention time window of any analyte that would prevent the determination of that analyte, determine the source and eliminate it, if possible, before processing the samples. The blanks should be carried through all stages of sample preparation and analysis. When new reagents or chemicals are received, the lab should monitor the preparation and/or analysis blanks associated with samples for any signs of contamination. It is not necessary to test every new batch of reagents or chemicals prior to sample preparation if the source shows no prior problems. However, if reagents are changed during a preparation batch, separate blanks need to be prepared for each set of reagents.

9.6      Sample QC for preparation and analysis

The laboratory must also have procedures for documenting the effect of the matrix on method performance (precision, accuracy, method sensitivity). At a minimum, this should include the analysis of QC samples including a method blank, a matrix spike, a duplicate, and a LCS in each analytical batch and the addition of surrogates to each field sample and QC sample when surrogates are used. Any method blanks, matrix spike samples, and replicate samples should be subjected to the same analytical procedures (Sec. 11.0) as those used on actual samples.

9.6.1 Documenting the effect of the matrix should include the analysis of at least one matrix spike and one duplicate unspiked sample or one matrix spike/matrix spike duplicate pair. The decision on whether to prepare and analyze duplicate samples or a matrix spike/matrix spike duplicate must be based on knowledge of the samples in the sample batch. If samples are expected to contain target analytes, laboratories may use a matrix spike and a duplicate analysis of an unspiked field sample. If samples are not expected to contain target analytes, then laboratories should use a matrix spike and matrix spike duplicate pair. Consult Method 8000 for information on developing acceptance criteria for the matrix spike and matrix spike duplicate.

9.6.2 An LCS should be included with each analytical batch. The LCS consists of an aliquot of a clean (control) matrix similar to the sample matrix and of the same weight or volume. The LCS is spiked with the same analytes at the same concentrations as the matrix spike, when appropriate. When the results of the matrix spike analysis indicate a potential problem due to the sample matrix itself, the LCS results are used to verify that the laboratory can perform the analysis in a clean matrix. Consult Method 8000 for information on developing acceptance criteria for the LCS.

9.6.3 Also see Method 8000 for the details on carrying out sample QC procedures for preparation and analysis. In-house method performance criteria for evaluating method performance should be developed using the guidance found in Method 8000.

9.6.4 Blanks – Before processing any samples, the analyst should demonstrate through the analysis of a method blank that equipment and reagents are free from contaminants and interferences. If a peak is found in the blank that would prevent the identification or bias the measurement of an analyte, the analyst should determine the source and eliminate it, if possible. As a continuing check, each time a batch of samples is extracted, cleaned up, and analyzed, and when there is a change in reagents, a method blank must be prepared and analyzed for the compounds of interest as a safeguard against chronic laboratory contamination. Method blanks, trip blanks, and other field blanks should be carried through all stages of sample preparation and analysis. At least one method blank or instrument blank must be analyzed on every instrument after calibration standard(s) and prior to the analysis of any samples.

9.6.5 Blanks are generally considered to be acceptable if target analyte concentrations are less than one-half the LLOQ or are less than project-specific requirements. Blanks may contain analyte concentrations greater than acceptance limits if the associated samples in the batch are unaffected (i.e., targets are not present in samples or sample concentrations are ≥10X the blank). Other criteria may be used depending on the needs of the project.

9.6.6 If an analyte of interest is found in a sample in the batch near a concentration confirmed in the blank (refer to Sec. 9.5.2), the presence and or/concentration of that analyte should be considered suspect and may require qualification. Contaminants in the blank should meet most or all of the qualitative identifiers in Section 11.6 to be considered. Samples may require re-extraction and/or reanalysis if the blanks do not meet lab established or project specific criteria. Re-extraction and/or re-analysis is not necessary if the analyte concentration falls well below the action or regulatory limit or if the analyte is deemed not important for the project.

9.6.7 When new reagents or chemicals are received, the lab should monitor the blanks associated with samples for any signs of contamination. It is not necessary to test every new batch of reagents or chemicals prior to sample preparation if the source shows no prior problems. However, if reagents are changed during a preparation batch, separate blanks need to be prepared for each set of reagents.

9.6.8 Method and/or solvent blanks may also be used to check for contamination by carryover from a high-concentration sample into subsequent samples (Sec. 4.2). When analysis of such blanks is not possible, such as when an unattended autosampler is employed, the analyst should carefully review the results for at least the next sample after the high-concentration sample. If analytes in the high-concentration sample are not present in the subsequent sample, then lack of carryover has been demonstrated. If there is evidence that carryover may have occurred, then the affected samples should be reanalyzed.

9.7      Surrogate recoveries

If surrogates are used, the laboratory should evaluate surrogate recovery data from individual samples versus the surrogate control limits developed by the laboratory. See Method 8000 for information on evaluating surrogate data and developing and updating surrogate limits. Procedures for evaluating the recoveries of multiple surrogates and the associated corrective actions should be defined in an approved project plan.

9.8      The experience of the analyst performing gas chromatography/mass spectrometry analyses is invaluable to the success of the methods. Each day that analysis is performed, the calibration verification standard should be evaluated to determine if the chromatographic system is operating properly. Questions that should be asked are: Do the peaks look normal? Is the response obtained comparable to the response from previous calibrations? Careful examination of the standard chromatogram can indicate whether the column is still performing acceptably, the injector is leaking, the injector septum needs replacing, etc. When any changes are made to the system (e.g., the column is changed, a septum is changed), see the guidance in Method 8000 regarding whether recalibration of the system must take place.

9.9      It is recommended that the laboratory adopt additional QA practices for use with this method. The specific practices that are most productive depend upon the needs of the laboratory and the nature of the samples. Whenever possible, the laboratory should analyze standard reference materials and participate in relevant performance evaluation studies.

9.10    Lower Limit of Quantitation (LLOQ)

The LLOQ is the lowest concentration at which the laboratory has demonstrated target analytes can be reliably measured and reported with a certain degree of confidence, which must be ≥ the lowest point in the calibration curve. The laboratory shall establish the LLOQ at concentrations where both quantitative and qualitative requirements can consistently be met (see Sections 9.9 and 11.6). The laboratory shall verify the LLOQ at least annually, and whenever significant changes are made to the preparation and/or analytical procedure, to demonstrate quantitation capability at lower analyte concentration levels. The verification is performed by the extraction and/or analysis of an LCS (or matrix spike) at 0.5-2 times the established LLOQ. Additional LLOQ verifications may be useful on a project-specific basis if a matrix is expected to contain significant interferences at the LLOQ. The verification may be accomplished with either clean control material (e.g., reagent water, solvent blank, Ottawa sand, diatomaceous earth) or a representative sample matrix, free of target compounds. Optimally, the LLOQ should be less than the desired decision level or regulatory action level based on the stated DQOs.

9.10.1 LLOQ Verification – The verification of LLOQs using spiked clean control material represents a best-case scenario because it does not evaluate the potential matrix effects of real-world samples. For the application of LLOQs on a project-specific basis, with established DQOs, a representative matrix-specific LLOQ verification may provide a more reliable estimate of the lower quantitation limit capabilities.

9.10.2 The LLOQ verification (to be performed after the initial calibration) is prepared by spiking a clean control material with the analyte(s) of interest at 0.5-2 times the LLOQ concentration level(s). Alternatively, a representative sample matrix free of targets may be spiked with the analytes of interest at 0.5-2 times the LLOQ concentration levels. The LLOQ check is carried through the same preparation and analytical procedures as environmental samples and other QC samples. It is recommended to analyze the LLOQ verification on every instrument where data is reported; however, at a minimum, the lab should rotate the verification among similar analytical instruments such that all are included within 3 years. Frequently performed analyses, such as 8270D, should have an LLOQ check standard be verified, at minimum, once a year.

9.10.3 Recovery of target analytes in the LLOQ verification should be within established in-house limits or within other such project-specific acceptance limits to demonstrate acceptable method performance at the LLOQ. Until the laboratory has sufficient data to determine acceptance limits, the LCS criteria ± 20% (i.e., lower limit minus 20% and upper limit plus 20%) may be used for the LLOQ acceptance criteria. This practice acknowledges the potential for greater uncertainty at the low end of the calibration curve. Where practical, historically based LLOQ acceptance criteria should be determined once sufficient data points have been acquired.

9.10.4 Reporting concentrations below LLOQ - Concentrations that are below the established LLOQ may still be reported; however, these analytes must be qualified as estimated. The procedure for reporting analytes below the LLOQ should be documented in the laboratory&apos;s SOP or in a project-specific plan. Analytes below the LLOQ that are reported should meet most or all of the qualitative identification requirements in Sec. 11.

10.0 CALIBRATION AND STANDARDIZATION

See Sec 11.3 for information on calibration and standardization.

11.0       PROCEDURE

11.1       Sample preparation

11.1.1 Samples are normally prepared by one of the following methods prior to gas chromatography/mass spectrometry analysis.

|  |  |
| --- | --- |
| Matrix | Methods |
| Air (particulates and sorbent resin) | 3542 |
| Water (including TCLP leachates) | 3510, 3520, 3535 |
| Soil/sediment | 3540, 3541, 3545, 3546, 3550, 3560, 3561 |
| Waste | 3540, 3541, 3545, 3546, 3550, 3560, 3561,  3580 |

11.1.2 In very limited applications, direct injection of the sample into the GC/MS system with a 10-µL syringe may be appropriate. The quantitation limit is very high (approximately 10,000 µg/L) when this procedure is used. Therefore, it is only appropriate where concentrations in excess of 10,000 µg/L are expected.

11.2 Extract cleanup - Cleanup procedures may not be necessary for a relatively clean sample matrix, but most extracts from environmental and waste samples will require additional preparation before analysis. The specific cleanup procedure used will depend on the nature of the sample to be analyzed and the DQOs for the measurements. General guidance for sample extract cleanup is provided in this section and in Method 3600.

Extracts may be cleaned up by any of the following methods prior to gas chromatography/mass spectrometry analysis.

|  |  |
| --- | --- |
| Analytes of Interest | Methods |
| Aniline and aniline derivatives | 3620 |
| Phenols | 3630, 3640, 8041a |
| Phthalate esters | 3610, 3620, 3640 |
| Nitrosamines | 3610, 3620, 3640 |
| Organochlorine pesticides | 3610, 3620, 3630, 3640, 3660 |
| PCBs | 3620, 3630, 3660, 3665 |
| Nitroaromatics and cyclic ketones | 3620, 3640 |
| PAHs | 3611, 3630, 3640 |
| Haloethers | 3620, 3640 |
| Chlorinated hydrocarbons | 3620, 3640 |
| Organophosphorus pesticides | 3620 |
| Petroleum waste  All base, neutral, and acid | 3611, 3650 |
| Priority pollutants | 3640 |

a Method 8041 includes a derivatization technique and a GC/electron capture detector (ECD) analysis, if interferences are encountered on GC/flame ionization detector (FID).

                            11.3 Initial calibration

Establish the GC/MS operating conditions, using the following recommendations as guidance.

|  |  |  |  |
| --- | --- | --- | --- |
| Mass range: | | 35-500 amu |  |
| Scan time: | | ≤1 sec/scan |  |
| Initial temperature: | | 40 °C, hold for 4 min |  |
| Temperature program: | | 40-320 °C at 10 °C/min |  |
| Final temperature: | | 320 °C, hold until 2 min after benzo[g,h,i]perylene elutes |  |
| Injector temperature: | | 250-300 °C |  |
| Transfer line temperature: | | 250-300 °C |  |
|  | Source temperature: | According to manufacturer&apos;s specifications | |
|  | Injector: | Grob-type, splitless | |
|  | Injection volume: | 1-2 µL | |
|  | Carrier gas: | Hydrogen at 50 cm/sec or helium at 30 cm/sec | |
|  | Ion trap only: | Set axial modulation, manifold temperature, and emission current to manufacturer&apos;s recommendations | |
|  |  |  |  |

Split injection is allowed if the sensitivity of the MS is sufficient.

11.3.1 The GC/MS system must be hardware-tuned such that injecting 50 ng or less of DFTPP meets the manufacturer&apos;s specified acceptance criteria or as listed in Table 3. The tuning criteria as outlined in Table 3 were developed using quadrupole MS instrumentation and it is recognized that other tuning criteria may be more effective depending on the type of instrumentation (e.g., Time-of-Flight, Ion Trap, etc.). In these cases it would be appropriate to follow the manufacturer&apos;s tuning instructions or some other consistent tuning criteria. However, no matter which tuning criteria is selected, the system calibration must not begin until the tuning acceptance criteria are met with the sample analyses performed under the same conditions as the calibration standards.

11.3.1.1 In the absence of specific recommendations on how to acquire the mass spectrum of DFTPP from the instrument manufacturer, the following approach should be used: Three scans (the peak apex scan and the scans immediately preceding and following the apex) are acquired and averaged. Background subtraction is required, and must be accomplished using a single scan acquired within 20 scans of the elution of DFTPP. The background subtraction should be designed only to eliminate column bleed or instrument background ions. Do not subtract part of the DFTPP peak or any other discrete peak that does not coelute with DFTPP.

11.3.1.2 Use the DFTPP mass intensity criteria in the manufacturer&apos;s instructions as primary tuning acceptance criteria or those in Table 3 as default tuning acceptance criteria if the primary tuning criteria are not available. Alternatively, other documented tuning criteria may be used (e.g., Contract Laboratory Program (CLP) or Method 625), provided that method performance is not adversely affected. The analyst is always free to choose criteria that are tighter than those included in this method or to use other documented criteria provided they are used consistently throughout the initial calibration, calibration verification, and sample analyses.

NOTE: All subsequent standards, samples, matrix spikes/matrix spike duplicates, and blanks associated with a DFTPP analysis must use identical MS instrument conditions.

11.3.1.3 The GC/MS tuning standard solution should also be used to assess the GC column performance and injection port inertness. Degradation of dichlorodiphenyltrichloroethane (DDT) to dichlorodiphenyldichloroethylene (DDE) and dichlorodiphenyldichloroethane (DDD) should not exceed 20%. (See Method 8081 for the percent breakdown calculation.) Benzidine and pentachlorophenol should be present at their normal responses, and should not exceed a tailing factor of 2 given by the following equation:

                                                                                                                                      BC

Tailing Factor = D:\document\convert_tasks\transweb\1464945_1477166\1464945.pdf.files\image005.gifAB

where the peak is defined as follows: AC is the width at 10% height; DE is the height of peak and B is the height at 10% of DE. This equation compares the width of the back half of the peak to the width of the front half of the peak at 10% of the height. (See Figure 1 for an example tailing factor calculation.)

11.3.1.4 If degradation is excessive and/or poor chromatography is noted, the injection port may require cleaning. It may also be necessary to cut off the first 6 to 12 in. of the capillary column to remove high boiling contaminants in the column. The use of a guard column (Sec. 6.1.6) between the injection port and the analytical column may help prolong analytical column performance life.

11.3.2 The internal standards selected in Sec. 7.5 should permit most of the components of interest in a chromatogram to have retention times of 0.80-1.20 relative to one of the internal standards. Use the base peak ion from the specific internal standard as the primary ion for quantitation (see Table 1). If interferences are noted, use the next most intense ion as the quantitation ion (e.g., for 1,4-dichlorobenzen-d4, use m/z 150 for quantitation).

11.3.3 Analyze a consistent volume (typically 1-2 µL) of each calibration standard (i.e., containing the compounds for quantitation and the appropriate surrogates and internal standards) and tabulate the area of the primary ion against concentration for each target analyte (as indicated in Table 1). A set of at least five calibration standards is necessary (see Sec. 7.7 and Method 8000). Alternate injection volumes may be used if the applicable QC requirements for using this method are met. The injection volume must be the same for all standards and sample extracts. Figure 2 shows a chromatogram of a calibration standard containing base/neutral and acid analytes.

                                11.3.4 Initial calibration calculations

Calculate response factors (RFs) for each target analyte relative to one of the internal standards (see Table 4) as follows:

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                                                                                        RF = ×Cis

    Ais ×Cs where:

As = Peak area (or height) of the analyte or surrogate Ais = Peak area (or height) of the internal standard

                                Cs = Concentration of the analyte or surrogate, in µg/L

                                Cis = Concentration of the internal standard, in µg/L

11.3.4.1 Calculate the mean RF and the relative standard deviation (RSD) of the RFs for each target analyte using the following equations. The RSD should be less than or equal to 20% for each target analyte. It is also recommended that a minimum RF for the most common target analytes, as noted in Table 4, be demonstrated for each individual calibration level as a means to ensure that these compounds are behaving as expected. In addition, meeting the minimum RF criteria for the lowest calibration standard is critical in establishing and demonstrating the desired sensitivity. Due to the large number of compounds that may be analyzed by this method, some compounds will fail to meet these criteria. For these occasions, it is acknowledged that the failing compounds may not be critical to the specific project and therefore they may be used as qualified data or estimated values for screening purposes. The analyst should also strive to place more emphasis on meeting the calibration criteria for those compounds that

are critical project compounds, rather than meeting the criteria for those less important compounds.

                                                                    n

                                                                  ∑RFi

D:\document\convert_tasks\transweb\1464945_1477166\1464945.pdf.files\image008.gif    mean RF = RF = SD= ni=1

SD

                                                                                        RSD=D:\document\convert_tasks\transweb\1464945_1477166\1464945.pdf.files\image009.gif×100

RF where:

                                RFi = RF for each of the calibration standards

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RF = mean RF for each compound from the initial calibration n = Number of calibration standards, e.g., 5

                                SD = Standard deviation

11.3.4.2 If more than 10% of the compounds included with the initial calibration exceed the 20% RSD limit and do not meet the minimum correlation coefficient (0.99) for alternative curve fits, then the chromatographic system is considered too reactive for analysis to begin. Clean or replace the injector liner and/or capillary column, then repeat the calibration procedure beginning with Sec.

11.3.

11.3.5 Evaluation of retention times - The RRT of each target analyte in each calibration standard should agree within 0.06 RRT units. Late-eluting target analytes usually have much better agreement.

RRT = Retention time of the analyte

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                                                                     Retention time of the internal standard

11.3.6 Linearity of target analytes - If the RSD of any target analyte is 20% or less, then the relative RF is assumed to be constant over the calibration range, and the average relative RF may be used for quantitation (Sec. 11.7.2).

                11.3.6.1 If the RSD of any target analyte is greater than 20%, refer to

Method 8000 for additional calibration options. One of the options must be applied to GC/MS calibration in this situation, or a new initial calibration must be

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performed. The RF should not be used for compounds that have an RSD greater than 20% unless the concentration is reported as estimated.

                11.3.6.2 When the RSD exceeds 20%, the plotting and visual

inspection of a calibration curve can be a useful diagnostic tool. The inspection may indicate analytical problems, including errors in standard preparation, the presence of active sites in the chromatographic system, analytes that exhibit poor chromatographic behavior, etc.

                11.3.6.3 Due to the large number of compounds that may be analyzed

by this method, some compounds may fail to meet either the 20% RSD, minimum correlation coefficient criteria (0.99), or the acceptance criteria for alternative calibration procedures in Method 8000. Any calibration method described in Method 8000 may be used, but it should be used consistently. It is considered inappropriate once the calibration analyses are completed to select an alternative calibration procedure in order to pass the recommended criteria on a case-by-case basis. If compounds fail to meet these criteria, the associate concentrations may

still be determined but they must be reported as estimated. In order to report nondetects, it must be demonstrated that there is adequate sensitivity to detect the failed compounds at the applicable lower quantitation limit.

11.4 GC/MS calibration verification - Calibration verification consists of three steps that are performed at the beginning of each twelve hour analytical shift.

11.4.1 Prior to the analysis of samples or calibration standards, inject 50 ng or less of the DFTPP standard into the GC/MS system. The resultant mass spectrum for DFTPP must meet the criteria as outlined in Sec. 11.3.1 before sample analysis begins. These criteria must be demonstrated each twelve hour shift during which samples are analyzed.

11.4.2 The initial calibration function for each target analyte should be checked immediately after the first occurrence in the region of the middle of the calibration range with a standard from a source different from that used for the initial calibration. The value determined from the second source check should be within 30% of the expected concentration. An alternative recovery limit may be appropriate based on the desired project-specific DQOs. Quantitative sample analyses should not proceed for those analytes that fail the second source standard initial calibration verification. However, analyses may continue for those analytes that fail the criteria with an understanding that these results could be used for screening purposes and would be considered estimated values.

11.4.3 The initial calibration (Sec. 11.3) for each compound of interest should be verified once every twelve hours prior to sample analysis, using the introduction technique and conditions used for samples. This is accomplished by analyzing a calibration standard (containing all the compounds for quantitation) at a concentration either near the midpoint concentration for the calibrating range of the GC/MS or near the action level for the project. The results must be compared against the most recent initial calibration curve and should meet the verification acceptance criteria provided in Secs. 11.4.5 through 11.4.7.

NOTE: The DFTPP and calibration verification standard may be combined into a single standard as long as both tuning and calibration verification acceptance criteria for the project can be met without interferences.

                11.4.4 A method blank should be analyzed prior to sample analyses in order to

ensure that the total system (i.e., introduction device, transfer lines and GC/MS system) is free of contaminants. If the method blank indicates contamination, then it may be appropriate to analyze a solvent blank to demonstrate that the contamination is not a result of carryover from standards or samples. See Method 8000 for information regarding method blank performance criteria.

                11.4.5 Calibration verification standard criteria

11.4.5.1 Each of the most common target analytes in the calibration verification standard should meet the minimum RFs as noted in Table 4. This criterion is particularly important when the common target analytes are also critical project-required compounds. This is the same check that is applied during the initial calibration.

                11.4.5.2 If the minimum RFs are not met, the system should be

evaluated, and corrective action should be taken before sample analysis begins.

Possible problems include standard mixture degradation, injection port inlet contamination, contamination at the front end of the analytical column, and active sites in the column or chromatographic system.

                11.4.5.3 All target compounds of interest must be evaluated using a

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20% criterion. Use percent difference when performing the RF model calibration. Use percent drift when calibrating using a regression fit model. Refer to Method 8000 for guidance on calculating percent difference and drift.

11.4.5.4 If the percent difference or percent drift for a compound is less than or equal to 20%, then the initial calibration for that compound is assumed to be valid. Due to the large numbers of compounds that may be analyzed by this method, it is expected that some compounds will fail to meet the criterion. If the criterion is not met (i.e., greater than 20% difference or drift) for more than 20% of the compounds included in the initial calibration, then corrective action must be taken prior to the analysis of samples. In cases where compounds fail, they may still be reported as non-detects if it can be demonstrated that there was adequate sensitivity to detect the compound at the applicable quantitation limit. For situations when the failed compound is present, the concentrations must be reported as estimated values.

11.4.5.5 Problems similar to those listed under initial calibration could affect the ability to pass the calibration verification standard analysis. If the problem cannot be corrected by other measures, a new initial calibration must be generated. The calibration verification criteria must be met before sample analysis begins.

                11.4.5.6 The method of linear regression analysis has the potential for

a significant bias to the lower portion of a calibration curve, while the relative percent difference and quadratic methods of calibration do not have this potential bias. When calculating the calibration curves using the linear regression model, a minimum quantitation check on the viability of the lowest calibration point should be performed by re-fitting the response from the low concentration calibration standard back into the curve (see Method 8000 for additional details). It is not necessary to re-analyze a low concentration standard; rather the data system can recalculate the concentrations as if it were an unknown sample. The recalculated concentration of the low calibration point should be within ± 30% of the standard&apos;s true concentration. Other recovery criteria may be applicable depending on the project&apos;s DQOs and for those situations the minimum quantitation check criteria should be outlined in a laboratory SOP, or a project-specific QAPP. Analytes which do not meet the minimum quantitation calibration re-fitting criteria should be considered "out of control" and corrective action such as redefining the LLOQ and/or reporting those "out of control" target analytes as estimated when the concentration is at or near the lowest calibration point may be appropriate.

11.4.6 Internal standard retention time - The retention times of the internal standards in the calibration verification standard must be evaluated immediately after or during data acquisition. If the absolute retention time for any internal standard changes by more than 30 sec from that in the mid-point standard level of the most recent initial calibration sequence, then the chromatographic system must be inspected for malfunctions and corrections must be made, as required. When corrections are made, reanalysis of samples analyzed while the system was malfunctioning is required.

11.4.7 Internal standard response - If the EICP area for any of the internal standards in the calibration verification standard changes by a factor of two (−50% to +100%) from that in the mid-point standard level of the most recent initial calibration sequence, the MS must be inspected for malfunctions and corrections must be made, as appropriate. When corrections are made, reanalysis of samples analyzed while the system was malfunctioning is required.

                11.5 Gas chromatography/mass spectrometry analysis of samples

11.5.1 It is highly recommended that sample extracts be screened on a GC/FID or GC/PID using the same type of capillary column used in the GC/MS system. This will minimize contamination of the GC/MS system from unexpectedly high concentrations of organic compounds.

11.5.2 Allow the sample extract to warm to room temperature. Just prior to analysis, add 10 µL of the internal standard solution to the 1 mL of concentrated sample extract obtained from sample preparation.

11.5.3 Inject an aliquot of the sample extract into the GC/MS system, using the same operating conditions that were used for the calibration (Sec. 11.3). The volume to be injected should include an appropriate concentration that is within the calibration range of base/neutral and acid surrogates using the surrogate solution as noted in Sec. 7.8. The injection volume must be the same volume that was used for the calibration standards.

11.5.4 If the response for any quantitation ion exceeds the initial calibration range of the GC/MS system, the sample extract must be diluted and reanalyzed. Additional internal standard solution must be added to the diluted extract to maintain the same concentration as in the calibration standards (usually 40 ng/µL, or other concentrations as appropriate, if a more sensitive GC/MS system is being used). Secondary ion quantitation should be used only when there are sample interferences with the primary ion.

NOTE: It may be a useful diagnostic tool to monitor internal standard retention times in all samples, spikes, blanks, and standards to effectively check drifting, method performance, poor injection execution, and anticipate the need for system inspection and/or maintenance. Internal standard responses (area counts) should be monitored in all samples, spikes and blanks for similar reasons. If the EICP area for any of the internal standards in samples, spikes, and blanks changes by a factor of two (−50% to +100%) from the areas determined in the continuing calibration analyzed that day, corrective action should be taken. The samples, spikes, or blanks should be reanalyzed or the data should be qualified.

                11.5.4.1 When ions from a compound in the sample saturate the

detector, this analysis should be followed by the analysis of an instrument blank consisting of clean solvent. If the blank analysis is not free of interferences, then the system must be decontaminated. Sample analysis may not resume until the blank analysis is demonstrated to be free of interferences. Contamination from one sample to the next on the instrument usually takes place in the syringe. If adequate syringe washers are employed, then carryover from high concentration samples can usually be avoided.

                11.5.4.2 All dilutions should keep the response of the major

constituents (previously saturated peaks) in the upper half of the linear range of the curve.

11.5.5 The use of a selected ion monitoring (SIM) technique is acceptable for applications requiring quantitation limits below the normal range of electron impact mass spectrometry. However, SIM may provide a lesser degree of confidence in the compound identification, since less mass spectral information is available. Using the primary ion for quantitation and the secondary ions for confirmation, set up the collection groups based on their retention times. The selected ions are nominal ions and most compounds have small mass defect, usually less than 0.2 amu, in their spectra. These mass defects should be used in the acquisition table. The dwell time may be automatically calculated by the laboratory&apos;s GC/MS software or manually calculated using the following formula. The total scan time should be less than 1,000 msec and produce at least 5 to 10 scans per chromatographic peak. The start and stop times for the SIM groups are determined from the full scan analysis using the formula below:

                                                                                                 Scan Time (msec)

                                Dwell Time for the Group = D:\document\convert_tasks\transweb\1464945_1477166\1464945.pdf.files\image012.gifTotal Ions in the Group

Additional guidance for performing SIM analyses, in particular for PAHs and phenol target analyte compounds, can be found in the most recent CLP semivolatile organic methods statement of work (SOW). See the SIM sections from the following CLP SOW for further details: EPA CLP Organics SOW (Reference 14).

                11.6 Analyte identification

11.6.1 The qualitative identification of compounds determined by this method is based on retention time and on comparison of the sample mass spectrum, after background correction, with characteristic ions in a reference mass spectrum. The reference mass spectrum must be generated by the laboratory using the conditions of this method. The characteristic ions from the reference mass spectrum are defined as the three ions of greatest relative intensity, or any ions over 30% relative intensity, if less than three such ions occur in the reference spectrum. Compounds are identified when the following criteria are met.

11.6.1.1 The intensities of the characteristic ions of a compound must maximize in the same scan or within one scan of each other. Selection of a peak by a data system target compound search routine where the search is based on the presence of a target chromatographic peak containing ions specific for the target compound at a compound-specific retention time will be accepted as meeting this criterion.

11.6.1.2 The RRT of the sample component is within ± 0.06 RRT units of the RRT of the standard component.

                11.6.1.3 The relative intensities of the characteristic ions agree within

30% of the relative intensities of these ions in the reference spectrum. For example, an ion with an abundance of 50% in the reference spectrum, the corresponding abundance in a sample spectrum can range between 20% and 80%. Use professional judgment in interpretation where interferences are observed.

11.6.1.4 Structural isomers that produce very similar mass spectra should be identified as individual isomers if they have sufficiently different gas chromatographic retention times. Sufficient gas chromatographic resolution is achieved if the height of the valley between two isomer peaks is less than 50% of

the average of the two peak heights. Otherwise, structural isomers are identified as isomeric pairs. The resolution should be verified on the mid-point concentration of the initial calibration as well as the laboratory designated continuing calibration verification level if closely eluting isomers are to be reported (e.g., benzo(b)fluoranthene and benzo(k)fluoranthene).

11.6.1.5 Identification is hampered when sample components are not resolved chromatographically and produce mass spectra containing ions contributed by more than one analyte. When gas chromatographic peaks obviously represent more than one sample component (i.e., a broadened peak with shoulder(s) or a valley between two or more maxima), appropriate selection of analyte spectra and background spectra is important.

                11.6.1.6 Examination of extracted ion current profiles of appropriate

ions can aid in the selection of spectra and in qualitative identification of compounds. When analytes co-elute (i.e., only one chromatographic peak is apparent), the identification criteria may be met, but each analyte spectrum will contain extraneous ions contributed by the co-eluting compound.

11.6.2 For samples containing components not associated with the calibration

standards, a library search may be made for the purpose of tentative identification. The necessity to perform this type of identification will be determined by the purpose of the analyses being conducted. Data system library search routines should not use normalization routines that would misrepresent the library or unknown spectra when compared to each other.

For example, the RCRA permit or waste delisting requirements may require the reporting of non-target analytes. Only after visual comparison of sample spectra with the nearest library searches may the analyst assign a tentative identification. Guidelines for tentative identification are:

(1) Relative intensities of major ions in the reference spectrum (i.e., ions > 10% of the most abundant ion) should be present in the sample spectrum.

(2) The relative intensities of the major ions should agree within ± 30%. For example, an ion with an abundance of 50% in the standard spectrum must have a corresponding sample ion abundance between 20 and 80%.

(3) Molecular ions present in the reference spectrum should be present in the sample spectrum.

(4) Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of co-eluting compounds.

(5) Ions present in the reference spectrum but not in the sample spectrum should be reviewed for possible subtraction from the sample spectrum because of background contamination or co-eluting peaks. Data system library reduction programs can sometimes create these discrepancies.

                            11.7 Quantitation

11.7.1 Once a target compound has been identified, the quantitation of that compound will be based on the integrated abundance of the primary characteristic ion from the EICP.

11.7.1.1       It is highly recommended to use the integration produced by

the software if the integration is correct because the software should produce more consistent integrations than an analyst will manually. However, manual integrations may be necessary when the software does not produce proper integrations because baseline selection is improper; the correct peak is missed; a coelution is integrated; the peak is partially integrated; etc. The analyst is responsible for ensuring that the integration is correct whether performed by the software or done manually.

11.7.1.2       Manual integrations should not be substituted for proper maintenance of the instrument or setup of the method (e.g., retention time updates, integration parameter files, etc.). The analyst should seek to minimize manual integration by properly maintaining the instrument, updating retention times, and configuring peak integration parameters.

11.7.2       If the RSD of a compound&apos;s RF is 20% or less, then the concentration in

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the extract may be determined using the RF from initial calibration data (Sec. 11.3.4). See Method 8000 for the equations describing internal standard calibration and either linear or non-linear calibrations.

11.7.3       Where applicable, the concentration of any non-target analytes identified in the sample (Sec. 11.6.2) should be estimated. The same formula as in Sec. 11.3.4 should be used with the following modifications: The areas Ax and Ais should be from the total ion chromatograms, and the RF for the compound should be assumed to be 1.

11.7.4       The resulting concentration should be reported indicating that the value is an estimate. Use the nearest internal standard free of interferences.

11.7.5       Quantitation of multicomponent compounds (e.g., toxaphene, Aroclors, etc.) is beyond the scope of Method 8270. Normally, quantitation is performed using a GC/ECD, for example by Methods 8081 or 8082. However, this method (8270) may be used to confirm the identification of these compounds, when the concentrations are at least 10 ng/µL in the concentrated sample extract.

11.7.6       Quantitation of multicomponent parameters such as diesel range organics (DROs) and total petroleum hydrocarbons (TPH) using the Method 8270 recommended internal standard quantitation technique is beyond the scope of this method. Typically, analyses for these parameters are performed using GC/FID or GC with a MS detector capability that is available with Method 8015.

11.7.7       Structural isomers that produce very similar mass spectra should be

quantitated as individual isomers if they have sufficiently different gas chromatographic retention times. Sufficient gas chromatographic resolution is achieved if the height of the valley between two isomer peaks is less than 50% of the average of the two peak heights. Otherwise, structural isomers are identified as isomeric pairs. The resolution should be verified on the mid-point concentration of the initial calibration as well as the laboratory designated continuing calibration verification level if closely eluting isomers are to be reported (e.g., benzo(b)fluoranthene and benzo(k)fluoranthene).

12.0 DATA ANALYSIS AND CALCULATIONS

                See Sec. 11.7 and Method 8000 for information on data analysis and calculations.

13.0   METHOD PERFORMANCE

13.1   Performance data and related information are provided in SW-846 methods only as examples and guidance. The data do not represent required performance criteria for users of the methods. Instead, performance criteria should be developed on a project-specific basis, and the laboratory should establish in-house QC performance criteria for the application of this method. These performance data are not intended to be and must not be used as absolute QC acceptance criteria for purposes of laboratory accreditation.

13.2   Single laboratory initial demonstration of capability data were generated from five replicate measurements using a modified continuous liquid-liquid extractor (Method 3520) with hydrophobic membrane. In this case only a single acid pH extraction was performed using the

CLP calibration criteria and the applicable CLP target analytes. These data are presented in Table 6. Laboratories should generate their own acceptance criteria depending on the extraction and instrument conditions. See Method 8000 for more detailed guidance.

13.3   Chromatograms from calibration standards analyzed with Day 0 and Day 7 samples were compared to detect possible deterioration of gas chromatographic performance. These recoveries (using Method 3510 extraction) are presented in Table 7. These data are provided for guidance purposes only.

13.4   Method performance data using Method 3541 (i.e., automated Soxhlet extraction) are presented in Tables 8 and 9. Single laboratory accuracy and precision data were obtained for semivolatile organics in a clay soil by spiking at a concentration of 6 mg/kg for each compound. The spiking solution was mixed into the soil during addition and then allowed to equilibrate for approximately one hour prior to extraction. The spiked samples were then extracted by Method 3541 (Automated Soxhlet). Three extractions were performed and each extract was analyzed by GC/MS following Method 8270. The low recovery of the more volatile compounds is probably due to volatilization losses during equilibration. These data as listed were taken from Reference 7 and are provided for guidance purposes only.

13.5   Surrogate precision and accuracy data are presented in Table 10 from a field dynamic spiking study based on air sampling by Method 0010. The trapping media were prepared for analysis by Method 3542 and subsequently analyzed by this method (i.e., 8270). These data are provided for guidance purposes only.

13.6   Single laboratory precision and bias data using Method 3545 (i.e., pressurized fluid extraction) for semivolatile organic compounds are presented in Table 11. The samples were conditioned spiked samples prepared and certified by a commercial supplier that contained 57 semivolatile organics at three concentrations (i.e., 250, 2500, and 12,500 µg/kg) on three types of soil (i.e, clay, loam, and sand). Spiked samples were extracted both by the Dionex ASE system and by the Perstorp Environmental SoxtecTM (i.e., automated Soxhlet). The data in Table 11 represent seven replicate extractions and analyses for each individual sample and were taken from Reference 9. The average recoveries from the three matrices for all analytes and all replicates relative to the automated Soxhlet data are as follows: clay 96.8%, loam

98.7% and sand 102.1%. The average recoveries from the three concentrations also relative to the automated Soxhlet data are as follows: low – 101.2%, mid – 97.2% and high – 99.2%. These data are provided for guidance purposes only.

13.7   Single laboratory precision and bias data using Method 3561 (i.e., SFE extraction of PAHs with a variable restrictor and solid trapping material) were obtained for the method analytes by the extraction of two certified reference materials (i.e., EC-1, a lake sediment from Environment Canada and HS-3, a marine sediment from the National Science and Engineering Research Council of Canada, both naturally contaminated with PAHs). The SFE instrument used for these extractions was a Hewlett-Packard Model 7680. Analysis was by GC/MS. Average recoveries from six replicate extractions ranged from 85 to 148%, with an overall average of 100%, based on the certified value (or a Soxhlet value if a certified value was unavailable for a specific analyte) for the lake sediment. Average recoveries from three replicate extractions ranged from 73 to 133%, with an overall average of 92%, based on the certified value for the marine sediment. The data are found in Tables 12 and 13 and were taken from Reference 10. These data are provided for guidance purposes only.

13.8   Single laboratory precision and accuracy using Method 3561 (i.e., SFE extraction of PAHs with a fixed restrictor and liquid trapping) were obtained for twelve of the method analytes by the extraction of a certified reference material (i.e., a soil naturally contaminated with PAHs). The SFE instrument used for these extractions was a Dionex Model 703-M. Analysis was by GC/MS. Average recoveries from four replicate extractions ranged from 60 to 122%, with an overall average of 89%, based on the certified value. The instrument conditions that were utilized to extract a 3.4 g sample were as follows: Pressure - 300 atm; time - 60 min; extraction fluid - CO2; modifier - 10% 1:1 (v/v) methanol/methylene chloride; Oven temperature - 80 °C; Restrictor temperature - 120 °C; and, trapping fluid - chloroform (methylene chloride has also been used). The data are found in Table 14 and were taken from Reference 11. These data are provided for guidance purposes only.

13.9   Tables 15 and 16 contain single-laboratory precision and accuracy data for solidphase extraction of TCLP buffer solutions spiked at two levels and extracted using Method 3535. These data are provided for guidance purposes only.

13.10Table 17 contains multiple-laboratory data for solid-phase extraction of spiked TCLP soil leachates extracted using Method 3535. These data are provided for guidance purposes only.

13.11Tables 18 through 22 contain single-laboratory PAH recovery data for microwave extraction of contaminated soils and standard reference materials using Method 3546. These data are provided for guidance purposes only.

14.0   POLLUTION PREVENTION

14.1   Pollution prevention encompasses any technique that reduces or eliminates the quantity and/or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operations. The EPA has established a preferred hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the Agency recommends recycling as the next best option.

14.2   For information about pollution prevention that may be applicable to laboratories and research institutions consult Less is Better: Laboratory Chemical Management for Waste Reduction, a free publication available from the ACS, Committee on Chemical Safety, . http://portal.acs.org/portal/fileFetch/C/WPCP\_012290/pdf/WPCP\_012290.pdf

15.0 WASTE MANAGEMENT

The EPA requires that laboratory waste management practices be conducted consistent with all applicable rules and regulations. The Agency urges laboratories to protect the air, water, and land by minimizing and controlling all releases from hoods and bench operations, complying with the letter and spirit of any sewer discharge permits and regulations, and by complying with all solid and hazardous waste regulations, particularly the hazardous waste identification rules and land disposal restrictions. For further information on waste management, consult The Waste Management Manual for Laboratory Personnel available from the ACS at the web address listed in Sec. 14.2.

16.0 REFERENCES

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17.0 TABLES, DIAGRAMS, FLOW CHARTS, AND VALIDATION DATA

                The following pages contain the tables and figures referenced by this method.

TABLE 1

# CHARACTERISTIC IONS FOR SEMIVOLATILE COMPOUNDS IN APPROXIMATE RETENTION TIME ORDER a

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                      Compound Primary Ion Secondary Ion(s)

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|  |  |  |
| --- | --- | --- |
| 2-Picoline | 93 | 66,92 |
| Aniline | 93 | 66,65 |
| Phenol | 94 | 65,66 |
| Bis(2-chloroethyl) ether | 93 | 63,95 |
| 2-Chlorophenol | 128 | 64,130 |
| 1,3-Dichlorobenzene | 146 | 148,111 |
| 1,4-Dichlorobenzene-d4 (IS) | 152 | 150,115 |
| 1,4-Dichlorobenzene | 146 | 148,111 |
| Benzyl alcohol | 108 | 79,77 |
| 1,2-Dichlorobenzene | 146 | 148,111 |
| N-Nitrosomethylethylamine | 88 | 42,43,56 |
| Bis(2-chloro-1-methylethyl)ether | 45 | 77,121 |
| Ethyl carbamate | 62 | 44,45,74 |
| Thiophenol (Benzenethiol) | 110 | 66,109,84 |
| Methyl methanesulfonate | 80 | 79,65,95 |
| N-Nitrosodi-n-propylamine | 70 | 42,101,130 |
| Hexachloroethane | 117 | 201,199 |
| Maleic anhydride | 54 | 98,53,44 |
| Nitrobenzene | 77 | 123,65 |
| Isophorone | 82 | 95,138 |
| N-Nitrosodiethylamine | 102 | 42,57,44,56 |
| 2-Nitrophenol | 139 | 109,65 |
| 2,4-Dimethylphenol | 122 | 107,121 |
| p-Benzoquinone | 108 | 54,82,80 |
| Bis(2-chloroethoxy)methane | 93 | 95,123 |
| Benzoic acid | 122 | 105,77 |
| 2,4-Dichlorophenol | 162 | 164,98 |
| Trimethyl phosphate | 110 | 79,95,109,140 |
| Ethyl methanesulfonate | 79 | 109,9745,65 |
| 1,2,4-Trichlorobenzene | 180 | 182,145 |
| Naphthalene-d8 (IS) | 136 | 68 |
| Naphthalene | 128 | 129,127 |
| Hexachlorobutadiene | 225 | 223,227 |
| Tetraethyl pyrophosphate | 99 | 155,127,81,109 |
| Diethyl sulfate | 139 | 45,59,99,111,125 |
| 4-Chloro-3-methylphenol | 107 | 144,142 |
| 2-Methylnaphthalene | 142 | 141 |
| 2-Methylphenol | 107 | 108,77,79,90 |
| Hexachloropropene | 213 | 211,215,117,106,141 |
| Hexachlorocyclopentadiene | 237 | 235,272 |
| N-Nitrosopyrrolidine | 100 | 41,42,68,69 |
| Acetophenone | 105 | 71,51,120 |
| 3/4-Methylphenol b | 107 | 108,77,79,90 |
| 2,4,6-Trichlorophenol | 196 | 198,200 |
| o-Toluidine | 106 | 107,77,51,79 |
| 2-Chloronaphthalene | 162 | 127,164 |
| N-Nitrosopiperidine | 114 | 42,55,56,41 |

|  |  |  |
| --- | --- | --- |
| 1,4-Phenylenediamine | 108 | 80,53,54,52 |
| 1-Chloronaphthalene | 162 | 127,164 |
| 2-Nitroaniline | 65 | 92,138 |
| 5-Chloro-2-methylaniline | 106 | 141,140,77,89 |
| Dimethyl phthalate | 163 | 194,164 |
| Acenaphthylene | 152 | 151,153 |
| 2,6-Dinitrotoluene | 165 | 63,89 |
| Phthalic anhydride | 104 | 76,50,148 |
| o-Anisidine | 108 | 80,123,52 |
| 3-Nitroaniline | 138 | 108,92 |
| Acenanaphthene-d10 (IS) | 164 | 162,160 |
| Acenaphthene | 154 | 153,152 |
| 2,4-Dinitrophenol | 184 | 63,154 |
| 2,6-Dinitrophenol | 162 | 164,126,98,63 |
| 4-Chloroaniline | 127 | 129,65,92 |
| Isosafrole | 162 | 131,104,77,51 |
| Dibenzofuran | 168 | 139 |
| 2,4-Diaminotoluene | 121 | 122,94,77,104 |
| 2,4-Dinitrotoluene | 165 | 63,89 |
| 4-Nitrophenol | 139 | 109,65 |
| 2-Naphthylamine | 143 | 115,116 |
| 1,4-Naphthoquinone | 158 | 104,102,76,50,130 |
| p-Cresidine | 122 | 94,137,77,93 |
| Dichlorovos | 109 | 185,79,145 |
| Diethyl phthalate | 149 | 177,150 |
| Fluorene | 166 | 165,167 |
| 2,4,5-Trimethylaniline | 120 | 135,134,91,77 |
| N-Nitrosodi-n-butylamine | 84 | 57,41,116,158 |
| 4-Chlorophenyl phenyl ether | 204 | 206,141 |
| Hydroquinone | 110 | 81,53,55 |
| 4,6-Dinitro-2-methylphenol | 198 | 51,105 |
| Resorcinol | 110 | 81,82,53,69 |
| N-Nitrosodiphenylamine | 169 | 168,167 |
| Safrole | 162 | 104,77,103,135 |
| Hexamethyl phosphoramide | 135 | 44,179,92,42 |
| 3-(Chloromethyl)pyridine hydrochloride | 92 | 127,129,65,39 |
| Diphenylamine | 169 | 168,167 |
| 1,2,4,5-Tetrachlorobenzene | 216 | 214,179,108,143,218 |
| 1-Naphthylamine | 143 | 115,89,63 |
| 1-Acetyl-2-thiourea | 118 | 43,42,76 |
| 4-Bromophenyl phenyl ether | 248 | 250,141 |
| Toluene diisocyanate | 174 | 145,173,146,132,91 |
| 2,4,5-Trichlorophenol | 196 | 198,97,132,99 |
| Hexachlorobenzene | 284 | 142,249 |
| Nicotine | 84 | 133,161,162 |
| Pentachlorophenol | 266 | 264,268 |
| 5-Nitro-o-toluidine | 152 | 77,79,106,94 |
| Thionazine | 107 | 96,97,143,79,68 |
| 4-Nitroaniline | 138 | 65,108,92,80,39 |
| Phenanthrene-d10 (IS) | 188 | 94,80 |
| Phenanthrene | 178 | 179,176 |
| Anthracene | 178 | 176,179 |

|  |  |  |
| --- | --- | --- |
| 1,4-Dinitrobenzene | 168 | 75,50,76,92,122 |
| Mevinphos | 127 | 192,109,67,164 |
| Naled | 109 | 145,147,301,79,189 |
| 1,3-Dinitrobenzene | 168 | 76,50,75,92,122 |
| Diallate (cis or trans) | 86 | 234,43,70 |
| 1,2-Dinitrobenzene | 168 | 50,63,74 |
| Diallate (trans or cis) | 86 | 234,43,70 |
| Pentachlorobenzene | 250 | 252,108,248,215,254 |
| 5-Nitro-o-anisidine | 168 | 79,52,138,153,77 |
| Pentachloronitrobenzene | 237 | 142,214,249,295,265 |
| 4-Nitroquinoline-1-oxide | 174 | 101,128,75,116 |
| Di-n-butyl phthalate | 149 | 150,104 |
| 2,3,4,6-Tetrachlorophenol | 232 | 131,230,166,234,168 |
| Dihydrosaffrole | 135 | 64,77 |
| Demeton-O | 88 | 89,60,61,115,171 |
| Fluoranthene | 202 | 101,203 |
| 1,3,5-Trinitrobenzene | 75 | 74,213,120,91,63 |
| Dicrotophos | 127 | 67,72,109,193,237 |
| Benzidine | 184 | 92,185 |
| Trifluralin | 306 | 43,264,41,290 |
| Bromoxynil | 277 | 279,88,275,168 |
| Pyrene | 202 | 200,203 |
| Monocrotophos | 127 | 192,67,97,109 |
| Phorate | 75 | 121,97,93,260 |
| Sulfallate | 188 | 88,72,60,44 |
| Demeton-S | 88 | 60,81,89,114,115 |
| Phenacetin | 108 | 180,179,109,137,80 |
| Dimethoate | 87 | 93,125,143,229 |
| Phenobarbital | 204 | 117,232,146,161 |
| Carbofuran | 164 | 149,131,122 |
| Octamethyl pyrophosphoramide | 135 | 44,199,286,153,243 |
| 4-Aminobiphenyl | 169 | 168,170,115 |
| Dioxathion | 97 | 125,270,153 |
| Terbufos | 231 | 57,97,153,103 |
| α,α-Dimethylphenylamine | 58 | 91,65,134,42 |
| Pronamide | 173 | 175,145,109,147 |
| Aminoazobenzene | 197 | 92,120,65,77 |
| Dichlone | 191 | 163,226,228,135,193 |
| Dinoseb | 211 | 163,147,117,240 |
| Disulfoton | 88 | 97,89,142,186 |
| Fluchloralin | 306 | 63,326,328,264,65 |
| Mexacarbate | 165 | 150,134,164,222 |
| 4,4&apos;-Oxydianiline | 200 | 108,171,80,65 |
| Butyl benzyl phthalate | 149 | 91,206 |
| 4-Nitrobiphenyl | 199 | 152,141,169,151 |
| Phosphamidon | 127 | 264,72,109,138 |
| 2-Cyclohexyl-4,6-Dinitrophenol | 231 | 185,41,193,266 |
| Methyl parathion | 109 | 125,263,79,93 |
| Carbaryl | 144 | 115,116,201 |
| Dimethylaminoazobenzene | 225 | 120,77,105,148,42 |
| Propylthiouracil | 170 | 142,114,83 |
| Benz(a)anthracene | 228 | 229,226 |

|  |  |  |
| --- | --- | --- |
| Chrysene-d12 (IS) | 240 | 120,236 |
| 3,3&apos;-Dichlorobenzidine | 252 | 254,126 |
| Chrysene | 228 | 226,229 |
| Malathion | 173 | 125,127,93,158 |
| Kepone | 272 | 274,237,178,143,270 |
| Fenthion | 278 | 125,109,169,153 |
| Parathion | 109 | 97,291,139,155 |
| Anilazine | 239 | 241,143,178,89 |
| Bis(2-ethylhexyl)phthalate | 149 | 167,279 |
| 3,3&apos;-Dimethylbenzidine | 212 | 106,196,180 |
| Carbophenothion | 157 | 97,121,342,159,199 |
| 5-Nitroacenaphthene | 199 | 152,169,141,115 |
| Methapyrilene | 97 | 50,191,71 |
| Isodrin | 193 | 66,195,263,265,147 |
| Captan | 79 | 149,77,119,117 |
| Chlorfenvinphos | 267 | 269,323,325,295 |
| Crotoxyphos | 127 | 105,193,166 |
| Phosmet | 160 | 77,93,317,76 |
| EPN | 157 | 169,185,141,323 |
| Tetrachlorvinphos | 329 | 109,331,79,333 |
| Di-n-octyl phthalate | 149 | 167,43 |
| 2-Aminoanthraquinone | 223 | 167,195 |
| Barban | 222 | 51,87,224,257,153 |
| Aramite | 185 | 191,319,334,197,321 |
| Benzo(b)fluoranthene | 252 | 253,125 |
| Nitrofen | 283 | 285,202,139,253 |
| Benzo(k)fluoranthene | 252 | 253,125 |
| Chlorobenzilate | 251 | 139,253,111,141 |
| Fensulfothion | 293 | 97,308,125,292 |
| Ethion | 231 | 97,153,125,121 |
| Diethylstilbestrol | 268 | 145,107,239,121,159 |
| Famphur | 218 | 125,93,109,217 |
| Tri-p-tolyl phosphatec | 368 | 367,107,165,198 |
| Benzo(a)pyrene | 252 | 253,125 |
| Perylene-d12 (IS) | 264 | 260,265 |
| 7,12-Dimethylbenz(a)anthracene | 256 | 241,239,120 |
| 5,5-Diphenylhydantoin | 180 | 104,252,223,209 |
| Captafol | 79 | 77,80,107 |
| Dinocap | 69 | 41,39 |
| Methoxychlor | 227 | 228,152,114,274,212 |
| 2-Acetylaminofluorene | 181 | 180,223,152 |
| 4,4&apos;-Methylenebis(2-chloroaniline) | 231 | 266,268,140,195 |
| 3,3&apos;-Dimethoxybenzidine | 244 | 201,229 |
| 3-Methylcholanthrene | 268 | 252,253,126,134,113 |
| Phosalone | 182 | 184,367,121,379 |
| Azinphos-methyl | 160 | 132,93,104,105 |
| Leptophos | 171 | 377,375,77,155,379 |
| Mirex | 272 | 237,274,270,239,235 |
| Tris(2,3-dibromopropyl)phosphate | 201 | 137,119,217,219,199 |
| Dibenz(a,j)acridine | 279 | 280,277,250 |
| Mestranol | 277 | 310,174,147,242 |
| Coumaphos | 362 | 226,210,364,97,109 |
| Indeno(1,2,3-cd)pyrene | 276 | 138,277 |
| Dibenz(a,h)anthracene | 278 | 139,279 |
| Benzo(g,h,i)perylene | 276 | 138,277 |
| 1,2:4,5-Dibenzopyrene | 302 | 151,150,300 |
| Strychnine | 334 | 334,335,333 |
| Piperonyl sulfoxide | 162 | 135,105,77 |
| Hexachlorophene | 196 | 198,209,211,406,408 |
| Aldrin | 66 | 263,220 |
| Aroclor 1016 | 222 | 260,292 |
| Aroclor 1221 | 190 | 224,260 |
| Aroclor 1232 | 190 | 224,260 |
| Aroclor 1242 | 222 | 256,292 |
| Aroclor 1248 | 292 | 362,326 |
| Aroclor 1254 | 292 | 362,326 |
| Aroclor 1260 | 360 | 362,394 |
| α-BHC | 183 | 181,109 |
| β-BHC | 181 | 183,109 |
| δ-BHC | 183 | 181,109 |
| γ-BHC (Lindane) | 183 | 181,109 |
| 4,4&apos;-DDD | 235 | 237,165 |
| 4,4&apos;-DDE | 246 | 248,176 |
| 4,4&apos;-DDT | 235 | 237,165 |
| Dieldrin | 79 | 263,279 |
| 1,2-Diphenylhydrazine | 77 | 105,182 |
| Endosulfan I | 195 | 339,341 |
| Endosulfan II | 337 | 339,341 |
| Endosulfan sulfate | 272 | 387,422 |
| Endrin | 263 | 82,81 |
| Endrin aldehyde | 67 | 345,250 |
| Endrin ketone | 317 | 67,319 |
| 2-Fluorobiphenyl (surr) | 172 | 171 |
| 2-Fluorophenol (surr) | 112 | 64 |
| Heptachlor | 100 | 272,274 |
| Heptachlor epoxide | 353 | 355,351 |
| Nitrobenzene-d5 (surr) | 82 | 128,54 |
| N-Nitrosodimethylamine | 42 | 74,44 |
| Phenol-d6 (surr) | 99 | 42,71 |
| Terphenyl-d14 (surr) | 244 | 122,212 |
| 2,4,6-Tribromophenol (surr) | 330 | 332,141 |
| Toxaphene | 159 | 231,233 |

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IS = internal standard surr = surrogate

a The data presented are representative of DB-5 type analytical columns.

b

Compounds cannot be separated for quantitation

c

Substitute for the non-specific mixture, tricresyl phosphate

# TABLE 2

EXAMPLE LOWER LIMITS OF QUANTITATION FOR SEMIVOLATILE ORGANICS

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# Lower Limits of Quantitationa

|  |  |  |  |  |
| --- | --- | --- | --- | --- |
| Compound | | Ground water (µg/L) | Low Soil/Sedimentb (µg/kg) | |
|  | Acenaphthene | 10 | 660 |  |
|  | Acenaphthylene | 10 | 660 |  |
|  | Acetophenone | 10 | ND |  |
|  | 2-Acetylaminofluorene | 20 | ND |  |
|  | 1-Acetyl-2-thiourea | 1000 | ND |  |
|  | 2-Aminoanthraquinone | 20 | ND |  |
|  | Aminoazobenzene | 10 | ND |  |
|  | 4-Aminobiphenyl | 20 | ND |  |
|  | Anilazine | 100 | ND |  |
|  | o-Anisidine | 10 | ND |  |
|  | Anthracene | 10 | 660 |  |
|  | Aramite | 20 | ND |  |
|  | Azinphos-methyl | 100 | ND |  |
|  | Barban | 200 | ND |  |
|  | Benz(a)anthracene | 10 | 660 |  |
|  | Benzo(b)fluoranthene | 10 | 660 |  |
|  | Benzo(k)fluoranthene | 10 | 660 |  |
|  | Benzoic acid | 50 | 3300 |  |
|  | Benzo(g,h,i)perylene | 10 | 660 |  |
|  | Benzo(a)pyrene | 10 | 660 |  |
|  | p-Benzoquinone | 10 | ND |  |
|  | Benzyl alcohol | 20 | 1300 |  |
|  | Bis(2-chloroethoxy)methane | 10 | 660 |  |
|  | Bis(2-chloroethyl) ether | 10 | 660 |  |
|  | Bis(2-chloro-1-methylethyl)ether | 10 | 660 |  |
|  | 4-Bromophenyl phenyl ether | 10 | 660 |  |
|  | Bromoxynil | 10 | ND |  |
|  | Butyl benzyl phthalate | 10 | 660 |  |
|  | Captafol | 20 | ND |  |
|  | Captan | 50 | ND |  |
|  | Carbaryl | 10 | ND |  |
|  | Carbofuran | 10 | ND |  |
|  | Carbophenothion | 10 | ND |  |
|  | Chlorfenvinphos | 20 | ND |  |
|  | 4-Chloroaniline | 20 | 1300 |  |
|  | Chlorobenzilate | 10 | ND |  |
|  | 5-Chloro-2-methylaniline | 10 | ND |  |
|  | 4-Chloro-3-methylphenol | 20 | 1300 |  |
|  | 3-(Chloromethyl)pyridine hydrochloride | 100 | ND |  |
|  | 2-Chloronaphthalene | 10 | 660 |  |
|  | 2-Chlorophenol | 10 | 660 |  |
|  | 4-Chlorophenyl phenyl ether | 10 | 660 |  |
|  | Chrysene | 10 | 660 |  |
|  | Coumaphos | 40 | ND |  |
|  | p-Cresidine | 10 | ND |  |
|  | Crotoxyphos | 20 | ND |  |
|  | 2-Cyclohexyl-4,6-dinitrophenol | 100 | ND |  |
|  |  |  |  |  |

|  |  |  |
| --- | --- | --- |
| Compound |  | (µg/kg) |
| Demeton-O | 10 | ND |
| Demeton-S | 10 | ND |
| Diallate (cis or trans) | 10 | ND |
| Diallate (trans or cis) | 10 | ND |
| 2,4-Diaminotoluene | 20 | ND |
| Dibenz(a,j)acridine | 10 | ND |
| Dibenz(a,h)anthracene | 10 | 660 |
| Dibenzofuran | 10 | 660 |
| Dibenzo(a,e)pyrene | 10 | ND |
| Di-n-butyl phthalate | 10 | ND |
| Dichlone | NA | ND |
| 1,2-Dichlorobenzene | 10 | 660 |
| 1,3-Dichlorobenzene | 10 | 660 |
| 1,4-Dichlorobenzene | 10 | 660 |
| 3,3&apos;-Dichlorobenzidine | 20 | 1300 |
| 2,4-Dichlorophenol | 10 | 660 |
| 2,6-Dichlorophenol | 10 | ND |
| Dichlorovos | 10 | ND |
| Dicrotophos | 10 | ND |
| Diethyl phthalate | 10 | 660 |
| Diethylstilbestrol | 20 | ND |
| Diethyl sulfate | 100 | ND |
| Dimethoate | 20 | ND |
| 3,3&apos;-Dimethoxybenzidine | 100 | ND |
| Dimethylaminoazobenzene | 10 | ND |
| 7,12-Dimethylbenz(a)anthracene | 10 | ND |
| 3,3&apos;-Dimethylbenzidine | 10 | ND |
| 2,4-Dimethylphenol | 10 | 660 |
| Dimethyl phthalate | 10 | 660 |
| 1,2-Dinitrobenzene | 40 | ND |
| 1,3-Dinitrobenzene | 20 | ND |
| 1,4-Dinitrobenzene | 40 | ND |
| 4,6-Dinitro-2-methylphenol | 50 | 3300 |
| 2,4-Dinitrophenol | 50 | 3300 |
| 2,4-Dinitrotoluene | 10 | 660 |
| 2,6-Dinitrotoluene | 10 | 660 |
| Dinocap | 100 | ND |
| Dinoseb | 20 | ND |
| 5,5-Diphenylhydantoin | 20 | ND |
| Di-n-octyl phthalate | 10 | 660 |
| Disulfoton | 10 | ND |
| EPN | 10 | ND |
| Ethion | 10 | ND |
| Ethyl carbamate | 50 | ND |
| Bis(2-ethylhexyl)phthalate | 10 | 660 |
| Ethyl methanesulfonate | 20 | ND |
| Famphur | 20 | ND |
| Fensulfothion | 40 | ND |
| Fenthion | 10 | ND |
| Fluchloralin | 20 | ND |
| Fluoranthene | 10 | 660 |

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|  |  |  |
| --- | --- | --- |
| Compound |  | (µg/kg) |
| Fluorene | 10 | 660 |
| Hexachlorobenzene | 10 | 660 |
| Hexachlorobutadiene | 10 | 660 |
| Hexachlorocyclopentadiene | 10 | 660 |
| Hexachloroethane | 10 | 660 |
| Hexachlorophene | 50 | ND |
| Hexachloropropene | 10 | ND |
| Hexamethylphosphoramide | 20 | ND |
| Indeno(1,2,3-cd)pyrene | 10 | 660 |
| Isodrin | 20 | ND |
| Isophorone | 10 | 660 |
| Isosafrole | 10 | ND |
| Kepone | 20 | ND |
| Leptophos | 10 | ND |
| Malathion | 50 | ND |
| Mestranol | 20 | ND |
| Methapyrilene | 100 | ND |
| Methoxychlor | 10 | ND |
| 3-Methylcholanthrene | 10 | ND |
| Methyl methanesulfonate | 10 | ND |
| 2-Methylnaphthalene | 10 | 660 |
| Methyl parathion | 10 | ND |
| 2-Methylphenol | 10 | 660 |
| 3-Methylphenol | 10 | ND |
| 4-Methylphenol | 10 | 660 |
| Mevinphos | 10 | ND |
| Mexacarbate | 20 | ND |
| Mirex | 10 | ND |
| Monocrotophos | 40 | ND |
| Naled | 20 | ND |
| Naphthalene | 10 | 660 |
| 1,4-Naphthoquinone | 10 | ND |
| 1-Naphthylamine | 10 | ND |
| 2-Naphthylamine | 10 | ND |
| Nicotine | 20 | ND |
| 5-Nitroacenaphthene | 10 | ND |
| 2-Nitroaniline | 50 | 3300 |
| 3-Nitroaniline | 50 | 3300 |
| 4-Nitroaniline | 20 | ND |
| 5-Nitro-o-anisidine | 10 | ND |
| Nitrobenzene | 10 | 660 |
| 4-Nitrobiphenyl | 10 | ND |
| Nitrofen | 20 | ND |
| 2-Nitrophenol | 10 | 660 |
| 4-Nitrophenol | 50 | 3300 |
| 5-Nitro-o-toluidine | 10 | ND |
| 4-Nitroquinoline-1-oxide | 40 | ND |
| N-Nitrosodi-n-butylamine | 10 | ND |
| N-Nitrosodiethylamine | 20 | ND |
| N-Nitrosodiphenylamine | 10 | 660 |
| N-Nitroso-di-n-propylamine | 10 | 660 |

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|  |  |  |
| --- | --- | --- |
| Compound | Ground water (µg/L) | Low Soil/Sedimentb (µg/kg) |
| N-Nitrosopiperidine | 20 | ND |
| N-Nitrosopyrrolidine | 40 | ND |
| Octamethyl pyrophosphoramide | 200 | ND |
| 4,4&apos;-Oxydianiline | 20 | ND |
| Parathion | 10 | ND |
| Pentachlorobenzene | 10 | ND |
| Pentachloronitrobenzene | 20 | ND |
| Pentachlorophenol | 50 | 3300 |
| Phenacetin | 20 | ND |
| Phenanthrene | 10 | 660 |
| Phenobarbital | 10 | ND |
| Phenol | 10 | 660 |
| 1,4-Phenylenediamine | 10 | ND |
| Phorate | 10 | ND |
| Phosalone | 100 | ND |
| Phosmet | 40 | ND |
| Phosphamidon | 100 | ND |
| Phthalic anhydride | 100 | ND |
| 2-Picoline | ND | ND |
| Piperonyl sulfoxide | 100 | ND |
| Pronamide | 10 | ND |
| Propylthiouracil | 100 | ND |
| Pyrene | 10 | 660 |
| Resorcinol | 100 | ND |
| Safrole | 10 | ND |
| Strychnine | 40 | ND |
| Sulfallate | 10 | ND |
| Terbufos | 20 | ND |
| 1,2,4,5-Tetrachlorobenzene | 10 | ND |
| 2,3,4,6-Tetrachlorophenol | 10 | ND |
| Tetrachlorvinphos | 20 | ND |
| Tetraethyl pyrophosphate | 40 | ND |
| Thionazine | 20 | ND |
| Thiophenol (Benzenethiol) | 20 | ND |
| o-Toluidine | 10 | ND |
| 1,2,4-Trichlorobenzene | 10 | 660 |
| 2,4,5-Trichlorophenol | 10 | 660 |
| 2,4,6-Trichlorophenol | 10 | 660 |
| Trifluralin | 10 | ND |
| 2,4,5-Trimethylaniline | 10 | ND |
| Trimethyl phosphate | 10 | ND |
| 1,3,5-Trinitrobenzene | 10 | ND |
| Tris(2,3-dibromopropyl)phosphate | 200 | ND |
| Tri-p-tolyl phosphate(h) | 10 | ND |

a Sample LLOQs are highly matrix-dependent and those listed here are provided for guidance and may not always be achievable.

b LLOQs listed for soil/sediment are based on wet weight. When data are reported on a dry weight basis, the lower limits will be higher based on the % dry weight of each sample. These lower limits are based on a 30-g sample and gel permeation chromatography cleanup.

ND = Not Determined

NA = Not Applicable

          Other Matrices Factorc

          High-concentration soil and sludges by ultrasonic extraction 7.5

         Non-water miscible waste 75

 c

LLOQ=(LLOQ for low soil/sediment given above in Table 2) x (Factor)

TABLE 3

# DFTPP KEY IONS AND ION ABUNDANCE CRITERIAa,b

|  |  |
| --- | --- |
| Mass | Ion Abundance Criteria |
| 51 | 10-80% of Base Peak |
| 68 | < 2% of mass 69 |
| 70 | < 2% of mass 69 |
| 127 | 10-80% of Base Peak |
| 197 | < 2% of mass 198 |
| 198 | Base peak, or > 50% of Mass 442 |
| 199 | 5-9% of mass 198 |
| 275 | 10-60% of Base Peak |
| 365 | > 1% of mass 198 |
| 441 | present but < 24% of mass 442 |
| 442 | Base Peak, or > 50% of mass 198 |
| 443 | 15-24% of mass 442 |

a The majority of the data are taken from Reference 13 (Method 525.2).

b The criteria in this table are intended to be used as default criteria for quadrupole instrumentation if optimized manufacturer&apos;s operating conditions are not available. Alternate tuning criteria may be employed (e.g., CLP or Method 625), provided that method performance is not adversely affected. See Sec. 11.3.1.

# TABLE 4

RECOMMENDED MINIMUM RESPONSE FACTOR CRITERIA FOR INITIAL AND

CONTINUING CALIBRATION VERIFICATION USING THE SUGGESTED IONS

FROM TABLE 1

# Minimum Response Factor (RF)

D:\document\convert_tasks\transweb\1464945_1477166\1464945.pdf.files\image019.gifSemivolatile Compounds

                                        Benzaldehyde 0.010

                                        Phenol 0.800

                                        Bis(2-chloroethyl)ether 0.700

                                        2-Chlorophenol 0.800

                                        2-Methylphenol 0.700

                                        2,2&apos;-Oxybis-(1-chloropropane) 0.010

                                        Acetophenone 0.010

                                        4-Methylphenol 0.600

                                        N-Nitroso-di-n-propylamine 0.500

Hexachloroethane 0.300 Nitrobenzene 0.200

                                        Isophorone 0.400

                                        2-Nitrophenol 0.100

                                        2,4-Dimethylphenol 0.200

                                        Bis(2-chloroethoxy)methane 0.300

                                        2,4-Dichlorophenol 0.200

                                        Naphthalene 0.700

4-Chloroaniline 0.010 Hexachlorobutadiene 0.010

                                        Caprolactam 0.010

4-Chloro-3-methylphenol 0.200 2-Methylnaphthalene 0.400 Hexachlorocyclopentadiene 0.050 2,4,6-Trichlorophenol 0.200

                                        2,4,5-Trichlorophenol 0.200

                                        1,1&apos;-Biphenyl 0.010

2-Chloronaphthalene 0.800 2-Nitroaniline 0.010 Dimethyl phthalate 0.010 2,6-Dinitrotoluene 0.200

                                        Acenaphthylene 0.900

                                        3-Nitroaniline 0.010

                                        Acenaphthene 0.900

                                        2,4-Dinitrophenol 0.010

                                        4-Nitrophenol 0.010

                                        Dibenzofuran 0.800

                                        2,4-Dinitrotoluene 0.200

                                        Diethyl phthalate 0.010

1,2,4,5-Tetrachlorobenzene 0.010 4-Chlorophenyl-phenyl ether 0.400

                                        Fluorene 0.900

                                        4-Nitroaniline 0.010

                                        4,6-Dinitro-2-methylphenol 0.010

                                        4-Bromophenyl-phenyl ether 0.100

N-Nitrosodiphenylamine 0.010 Hexachlorobenzene 0.100

|  |  |
| --- | --- |
| Semivolatile Compounds | Minimum Response Factor (RF) |
| Atrazine | 0.010 |
| Pentachlorophenol | 0.050 |
| Phenanthrene | 0.700 |
| Anthracene | 0.700 |
| Carbazole | 0.010 |
| Di-n-butyl phthalate | 0.010 |
| Fluoranthene | 0.600 |
| Pyrene | 0.600 |
| Butyl benzyl phthalate | 0.010 |
| 3,3&apos;-Dichlorobenzidine | 0.010 |
| Benzo(a)anthracene | 0.800 |
| Chrysene | 0.700 |
| Bis-(2-ethylhexyl)phthalate | 0.010 |
| Di-n-octyl phthalate | 0.010 |
| Benzo(b)fluoranthene | 0.700 |
| Benzo(k)fluoranthene | 0.700 |
| Benzo(a)pyrene | 0.700 |
| Indeno(1,2,3-cd)pyrene | 0.500 |
| Dibenz(a,h)anthracene | 0.400 |
| Benzo(g,h,i)perylene | 0.500 |
| 2,3,4,6-Tetrachlorophenol | 0.010 |

TABLE 5

|  |  |  |
| --- | --- | --- |
|  |  |  |
| 1,4-Dichlorobenzene-d4 | Naphthalene-d8 | Acenaphthene-d10 |
| Aniline | Acetophenone | Acenaphthene |
| Benzyl alcohol | Benzoic acid | Acenaphthylene |
| Bis(2-chloroethyl)ether | Bis(2-chloroethoxy)methane | 1-Chloronaphthalene |
| Bis(2-chloro-1-methylethyl) ether | 4-Chloroaniline | 2-Chloronaphthalene |
| 2-Chlorophenol | 4-Chloro-3-methylphenol | 4-Chlorophenyl phenyl ether |
| 1,3-Dichlorobenzene | 2,4-Dichlorophenol | Dibenzofuran |
| 1,4-Dichlorobenzene | 2,6-Dichlorophenol | Diethyl phthalate |
| 1,2-Dichlorobenzene | α,α-Dimethylphenethylamine | Dimethyl phthalate |
| Ethyl methanesulfonate | 2,4-Dimethylphenol | 2,4-Dinitrophenol |
| 2-Fluorophenol (surr) | Hexachlorobutadiene | 2,4-Dinitrotoluene |
| Hexachloroethane | Isophorone | 2,6-Dinitrotoluene |
| Methyl methanesulfonate | 2-Methylnaphthalene | Fluorene |
| 2-Methylphenol | Naphthalene | 2-Fluorobiphenyl (surr) |
| 4-Methylphenol | Nitrobenzene | Hexachlorocyclopentadiene |
| N-Nitrosodimethylamine | Nitrobenzene-d8 (surr) | 1-Naphthylamine |
| N-Nitroso-di-n-propylamine | 2-Nitrophenol | 2-Naphthylamine |
| Phenol | N-Nitrosodi-n-butylamine | 2-Nitroaniline |
| Phenol-d6 (surr) | N-Nitrosopiperidine | 3-Nitroaniline |
| 2-Picoline | 1,2,4-Trichlorobenzene | 4-Nitroaniline |
|  |  | 4-Nitrophenol |
|  |  | Pentachlorobenzene |
|  |  | 1,2,4,5-Tetrachlorobenzene |
|  |  | 2,3,4,6-Tetrachlorophenol |
|  |  | 2,4,6-Tribromophenol (surr) |
|  |  | 2,4,6-Trichlorophenol |
|  |  | 2,4,5-Trichlorophenol |

# D:\document\convert_tasks\transweb\1464945_1477166\1464945.pdf.files\image020.gifSEMIVOLATILE INTERNAL STANDARDS WITH CORRESPONDING ANALYTES ASSIGNED FOR QUANTITATION

(surr) = surrogate

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|  |  |
| --- | --- |
|  | TABLE 5  (continued) |
| Phenanthrene-d | Chrysene-d Perylene-d12 |
| 4-Aminobiphenyl | Benzidine Benzo(b)fluoranthene |
| Anthracene | Benzo(a)anthracene Benzo(k)fluoranthene |
| 4-Bromophenyl phenyl ether | Bis(2-ethylhexyl)phthalate Benzo(g,h,i)perylene |
| Di-n-butyl phthalate | Butyl benzyl phthalate Benzo(a)pyrene |
| 4,6-Dinitro-2-methylphenol | Chrysene Dibenz(a,j)acridine |
| Diphenylamine | 3,3&apos;-Dichlorobenzidine Dibenz(a,h)anthracene |
| Fluoranthene | p-Dimethyl aminoazobenzene 7,12-Dimethylbenz(a)anthracene |
| Hexachlorobenzene | Pyrene Di-n-octyl phthalate |
| N-Nitrosodiphenylamine | Terphenyl-d14 (surr) Indeno(1,2,3-cd)pyrene |
| Pentachlorophenol | 3-Methylcholanthrene |
| Pentachloronitrobenzene |  |
| Phenacetin |  |
| Phenanthrene |  |
| Pronamide |  |

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(surr) = surrogate

TABLE 6

# EXAMPLE SINGLE LABORATORY PERFORMANCE DATAa

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                                                                                                                   Test conc. % Recovery xof 5

          Compound (µg/L) replicates of Avg.

# (µg/L)

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          Acenaphthene 50 46.7 93.4

          Acenaphthylene 50 46.1 92.2

          Aniline 50 8.3 16.7

          Anthracene 50 48.4 96.8

         Benzoic acid 50 43.7 87.4

          Benzo(a)anthracene 50 49.6 99.2

          Benzo(b)fluoranthene 50 49.8 99.6

          Benzo(k)fluoranthene 50 50.6 101

          Benzo(a)pyrene 50 47.7 95.5

          Benzo(g,h,i)perylene 50 52.6 105

          Benzyl alcohol 50 44.4 88.8

          Bis(2-chloroethyl)ether 50 44.2 88.4

          Bis(2-chloroethoxy)methane 50 46.6 93.1

          Bis(2-chloro-1-methylethyl)ether 50 43.4 86.8

          Bis(2-ethylhexyl)phthalate 50 50.2 100

          4-Bromophenyl phenyl ether 50 48.6 97.2

          Butyl benzyl phthalate 50 49.6 99.3

          Carbazole 50 52.1 104

          2-Chloroaniline 50 38.9 77.7

          4-Chloro-3-methylphenol 50 47.3 94.6

          2-Chloronaphthalene 50 45.3 90.8

          2-Chlorophenol 50 43.1 86.2

          4-Chlorophenyl phenyl ether 50 47.3 94.6

          Chrysene 50 50.3 101

          Dibenzofuran 50 47.4 94.7

          Dibenz(a,h)anthracene 50 51.6 103

          Di-n-butyl phthalate 50 50.5 101

          1,2-Dichlorobenzene 50 35.8 71.6

          1,3-Dichlorobenzene 50 33.3 66.7

          1,4-Dichlorobenzene 50 34.4 68.7

          3,3&apos;-Dichlorobenzidine 50 32.0 64.0

          2,4-Dichlorophenol 50 47.4 94.8

          Diethyl phthalate 50 50.0 99.9

          Dimethyl phthalate 50 48.5 97.0

          2,4-Dimethylphenol 50 31.2 62.3

          4,6-Dinitro-2-methylphenol 50 57.6 115

          2,4-Dinitrophenol 50 58.7 117

          2,4-Dinitrotoluene 50 51.3 103

          2,6-Dinitrotoluene 50 50.2 100

          Di-n-octyl phthalate 50 51.1 102

          Fluoranthene 50 51.0 102

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                                                                                                                   Test conc. % Recovery xof 5

          Compound (µg/L) replicates of Avg.

# (µg/L)

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          Fluorene 50 48.5 97.0

          Hexachlorobenzene 50 49.0 97.9

          Hexachlorobutadiene 50 34.7 69.5

          Hexachlorocyclopentadiene 50 1.9 3.8

          Hexachloroethane 50 29.9 58.8

          Indeno(1,2,3-cd)pyrene 50 51.7 103

          Isophorone 50 47.1 94.3

          2-Methylnaphthalene 50 44.7 89.4

          2-Methylphenol 50 41.7 83.4

          4-Methylphenol 50 42.6 85.2

          Naphthalene 50 43.4 86.8

          2-Nitroaniline 50 48.4 96.7

          3-Nitroaniline 50 46.8 93.6

          4-Nitroaniline 50 56.1 112

          Nitrobenzene 50 47.1 94.1

          2-Nitrophenol 50 47.3 94.6

          4-Nitrophenol 50 55.4 111

          N-Nitrosodiphenylamine 50 46.7 93.4

          N-Nitroso-di-propylamine 50 44.6 89.3

          Pentachlorophenol 50 56.9 114

          Phenanthrene 50 49.7 99.4

          Phenol 50 40.9 81.8

          Pyrene 50 49.2 98.4

          1,2,4-Trichlorobenzene 50 39.1 78.2

          2,4,5-Trichlorophenol 50 47.7 95.4

          2,4,6-Trichlorophenol 50 49.2 98.4

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x=Average recovery for five initial demonstrations of capability measurements, in µg/L

a Extraction using acidic pH only with a modified continuous liquid-liquid extractor with hydrophobic membrane according to Method 3520. These values are for guidance only. Appropriate derivation of acceptance criteria for similar extraction conditions may result in much different recovery ranges. See Method 8000 for information on developing and updating acceptance criteria for method performance.

TABLE 7

# EXTRACTION EFFICIENCY AND AQUEOUS STABILITY RESULTS

|  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- |
| Compound | Percent Recovery, Day 0 | | Percent Recovery, Day 7 | | |
| Mean | RSD | | Mean | RSD |
| 3-Amino-9-ethylcarbazole | 80 | 8 | | 73 | 3 |
| 4-Chloro-1,2-phenylenediamine | 91 | 1 | | 108 | 4 |
| 4-Chloro-1,3-phenylenediamine | 84 | 3 | | 70 | 3 |
| 1,2-Dibromo-3-chloropropane | 97 | 2 | | 98 | 5 |
| Dinoseb | 99 | 3 | | 97 | 6 |
| Parathion | 100 | 2 | | 103 | 4 |
| 4,4&apos;-Methylenebis(N,Ndimethylaniline) | 108 | 4 | | 90 | 4 |
| 5-Nitro-o-toluidine | 99 | 10 | | 93 | 4 |
| 2-Picoline | 80 | 4 | | 83 | 4 |
| Tetraethyl dithiopyrophosphate | 92 | 7 | | 70 | 1 |
|  |  |  |  |  |  |

Data taken from Reference 6.

TABLE 8

# MEAN PERCENT RECOVERIES AND PERCENT RSD VALUES FOR SEMIVOLATILE

ORGANIC FROM SPIKED CLAY SOIL AND TOPSOIL BY AUTOMATED SOXHLET (SOXTEC) EXTRACTION (METHOD 3541) WITH HEXANE-ACETONE (1:1)a

|  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- |
| Compound | Clay Soil | | | Top Soil | |
| Mean Recovery | RSD | Mean Recovery | | RSD |
| 1,3-Dichlorobenzene | 0 | -- | 0 | | -- |
| 1,2-Dichlorobenzene | 0 | -- | 0 | | -- |
| Nitrobenzene | 0 | -- | 0 | | -- |
| Benzal chloride | 0 | -- | 0 | | -- |
| Benzotrichloride | 0 | -- | 0 | | -- |
| 4-Chloro-2-nitrotoluene | 0 | -- | 0 | | -- |
| Hexachlorocyclopentadiene | 4.1 | 15 | 7.8 | | 23 |
| 2,4-Dichloronitrobenzene | 35.2 | 7.6 | 21.2 | | 15 |
| 3,4-Dichloronitrobenzene | 34.9 | 15 | 20.4 | | 11 |
| Pentachlorobenzene | 13.7 | 7.3 | 14.8 | | 13 |
| 2,3,4,5-Tetrachloronitrobenzene | 55.9 | 6.7 | 50.4 | | 6.0 |
| Benefin | 62.6 | 4.8 | 62.7 | | 2.9 |
| alpha-BHC | 58.2 | 7.3 | 54.8 | | 4.8 |
| Hexachlorobenzene | 26.9 | 13 | 25.1 | | 5.7 |
| delta-BHC | 95.8 | 4.6 | 99.2 | | 1.3 |
| Heptachlor | 46.9 | 9.2 | 49.1 | | 6.3 |
| Aldrin | 97.7 | 12 | 102 | | 7.4 |
| Isopropalin | 102 | 4.3 | 105 | | 2.3 |
| Heptachlor epoxide | 90.4 | 4.4 | 93.6 | | 2.4 |
| trans-Chlordane | 90.1 | 4.5 | 95.0 | | 2.3 |
| Endosulfan I | 96.3 | 4.4 | 101 | | 2.2 |
| Dieldrin | 129 | 4.7 | 104 | | 1.9 |
| 2,5-Dichlorophenyl-4-nitrophenyl ether | 110 | 4.1 | 112 | | 2.1 |
| Endrin | 102 | 4.5 | 106 | | 3.7 |
| Endosulfan II | 104 | 4.1 | 105 | | 0.4 |
| p,p&apos;-DDT | 134 | 2.1 | 111 | | 2.0 |
| 2,3,6-Trichlorophenyl-4&apos;-nitrophenyl ether | 110 | 4.8 | 110 | | 2.8 |
| 2,3,4-Trichlorophenyl-4&apos;-nitrophenyl ether | 112 | 4.4 | 112 | | 3.3 |
| Mirex 104 5.3 108 2.2 | | | | | |
|  |  |  |  |  |  |

a The operating conditions for the Soxtec apparatus were as follows: immersion time 45 min; the sample size was 10 g; the spiking concentration was 500 ng/g, except for the surrogate compounds at 1000 ng/g, 2,5-Dichlorophenyl-4-nitrophenyl ether, 2,3,6-Trichlorophenyl-4nitrophenyl ether, and 2,3,4-Trichlorophenyl-4-nitrophenyl ether at 1500 ng/g, Nitrobenzene at 2000 ng/g, and 1,3-Dichlorobenzene and 1,2-Dichlorobenzene at 5000 ng/g.

TABLE 9

SINGLE LABORATORY ACCURACY AND PRECISION DATA FOR THE EXTRACTION

OF SEMIVOLATILE ORGANICS FROM SPIKED CLAY BY AUTOMATED SOXHLET (SOXTEC) (METHOD 3541)a

# D:\document\convert_tasks\transweb\1464945_1477166\1464945.pdf.files\image025.gifRSD 47.8 5.6

                              Bis(2-chloroethyl) ether 25.4 13

                              2-Chlorophenol 42.7 4.3

                              Benzyl alcohol 55.9 7.2

                              2-Methylphenol 17.6 6.6

                              Bis(2-chloro-1-methylethyl)ether 15.0 15

                              4-Methylphenol 23.4 6.7

                              N-Nitroso-di-n-propylamine 41.4 6.2

                              Nitrobenzene 28.2 7.7

                              Isophorone 56.1 4.2

                              2-Nitrophenol 36.0 6.5

                              2,4-Dimethylphenol 50.1 5.7

                              Benzoic acid 40.6 7.7

                              Bis(2-chloroethoxy)methane 44.1 3.0

                              2,4-Dichlorophenol 55.6 4.6

                              1,2,4-Trichlorobenzene 18.1 31

                              Naphthalene 26.2 15

                              4-Chloroaniline 55.7 12

                              4-Chloro-3-methylphenol 65.1 5.1

                              2-Methylnaphthalene 47.0 8.6

                              Hexachlorocyclopentadiene 19.3 19

                              2,4,6-Trichlorophenol 70.2 6.3

                              2,4,5-Trichlorophenol 26.8 2.9

                              2-Chloronaphthalene 61.2 6.0

                              2-Nitroaniline 73.8 6.0

                              Dimethyl phthalate 74.6 5.2

                              Acenaphthylene 71.6 5.7

                              3-Nitroaniline 77.6 5.3

                              Acenaphthene 79.2 4.0

                              2,4-Dinitrophenol 91.9 8.9

                              4-Nitrophenol 62.9 16

                              Dibenzofuran 82.1 5.9

                              2,4-Dinotrotoluene 84.2 5.4

                              2,6-Dinitrotoluene 68.3 5.8

                              Diethyl phthalate 74.9 5.4

                              4-Chlorophenyl-phenyl ether 67.2 3.2

                              Fluorene 82.1 3.4

                              4-Nitroaniline 79.0 7.9

                              4,6-Dinitro-2-methylphenol 63.4 6.8

                              N-Nitrosodiphenylamine 77.0 3.4

                              4-Bromophenyl-phenyl ether 62.4 3.0

|  |  |  |
| --- | --- | --- |
| Compound | Mean Recovery | RSD |
| Hexachlorobenzene | 72.6 | 3.7 |
| Pentachlorophenol | 62.7 | 6.1 |
| Phenanthrene | 83.9 | 5.4 |
| Anthracene | 96.3 | 3.9 |
| Di-n-butyl phthalate | 78.3 | 40 |
| Fluoranthene | 87.7 | 6.9 |
| Pyrene | 102 | 0.8 |
| Butyl benzyl phthalate | 66.3 | 5.2 |
| 3,3&apos;-Dichlorobenzidine | 25.2 | 11 |
| Benzo(a)anthracene | 73.4 | 3.8 |
| Bis(2-ethylhexyl)phthalate | 77.2 | 4.8 |
| Chrysene | 76.2 | 4.4 |
| Di-n-octyl phthalate | 83.1 | 4.8 |
| Benzo(b)fluoranthene | 82.7 | 5.0 |
| Benzo(k)fluoranthene | 71.7 | 4.1 |
| Benzo(a)pyrene | 71.7 | 4.1 |
| Indeno(1,2,3-cd)pyrene | 72.2 | 4.3 |
| Dibenz(a,h)anthracene | 66.7 | 6.3 |
| Benzo(g,h,i)perylene | 63.9 | 8.0 |
| 1,2-Dichlorobenzene | 0 | -- |
| 1,3-Dichlorobenzene | 0 | -- |
| 1,4-Dichlorobenzene | 0 | -- |
| Hexachloroethane | 0 | -- |
| Hexachlorobutadiene 0 -- | | |

a Number of determinations was three. The operating conditions for the Soxtec apparatus were as follows: immersion time 45 min; the sample size was 10 g clay soil; the spike concentration was 6 mg/kg per compound. The sample was allowed to equilibrate 1 hour after spiking.

Data taken from Reference 7.

# TABLE 10

PRECISION AND BIAS VALUES FOR METHOD 3542a

|  |  |  |  |
| --- | --- | --- | --- |
| Compound | Mean Recovery | Standard Deviation | % RSD |
| 2-Fluorophenol | 74.6 | 28.6 | 38.3 |
| Phenol-d5 | 77.8 | 27.7 | 35.6 |
| Nitrobenzene-d5 | 65.6 | 32.5 | 49.6 |
| 2-Fluorobiphenyl | 75.9 | 30.3 | 39.9 |
| 2,4,6-Tribromophenol | 67.0 | 34.0 | 50.7 |
| Terphenyl-d14 78.6 32.4 41.3 | | | |

a The surrogate values shown in Table 10 represent mean recoveries for surrogates in all Method 0010 matrices in a field dynamic spiking study.

TABLE 11

# PRESSURIZED FLUID EXTRACTION (METHOD 3545) RECOVERY VALUES AS PERCENT OF SOXTECTM

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                                                           Low Mid High Low Mid High Low Mid High

Phenol 93.3 78.7 135.9 73.9 82.8 124.6 108.8 130.6 89.7 102.0

Bis(2-chloroethyl)ether 102.1 85.1 109.1 96.0 88.0 103.6 122.3 119.9 90.8 101.9

2-Chlorophenol 100.8 82.6 115.0 93.8 88.9 111.1 115.0 115.3 91.9 101.6

1,3-Dichlorobenzene 127.7 129.7 110.0 \*364.2 129.9 119.0 \*241.3 \*163.7 107.1 120.6

1,4-Dichlorobenzene 127.9 127.0 110.5 \*365.9 127.8 116.4 \*309.6 \*164.1 105.8 119.2

1,2-Dichlorobenzene 116.8 115.8 101.3 \*159.2 113.4 105.5 \*189.3 134.0 100.4 112.5

2-Methylphenol 98.9 82.1 119.7 87.6 89.4 111.0 133.2 128.0 92.1 104.7

Bis(2-chloro-1-methylethyl)

                                                          109.4         71.5         108.0         81.8          81.0          88.6         118.1        148.3         94.8        100.2

ether

o-Toluidine 100.0 89.7 117.2 100.0 \*152.5 120.3 100.0 \*199.5 102.7 110.3

N-Nitroso-di-n-propylamine 103.0 79.1 107.7 83.9 88.1 96.2 109.9 123.3 91.4 98.1

Hexachloroethane 97.1 125.1 111.0 \*245.4 117.1 128.1 \*566.7 147.9 103.7 118.6

Nitrobenzene 104.8 82.4 106.6 86.8 84.6 101.7 119.7 122.1 93.3 100.2

Isophorone 100.0 86.4 98.2 87.1 87.5 109.7 135.5 118.4 92.7 101.7

2,4-Dimethylphenol 100.0 104.5 140.0 100.0 114.4 123.1 100.0 \*180.6 96.3 109.8

2-Nitrophenol 80.7 80.5 107.9 91.4 86.7 103.2 122.1 107.1 87.0 96.3

Bis(chloroethoxy)methane 94.4 80.6 94.7 86.5 84.4 99.6 130.6 110.7 93.2 97.2

2,4-Dichlorophenol 88.9 87.8 111.4 85.9 87.6 103.5 123.3 107.0 92.1 98.6

1,2,4-Trichlorobenzene 98.0 97.8 98.8 123.0 93.7 94.5 137.0 99.4 95.3 104.2

Naphthalene 101.7 97.2 123.6 113.2 102.9 129.5 \*174.5 114.0 89.8 106.1

4-Chloroaniline 100.0 \*150.2 \*162.4 100.0 125.5 \*263.6 100.0 \*250.8 114.9 108.1

Hexachlorobutadiene 101.1 98.7 102.2 124.1 90.3 98.0 134.9 96.1 96.8 104.7

4-Chloro-3-methylphenol 90.4 80.2 114.7 79.0 85.2 109.8 131.6 116.2 90.1 99.7

2-Methylnaphthalene 93.2 89.9 94.6 104.1 92.2 105.9 146.2 99.1 93.3 102.1

Hexachlorocyclopentadiene 100.0 100.0 0.0 100.0 100.0 6.8 100.0 100.0 \*238.3 75.8

2,4,6-Trichlorophenol 94.6 90.0 112.0 84.2 91.2 103.6 101.6 95.9 89.8 95.9

2,4,5-Trichlorophenol 84.4 91.9 109.6 96.1 80.7 103.6 108.9 83.9 87.9 94.1

2-Chloronaphthalene 100.0 91.3 93.6 97.6 93.4 98.3 106.8 93.0 92.0 96.2

2-Nitroaniline 90.0 83.4 97.4 71.3 88.4 89.9 112.1 113.3 87.7 92.6

2,6-Dinitrotoluene 83.1 90.6 91.6 86.4 90.6 90.3 104.3 84.7 90.9 90.3

Acenaphthylene 104.9 95.9 100.5 99.0 97.9 108.8 118.5 97.8 92.0 101.7

3-Nitroaniline \*224.0 115.6 97.6 100.0 111.8 107.8 0.0 111.7 99.0 92.9

Acenaphthene 102.1 92.6 97.6 97.2 96.9 104.4 114.2 92.0 89.0 98.4

4-Nitrophenol 0.0 93.2 121.5 18.1 87.1 116.6 69.1 90.5 84.5 75.6

2,4-Dinitrotoluene 73.9 91.9 100.2 84.7 93.8 98.9 100.9 84.3 87.3 90.7

Dibenzofuran 89.5 91.7 109.3 98.5 92.2 111.4 113.8 92.7 90.4 98.8

4-Chlorophenyl phenyl ether 83.0 94.5 98.7 95.7 94.3 94.2 111.4 87.7 90.3 94.4

Fluorene 85.2 94.9 89.2 102.0 95.5 93.8 121.3 85.7 90.9 95.4

4-Nitroaniline 77.8 114.8 94.5 129.6 103.6 95.4 \*154.1 89.3 87.5 99.1

N-Nitrosodiphenylamine 82.6 96.7 93.8 92.9 93.4 116.4 97.5 110.9 86.7 96.8

4-Bromophenyl phenyl ether 85.6 92.9 92.8 91.1 107.6 89.4 118.0 97.5 87.1 95.8

Hexachlorobenzene 95.4 91.7 92.3 95.4 93.6 83.7 106.8 94.3 90.0 93.7

D:\document\convert_tasks\transweb\1464945_1477166\1464945.pdf.files\image027.gifClay Loam Sand Mean

Compound

                                                           Low Mid High Low Mid High Low Mid High Rec.

|  |  |  |  |  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- |
| Pentachlorophenol | 68.2 | 85.9 | 107.7 | 53.2 | 89.8 | 88.1 | 96.6 | 59.8 | 81.3 | 81.2 |
| Phenanthrene | 92.1 | 93.7 | 93.3 | 100.0 | 97.8 | 113.3 | 124.4 | 101.0 | 89.9 | 100.6 |
| Anthracene | 101.6 | 95.0 | 93.5 | 92.5 | 101.8 | 118.4 | 123.0 | 94.5 | 90.6 | 101.2 |
| Carbazole | 94.4 | 99.3 | 96.6 | 105.5 | 96.7 | 111.4 | 115.7 | 83.2 | 88.9 | 99.1 |
| Fluoranthene | 109.9 | 101.4 | 94.3 | 111.6 | 96.6 | 109.6 | 123.2 | 85.4 | 92.7 | 102.7 |
| Pyrene | 106.5 | 105.8 | 107.6 | 116.7 | 90.7 | 127.5 | 103.4 | 95.5 | 93.2 | 105.2 |
| 3,3&apos;-Dichlorobenzidine | 100.0 | \*492.3 | 131.4 | 100.0 | \*217.6 | \*167.6 | 100.0 | \*748.8 | 100.0 | 116.5 |
| Benzo(a)anthracene | 98.1 | 107.0 | 98.4 | 119.3 | 98.6 | 104.0 | 105.0 | 93.4 | 89.3 | 101.5 |
| Chrysene | 100.0 | 108.5 | 100.2 | 116.8 | 93.0 | 117.0 | 106.7 | 93.6 | 90.2 | 102.9 |
| Benzo(b)fluoranthene | 106.6 | 109.9 | 75.6 | 121.7 | 100.7 | 93.9 | 106.9 | 81.9 | 93.6 | 99.0 |
| Benzo(k)fluoranthene | 102.4 | 105.2 | 88.4 | 125.5 | 99.4 | 95.1 | 144.7 | 89.2 | 78.1 | 103.1 |
| Benzo(a)pyrene | 107.9 | 105.5 | 80.8 | 122.3 | 97.7 | 104.6 | 101.7 | 86.2 | 92.0 | 99.9 |
| Indeno(1,2,3-cd)pyrene | 95.1 | 105.7 | 93.8 | 126.0 | 105.2 | 90.4 | 133.6 | 82.6 | 91.9 | 102.7 |
| Dibenz(a,h)anthracene | 85.0 | 102.6 | 82.0 | 118.8 | 100.7 | 91.9 | 142.3 | 71.0 | 93.1 | 98.6 |
| Benzo(g,h,i)perylene | 98.0 | 0.0 | 81.2 | 0.0 | 33.6 | 78.6 | 128.7 | 83.0 | 94.2 | 66.4 |
| Mean | 95.1 | 94.3 | 101.0 | 95.5 | 96.5 | 104.1 | 113.0 | 100.9 | 92.5 |  |

\*Values greater than 150% were not used to determine the averages, but the 0% values were used.

TABLE 12

FROM A CERTIFIED REFERENCE SEDIMENT EC-1, USING METHOD 3561

# (SFE - SOLID TRAP)

|  |  |  |  |  |
| --- | --- | --- | --- | --- |
| Compound | Certified Value (mg/kg) | SFE Valuea (mg/kg) | Percent of Certified Value | SFE RSD |
| Naphthalene | (27.9)b | 41.3 ± 3.6 | (148) | 8.7 |
| Acenaphthylene | (0.8) | 0.9 ± 0.1 | (112) | 11.1 |
| Acenaphthene | (0.2) | 0.2 ± 0.01 | (100) | 0.05 |
| Fluorene | (15.3) | 15.6 ± 1.8 | (102) | 11.5 |
| Phenanthrene | 15.8 ± 1.2 | 16.1 ± 1.8 | 102 | 11.2 |
| Anthracene | (1.3) | 1.1 ± 0.2 | (88) | 18.2 |
| Fluoranthene | 23.2 ± 2.0 | 24.1 ± 2.1 | 104 | 8.7 |
| Pyrene | 16.7 ± 2.0 | 17.2 ± 1.9 | 103 | 11.0 |
| Benz(a)anthracene | 8.7 ± 0.8 | 8.8 ± 1.0 | 101 | 11.4 |
| Chrysene | (9.2) | 7.9 ± 0.9 | (86) | 11.4 |
| Benzo(b)fluoranthene | 7.9 ± 0.9 | 8.5 ± 1.1 | 108 | 12.9 |
| Benzo(k)fluoranthene | 4.4 ± 0.5 | 4.1 ± 0.5 | 91 | 12.2 |
| Benzo(a)pyrene | 5.3 ± 0.7 | 5.1 ± 0.6 | 96 | 11.8 |
| Indeno(1,2,3-cd)pyrene | 5.7 ± 0.6 | 5.2 ± 0.6 | 91 | 11.5 |
| Benzo(g,h,i)perylene | 4.9 ± 0.7 | 4.3 ± 0.5 | 88 | 11.6 |
| Dibenz(a,h)anthracene (1.3) 1.1 ± 0.2 (85) 18.2 | | | | |

a RSDs for the SFE values are based on six replicate extractions.

b Values in parentheses were obtained from, or compared to, Soxhlet extraction results which were not certified.

Data are taken from Reference 10.

TABLE 13

# FROM A CERTIFIED REFERENCE SEDIMENT HS-3, USING METHOD 3561 (SFE - SOLID TRAP)

|  |  |  |  |  |
| --- | --- | --- | --- | --- |
| Compound | Certified Value (mg/kg) | SFE Valuea (mg/kg) | Percent of Certified Value | SFE RSD |
| Naphthalene | 9.0 ± 0.7 | 7.4 ± 0.6 | 82 | 8.1 |
| Acenaphthylene | 0.3 ± 0.1 | 0.4 ± 0.1 | 133 | 25.0 |
| Acenaphthene | 4.5 ± 1.5 | 3.3 ± 0.3 | 73 | 9.0 |
| Fluorene | 13.6 ± 3.1 | 10.4 ± 1.3 | 77 | 12.5 |
| Phenanthrene | 85.0 ± 20.0 | 86.2 ± 9.5 | 101 | 11.0 |
| Anthracene | 13.4 ± 0.5 | 12.1 ± 1.5 | 90 | 12.4 |
| Fluoranthene | 60.0 ± 9.0 | 54.0 ± 6.1 | 90 | 11.3 |
| Pyrene | 39.0 ± 9.0 | 32.7 ± 3.7 | 84 | 11.3 |
| Benz(a)anthracene | 14.6 ± 2.0 | 12.1 ± 1.3 | 83 | 10.7 |
| Chrysene | 14.1 ± 2.0 | 12.0 ± 1.3 | 85 | 10.8 |
| Benzo(b)fluoranthene | 7.7 ± 1.2 | 8.4 ± 0.9 | 109 | 10.7 |
| Benzo(k)fluoranthene | 2.8 ± 2.0 | 3.2 ± 0.5 | 114 | 15.6 |
| Benzo(a)pyrene | 7.4 ± 3.6 | 6.6 ± 0.8 | 89 | 12.1 |
| Indeno(1,2,3-cd)pyrene | 5.0 ± 2.0 | 4.5 ± 0.6 | 90 | 13.3 |
| Benzo(g,h,i)perylene | 5.4 ± 1.3 | 4.4 ± 0.6 | 82 | 13.6 |
| Dibenz(a,h)anthracene 1.3 ± 0.5 1.1 ± 0.3 85 27.3 | | | | |

a RSDs for the SFE values are based on three replicate extractions.

Data are taken from Reference 10.

TABLE 14

# FROM A CERTIFIED REFERENCE SOIL SRS103-100, USING METHOD 3561 (SFE – LIQUID TRAP)

|  |  |  |  |  |
| --- | --- | --- | --- | --- |
| Compound | Certified Value (mg/kg) | SFE Valuea (mg/kg) | Percent of Certified Value | SFE RSD |
| Naphthalene | 32.4 ± 8.2 | 29.55 | 91 | 10.5 |
| 2-Methylnaphthalene | 62.1 ± 11.5 | 76.13 | 122 | 2.0 |
| Acenaphthene | 632 ± 105 | 577.28 | 91 | 2.9 |
| Dibenzofuran | 307 ± 49 | 302.25 | 98 | 4.1 |
| Fluorene | 492 ± 78 | 427.15 | 87 | 3.0 |
| Phenanthrene | 1618 ± 340 | 1278.03 | 79 | 3.4 |
| Anthracene | 422 ± 49 | 400.80 | 95 | 2.6 |
| Fluoranthene | 1280 ± 220 | 1019.13 | 80 | 4.5 |
| Pyrene | 1033 ± 285 | 911.82 | 88 | 3.1 |
| Benz(a)anthracene | 252 ± 8 | 225.50 | 89 | 4.8 |
| Chrysene | 297 ± 26 | 283.00 | 95 | 3.8 |
| Benzo(a)pyrene | 97.2 ± 17.1 | 58.28 | 60 | 6.5 |
| Benzo(b)fluoranthene +  Benzo(k)fluoranthene | 153 ± 22 | 130.88 | 86 | 10.7 |

a RSDs for the SFE values are based on four replicate extractions.

Data are taken from Reference 11.

TABLE 15

# SINGLE LABORATORY RECOVERY DATA FOR SPE (METHOD 3535) OF BASE/NEUTRAL/ACID EXTRACTABLES FROM SPIKED TCLP BUFFERS LOW SPIKE LEVEL

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| Analyte | Spike  Level  (µg/L) | Buffer 1 (pH=2.886) | | | Buffer 2 (pH=4.937) | |
| Recovery (%) | RSD | Recovery (%) | | RSD |
| 1,4-Dichlorobenzene | 3,750 | 63 | 10 | 63 | | 9 |
| Hexachloroethane | 1,500 | 55 | 6 | 77 | | 4 |
| Nitrobenzene | 1,000 | 82 | 10 | 100 | | 5 |
| Hexachlorobutadiene | 250 | 65 | 3 | 56 | | 4 |
| 2,4-Dinitrotoluene | 65 | 89 | 4 | 101 | | 5 |
| Hexachlorobenzene | 65 | 98 | 5 | 95 | | 6 |
| o-Cresol | 100,000 | 83 | 10 | 85 | | 5 |
| m-Cresol\* | 100,000 | 86 | 8 | 85 | | 3 |
| p-Cresol\* | 100,000 | \* | \* | \* | | \* |
| 2,4,6-Trichlorophenol | 1,000 | 84 | 12 | 95 | | 12 |
| 2,4,5-Trichlorophenol | 200,000 | 83 | 11 | 88 | | 3 |
| Pentachlorophenol 50,000 82 9 78 9 | | | | | | |
|  |  |  |  |  |  |  |

Results from seven replicate spiked buffer samples.

\*In this study, m-cresol and p-cresol co-eluted and were quantified as a mixture of both isomers.

Data from Reference 12.

# TABLE 16

SINGLE LABORATORY RECOVERY DATA FOR SPE (METHOD 3535) OF

BASE/NEUTRAL/ACID EXTRACTABLES FROM SPIKED TCLP BUFFERS

# HIGH SPIKE LEVEL

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| Analyte | Spike  Level  (µg/L) | Buffer 1 (pH=2.886) | | | Buffer 2 (pH=4.937) | |
| Recovery (%) | RSD | Recovery (%) | | RSD |
| 1,4-Dichlorobenzene | 15,000 | 63 | 10 | 63 | | 9 |
| Hexachloroethane | 6,000 | 54 | 7 | 46 | | 7 |
| Nitrobenzene | 4,000 | 81 | 4 | 81 | | 13 |
| Hexachlorobutadiene | 1,000 | 81 | 5 | 70 | | 11 |
| 2,4-Dinitrotoluene | 260 | 99 | 8 | 98 | | 3 |
| Hexachlorobenzene | 260 | 89 | 8 | 91 | | 9 |
| o-Cresol\* | 400,000 | 92 | 15 | 90 | | 4 |
| m-Cresol\* | 400,000 | 95 | 8 | 82 | | 6 |
| p-Cresol\* | 400,000 | 82 | 14 | 84 | | 7 |
| 2,4,6-Trichlorophenol | 4,000 | 93 | 12 | 104 | | 12 |
| 2,4,5-Trichlorophenol | 800,000 | 93 | 14 | 97 | | 23 |
| Pentachlorophenol 200,000 84 9 73 8 | | | | | | |
|  |  |  |  |  |  |  |

Results from seven replicate spiked buffer samples.

\*In this study, recoveries of these compounds were determined from triplicate spikes of the individual compounds into separate buffer solutions.

Data from Reference 12.

TABLE 17

RECOVERY DATA FROM THREE LABORATORIES FOR SPE (METHOD 3535)

OF BASE/NEUTRAL/ACID/EXTRACTABLES FROM SPIKED TCLP LEACHATES FROM SOIL

# SAMPLES

|  |  |  |  |  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- |
| Buffer 1 pH=2.886 |  | Lab 1 | |  | Lab 2 | |  | Lab 3 | |  |
| Analyte | Spike  Level  (µg/L)\* | %R | RSD | n | %R | RSD | n | %R | RSD | n |
| o-Cresol | 200,000 | 86 | 8 | 7 | 35.3 | 0.7 | 3 | 7.6 | 6 | 3 |
| m-Cresol\*\* | -- | 77 | 8 | 7 | -- | -- | -- | -- | -- | -- |
| p-Cresol\*\* | -- | -- | -- | -- | -- | -- | -- | 7.7 | 11 | 3 |
| 2,4,6-Trichlorophenol | 2,000 | 106 | 6 | 7 | 96.3 | 3.9 | 3 | 44.8 | 5 | 3 |
| 2,4,5-Trichlorophenol | 400,000 | 93 | 3 | 7 | 80.5 | 4.5 | 3 | 63.3 | 11 | 3 |
| Pentachlorophenol | 100,000 | 79 | 2 | 7 | 33.8 | 12.2 | 3 | 29.2 | 13 | 3 |
| 1,4-Dichlorobenzene | 7,500 | 51 | 5 | 7 | 81.3 | 5.3 | 3 | 19.2 | 7 | 3 |
| Hexachloroethane | 3,000 | 50 | 5 | 7 | 66.2 | 2.1 | 3 | 12.6 | 11 | 3 |
| Nitrobenzene | 2,000 | 80 | 8 | 7 | 76.3 | 5.3 | 3 | 63.9 | 12 | 3 |
| Hexachlorobutadiene | 500 | 53 | 8 | 7 | 63.3 | 4.8 | 3 | 9.6 | 9 | 3 |
| 2,4-Dinitrotoluene | 130 | 89 | 8 | 7 | 35.7 | 2.6 | 3 | 58.2 | 17 | 3 |
| Hexachlorobenzene | 130 | 84 | 21 | 7 | 92.3 | 1.6 | 3 | 71.7 | 9 | 3 |

(continued)

# TABLE 17 (continued)

|  |  |  |  |  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- |
| Buffer 2 pH=4.937 |  | Lab 1 | |  | Lab 2 | |  | Lab 3 | |  |
| Analyte | Spike  Level  (µg/L)\* | %R | RSD | n | %R | RSD | n | %R | RSD | n |
| o-Cresol | 200,000 | 97 | 13 | 7 | 37.8 | 4.5 | 3 | 6.1 | 24 | 3 |
| m-Cresol\*\* | -- | 83 | 4 | 7 | -- | -- | -- | 6.0 | 25 | 3 |
| p-Cresol\*\* | -- | -- | -- | -- | -- | -- | -- | -- | -- | -- |
| 2,4,6-Trichlorophenol | 2,000 | 104 | 4 | 7 | 91.7 | 8.0 | 3 | 37.7 | 25 | 3 |
| 2,4,5-Trichlorophenol | 400,000 | 94 | 4 | 7 | 85.2 | 0.4 | 3 | 64.4 | 10 | 3 |
| Pentachlorophenol | 100,000 | 109 | 11 | 7 | 41.9 | 28.2 | 3 | 36.6 | 32 | 3 |
| 1,4-Dichlorobenzene | 7,500 | 50 | 5 | 7 | 79.7 | 1.0 | 3 | 26.5 | 68 | 3 |
| Hexachloroethane | 3,000 | 51 | 3 | 7 | 64.9 | 2.0 | 3 | 20.3 | 90 | 3 |
| Nitrobenzene | 2,000 | 80 | 4 | 7 | 79.0 | 2.3 | 3 | 59.4 | 6 | 3 |
| Hexachlorobutadiene | 500 | 57 | 5 | 7 | 60 | 3.3 | 3 | 16.6 | 107 | 3 |
| 2,4-Dinitrotoluene | 130 | 86 | 6 | 7 | 38.5 | 5.2 | 3 | 62.2 | 6 | 3 |
| Hexachlorobenzene | 130 | 86 | 7 | 7 | 91.3 | 0.9 | 3 | 75.5 | 5 | 3 |

\* 250-mL aliquots of leachate were spiked. Lab 1 spiked at one-half these levels.

\*\* m-Cresol and p-Cresol co-elute. Lab 1 and Lab 3 reported o-Cresol and the sum of m- and pCresol. Lab 2 reported the sum of all three isomers of Cresol.

Data from Reference 12.

TABLE 18

SINGLE LABORATORY PAH ANALYSIS DATA FROM:

# A REAL SOIL CONTAMINATED WITH CREOSOTE, USING METHOD 3546 (MICROWAVE EXTRACTION)

|  |  |  |  |
| --- | --- | --- | --- |
| Compound | Concentration (µg/kg) | RSD (%) | REAC values (µg/kg) |
| Naphthalene | 2,170 | 12.4 | 710,000 |
| 2-Methylnaphthalene | 28,710 | 3.1 | N/R |
| 1-Methylnaphthalene | 33,180 | 2.4 | N/R |
| Biphenyl | 13,440 | 6.0 | N/R |
| 2,6-Dimethylnaphthalene | 52,990 | 3.8 | N/R |
| Acenaphthylene | 16,320 | 3.1 | 21,000 |
| Acenaphthene | 801,210 | 6.0 | 1,700,000 |
| Fluorene | 789,980 | 3.4 | 990,000 |
| Phenanthrene | 1,627,480 | 0.7 | 3,300,00 |
| Anthracene | 346,010 | 4.0 | 360,000 |
| Benzo(a)anthracene | 300,380 | 2.7 | 310,000 |
| Fluoranthene | 1,331,690 | 1.6 | 1,600,000 |
| Pyrene | 1,037,710 | 3.0 | 1,100,000 |
| Chrysene | 293,200 | 3.4 | 320,000 |
| Benzo(b)fluoranthene | 152,000 | 3.8 | 140,000 |
| Benzo(k)fluoranthene | 127,740 | 3.6 | 130,000 |
| Benzo(e)pyrene | 87,610 | 3.9 | N/R |
| Benzo(a)pyrene | 128,330 | 3.9 | 110,000 |
| Perylene | 35,260 | 4.3 | N/R |
| Indeno(1,2,3-cd)pyrene | 63,900 | 5.0 | 25,000 |
| Dibenz(a,h)anthracene | 17,290 | 6.9 | N/R |
| Benzo(g,h,i)perylene 42,270 6.9 20,000 | | | |

n = 4

Soil samples obtained from U.S. EPA Emergency Response Center archive bank through their

Response Engineering and Analytical Contract (REAC) laboratory (Edison, NJ). The standard Soxhlet extraction procedures were performed by REAC three years earlier; this long storage period is believed to account for the low naphthalene recovery data in the present study.

REAC data labeled N/R = not reported

TABLE 19

# HS-5 MARINE SEDIMENT MATERIALS, USING METHOD 3546 (MICROWAVE EXTRACTION)

|  |  |  |  |
| --- | --- | --- | --- |
| Compound | Certified  Value  (µg/kg) | Confidence  Interval  (µg/kg) | Recovery (%) |
| Naphthalene | 250 | 180 - 320 | 76 |
| Acenaphthylene | 150 | \* | 107 |
| Acenaphthene | 230 | 130 - 330 | 61 |
| Fluorene | 400 | 300 - 500 | 63 |
| Phenanthrene | 5,200 | 4,200 - 6,200 | 72 |
| Anthracene | 380 | 230 - 530 | 84 |
| Fluoranthene | 8,400 | 5,800 - 10,000 | 81 |
| Pyrene | 5,800 | 4,000 - 7,600 | 69 |
| Benzo(a)anthracene | 2,900 | 1,700 - 4,100 | 53 |
| Chrysene | 2,800 | 1,900 - 3,700 | 76 |
| Benzo(b)fluoranthene | 2,000 | 1,000 - 3,000 | 84 |
| Benzo(k)fluoranthene | 1,000 | 600 - 1,400 | 137 |
| Benzo(a)pyrene | 1,700 | 900 - 2,500 | 52 |
| Indeno(1,2,3-cd)pyrene | 1,300 | 600 - 2,000 | 63 |
| Dibenz(a,h)anthracene | 200 | 100 - 300 | 125 |
| Benzo(g,h,i)perylene 1,300 1000 - 1600 64 | | | |

n = 3

\* values not certified

The uncertainties represent 90% confidence intervals.

TABLE 20

# HS-4 MARINE SEDIMENT MATERIALS, USING METHOD 3546 (MICROWAVE EXTRACTION)

|  |  |  |  |
| --- | --- | --- | --- |
| Compound | Certified  Value  (µg/kg) | Confidence  Interval  (µg/kg) | Recovery (%) |
| Naphthalene | 150 | \* | 54 |
| Acenaphthylene | 150 | \* | 82 |
| Acenaphthene | 150 | \* | 63 |
| Fluorene | 150 | \* | 81 |
| Phenanthrene | 680 | 600 - 760 | 81 |
| Anthracene | 140 | 70 - 210 | 108 |
| Fluoranthene | 1250 | 1,150 - 1,350 | 84 |
| Pyrene | 940 | 820 - 1,060 | 85 |
| Benzo(a)anthracene | 530 | 470 - 580 | 78 |
| Chrysene | 650 | 570 - 730 | 84 |
| Benzo(b)fluoranthene | 700 | 550 - 850 | 84 |
| Benzo(k)fluoranthene | 360 | 310 - 410 | 156 |
| Benzo(a)pyrene | 650 | 570 - 730 | 73 |
| Indeno(1,2,3-cd)pyrene | 510 | 360 - 660 | 88 |
| Dibenz(a,h)anthracene | 120 | 70 - 170 | 117 |
| Benzo(g,h,i)perylene 580 360 - 800 91 | | | |

n = 3

\* values not certified

The uncertainties represent 90% confidence intervals.

TABLE 21

# HS-3 MARINE SEDIMENT MATERIALS, USING METHOD 3546 (MICROWAVE EXTRACTION)

|  |  |  |  |
| --- | --- | --- | --- |
| Compound | Certified  Value  (µg/kg) | Confidence  Interval  (µg/kg) | Recovery (%) |
| Naphthalene | 9,000 | 8300 - 9,700 | 61 |
| Acenaphthylene | 300 | 200 - 400 | 199 |
| Acenaphthene | 4,500 | 3,000 - 6,000 | 80 |
| Fluorene | 13,300 | 10,200 - 16,400 | 58 |
| Phenanthrene | 85,000 | 65000 - 105,000 | 87 |
| Anthracene | 13,400 | 12,900 - 13,900 | 47 |
| Fluoranthene | 60,000 | 51,000 - 69,000 | 91 |
| Pyrene | 39,000 | 30,000 - 48,000 | 86 |
| Benzo(a)anthracene | 14,600 | 12,600 - 16,600 | 78 |
| Chrysene | 14,100 | 12,100 - 16,100 | 91 |
| Benzo(b)fluoranthene | 7,700 | 6,500 - 8,900 | 101 |
| Benzo(k)fluoranthene | 2,800 | 800 - 4,800 | 275 |
| Benzo(a)pyrene | 7,400 | 3,000 - 7,000 | 74 |
| Indeno(1,2,3-cd)pyrene | 5,400 | 4,100 - 6,700 | 100 |
| Dibenz(a,h)anthracene | 1,300 | 800 - 1,800 | 118 |
| Benzo(g,h,i)perylene 5,000 3,000 - 7,000 99 | | | |

n = 3

\* values not certified

The uncertainties represent 90% confidence intervals.

TABLE 22

SINGLE LABORATORY PAH RECOVERY DATA FROM:

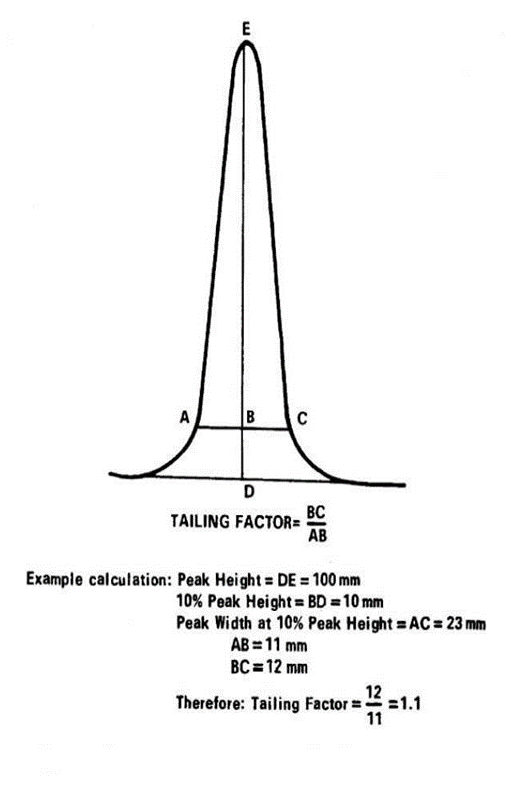
# SRM 1941 MARINE SEDIMENT MATERIALS, USING METHOD 3546 (MICROWAVE EXTRACTION)

|  |  |  |
| --- | --- | --- |
| Compound | Certified Value (µg/kg) | Recovery (%) |
| Naphthalene | 1010 | 97.4 |
| Fluorene | 100 | 100.0 |
| Phenanthrene | 490 | 102.0 |
| Fluoranthene | 980 | 116.7 |
| Pyrene | 810 | 97.3 |
| Benzo(a)anthracene | 430 | 89.8 |
| Chrysene | 380 | 130.3 |
| Benzo(b)fluoranthene | 740 | 95.8 |
| Benzo(k)fluoranthene | 360 | 130.2 |
| Benzo(e)pyrene | 550 | 81.0 |
| Benzo(a)pyrene | 630 | 76.0 |
| Perylene | 450 | 72.4 |
| Indeno(1,2,3-cd)pyrene | 500 | 126.0 |
| Dibenz(a,h)anthracene | 110 | 78.7 |
| Benzo(g,h,i)perylene 530 85.2 | | |

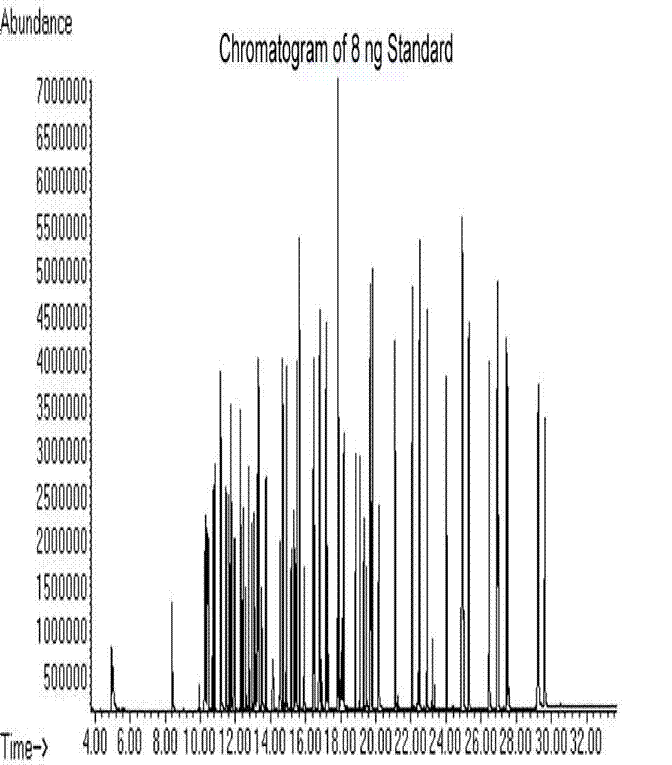
n = 3

All RSDs < 10%.

FIGURE 1 TAILING FACTOR CALCULATION



# FIGURE 2 GAS CHROMATOGRAM OF BASE/NEUTRAL AND ACID CALIBRATION STANDARD



Appendix A:

Summary of Revisions to Method 8270D (From Revision 4 Feb 2007)

1.    Improved overall method formatting for consistency with new SW-846 methods style guidance. The format was updated to Microsoft Word .docx.

2.    Many minor editorial and technical revisions were made throughout to improve method clarity.

3.    The revision number was changed to 5 and the date published was changed to July 2014.

4.    This appendix was added showing changes from the previous revision.

5.    Chemical name was changed by the Integrated Risk Information System (IRIS) on November 30, 2007 from Bis(2-chloroisopropyl)ether to Bis(2-chloro-1-methylethyl)ether (common name). This compound is also known as 2,2&apos;-oxybis(1-chloropropane) (CAS index name). See the link at , Section VII for the “Revision History” and Section VIII, for “Synonyms” of this chemical. http://www.epa.gov/iris/subst/0407.htm

6.    Updated information on LLOQ and method blank evaluation was included based on language found in Method 8000D.